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## ECCC RECOMMENDATIONS - VOLUME 6 [Issue 1]

# **Residual Life Assessment and Microstructure**

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## ECCC RECOMMENDATIONS - VOLUME 6 [Issue 1]

## **Residual life assessment and microstructure**

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#### ABSTRACT

ECCC Recommendations - Volume 6 Part x gives an overview of the correlation among residual life assessment and microstructural aspect, including also hardness, for post exposed material in creep conditions. Overview includes also description of techniques applied for microstructural investigation of in-service components. Recommendations on utilisation of the correlation and for further development of this subject are presented. This document is in the first issues and is not yet to be considered as an exhaustive reference source; the volume 6 part x would be expanded and enhanced based on feedback from users.

ECCC Recommendations Volume 6 user feedback is encouraged and should be sent to:

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# **Residual Life Assessment and Microstructure**

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#### Foreword

"Components subjected to creep stress have a limited lifetime. Under the influence of temperature and stresses, the heat resistant materials react by creeping, whereby transformations, and segregations, precipitations occur in the structure of these materials."

As stated above by W. Arnswald, R. Blum, B. Neubauer, K.E. Poulsen in a document published on VGB Kraftwerkstechnik in July 1979, creep and material structure evolution are tightly related and according to this rule a large number of studies have been performed in order to relate microstructural investigation and service exposure or residual life.

This chapter is intended to give an overview of the existing applied (more or less frequently) techniques of microstructural investigation in order to assess residual life, and to state recommendations about their applicability.

#### 1 Microstructure

#### 1.1 Ferritic steels

As far as this type of material is concerned, the aspects mainly considered valid as an index of creep exposure are:

- Microstructural phase evolution
- Microvoid formation at grain boundaries (Neubauer etc.)
- Evolution of carbides (eutectoidic and ferritic fine precipitation)
- Interparticle distance

#### 1.1.1 Microstructural phase evolution

This aspect is mainly related to the effect of temperature and is not so strictly connected to load application. It is commonly accepted as a qualitative thermal degradation index shown through:

- tendency to pearlite/bainite spheroidisation
- coarsening of precipitates in the ferritic matrix and at grain boundaries,
- broadening of denuded zones (no precipitates) along grain boundaries.

This microstructural evolution is an index of an improved ductility of material but is often considered as a demonstration of overheating exposure.

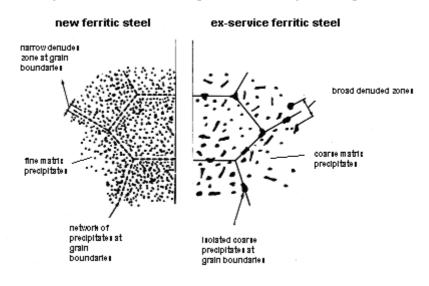
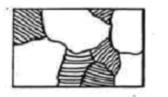


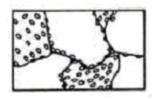
Figure 1 – Microstructural phase evolution generic aspect

The recently applied Italian new reference document for life extension of creep exposed components (ISPESL guidelines circ 48/03) introduces a classification defined as microstructure evolution based on a reference example relevant to carbon or low-alloy steel. The classification with 6 different levels is reported in figure 2.

Discussion on this subject is in course among technicians and laboratories, especially in relation to the possibility that some materials can be purchased according to international standard requirements and depending on the heat treatment applied (satisfactory of standard requirements) can present microstructure not strictly corresponding to level A but with some aspects of microstructure similar to other levels.



Level A - Ferrite and lamellar pearlite



Level D - Pearlite completely spheroidised but carbides still included in primary pearlitic grains



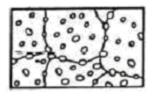
Level B - Initial spheroidisation and carbides precipitation at grain boundary



Level E - Carbides homogeneously distributed (no evidence of ferrite and pearlite original structure)



Level C - Intermediate level of spheroidisation, pearlite is partially globular but lamellar structure is still evident



Level F - Carbides homogeneously distributed and partially coarsened through coalescence

## 1.1.1.1 Coarsening coefficient of carbides

Studies have been performed from CEGB in late seventies early eighties on estimation of average particle size and remaining life fraction for steel as CrMoV. In particular the attention has been oriented to VC like carbide size and although different results have been obtained from different author in terms of absolute size value a good agreement is shown in terms of correlation (linear in log-log scales) of average particle size on temperature exposure (see figure 3) and on time maintaining at a certain temperature (see figure 4). The significant difference in terms of absolute size value is strongly influenced by statistical approach in estimating average particle size, accuracy in measurement at high magnification (10k to 100k) and sampling technique adopted (thin foil or carbon extractive replica).

Figure 2 – ISPESL classification for microstructure evolution of ferritic steels

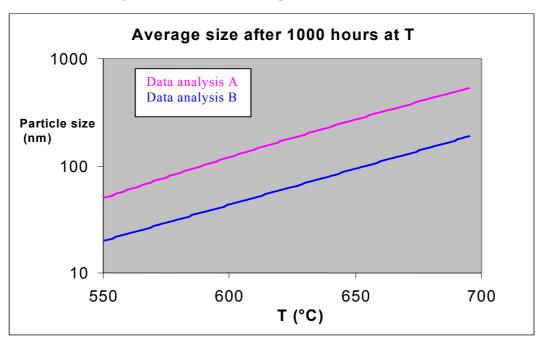


Figure 3 Particle size vs. temperature (for defined time)

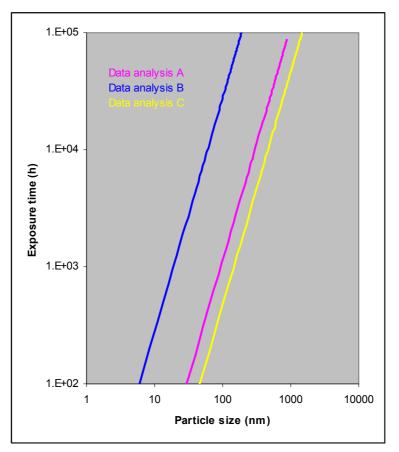


Figure 4 Temperature exposure time vs. particle size

#### 1.1.2 Microvoids formation at grain boundaries

The concept has been mainly studied and developed by TuV in the 70's and is commonly recognised and applied in all European countries with the Neubauer classification and derived methods.

The principle is based on the fact that creep evolution of heat resistant steels is related to the appearance of cavities some time before rupture. These cavities gradually form microcracks by interlinkage and at the end come to initiate the rupture. Size and density of the cavities increase as creep progresses from secondary to tertiary. Cavity size is largely dependant also on material type, however it is in the range of micron size (often also lower), therefore they are usually called "microvoids" or "micro-cavities". Due to their small size, they cannot be detected by conventional NDT techniques such as PT, UT, MT, RT, and metallographic investigation is required.

A first classification scale for creep damage has been proposed by Neubauer as reported in table 1.

Table 1 Neubauer schematic assessment of the microstructure

Grade of evolution	Structural assessment observed by metallography
0	Normal microstructure for new component
1	Normal microstructure for service conditions, incipient or
	advanced structural transformation or precipitation
2	Advanced creep load
	a) chain shaped, oriented carbides on grain boundaries
	b) isolated micropores (cavities on grain boundaries),
	irregularly distributed
	c) few micropores (cavities on grain boundaries) irregularly
	distributed
3	Incipient creep damage
	a) micropores (cavities on grain boundaries) orientation
	vertically to the direction of main stress
	b) grain boundary separation (length of one grain)
4	Advanced creep damage
	Microcracks (length of several grains) identifiable
5	Structural loosening (grain disintegration)
	Macrocracks (length of the millimetre range)

Evolution of Neubauer classification can be found in different European countries varying from a simplified approach to a more detailed classification.

A simplified approach of Neubauer damage classification can be found in the Italian ISPESL Guidelines 002 (reference document for creep classification applied in Italy) where the Neubauer classification is limited to the five different grades with an indicative representation of damage aspect as reported in table 2.

Table 2

ISPESL simplified Neubauer classification

Grade	Microstructure	Picture
0	New material	
1	Normal (no cavities)	

2	Presence of isolated microcavities	
3	Presence of directional oriented microcavities	
4	Presence of microcracks	
5	Presence of macrocracks	

A cavities density approach, always derived from Neubauer classification, has been applied in Sweden and other Northern European countries by SAQ as resumed in table 3.

Table 3SAQ modified Neubauer classification

Grade	Definition	Rating	Cavities density

1	No cavities			
2	Separated cavities	2b	Small extent 1-100 cavities/mm2	
		2c	Medium extent 100-1000 cavities/mm2	
		2d	Large extent 1000 and more cavities/mm2	
3	Strings of cavities	3b	Few strings and separated cavities of rating 2b	
		3c	Few string and separated cavities of rating 2c or 2d	
		3d	Numerous strings and separated cavities up to rating 2d	
4	Microcracks < 0.1 mm	4a	Reheat cracks	
		4b	Few microcracks and creep damage of rating 2b,	
			3b	
		4c	Few microcracks and creep damage of rating 2c,	
			2d, 3c,3d	
		4d	Numerous microcracks and creep damage of	
			rating 2c, 2d, 3c, 3d	
5	Macrocracks >0.1 mm	5a	Reheat cracks	
		5b	One macrocracks (<1 mm) and creep damage of	
			rating 2b, 3b, 4b	
		5c	Macrocracks (<5 mm) and creep damage of	
			rating 2c, 3c, 4c	
		5d	Macrocracks (<5 mm) and creep damage of	
			rating 2d, 3d, 4d or creep crack > 5mm	

Another revision of Neubauer classification is presented in the German VGB "Guidelines for the assessment of microstructure and damage development of creep exposed materials for pipes and boiler components" that is considered as one of the most updated reference document also in other European countries. The new proposed damage rating is reproduced in table 4.

VGB Guidelines classification				
Assessment class	Structural and damage conditions			
0	As received, without thermal service load			
1	Creep exposed, without cavities			
2a	Advanced creep exposure, isolated cavities			
2b	More advanced creep exposure, numerous cavities without preferred orientation			
3a	Creep damage, numerous orientated cavities			
3b	Advanced creep damage, chains of cavities and/or grain boundary separations			
4	Advanced creep damage, microcracks			
5	Large creep damage, macrocracks			

Table 4

In addition to the above revised classification one of the most important aspects of this document is the presentation of micrographs with real damages appearance for the 5 steels that guidelines cover (13CrMo4-4, 10CrMo9-10, 14MoV6-3, X20CrMoV12-1, X8CrNiNb16-13).

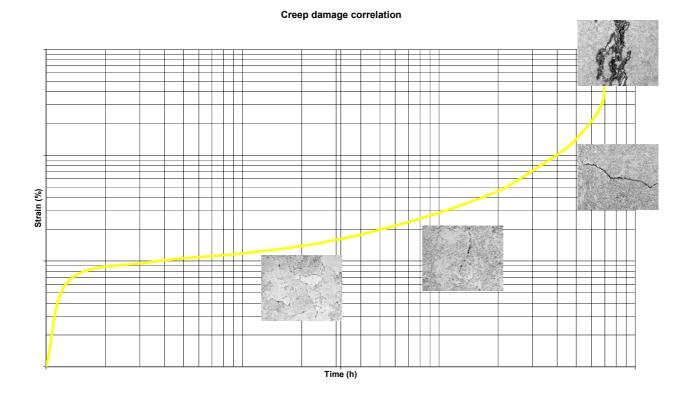
Cavitation derived parameter such as the A-parameter (number fraction of cavitated grain boundaries) have been defined and used in some studies related to low alloy ferritic steel for pipe and rotor. Successive development of studies indicates the utilisation of a modified A\*-parameter as more appropriate for rotor steel, calculated dividing A-parameter by grain size of prior austenitic grain.

#### **1.1.2.1** Correlation of Neubauer classification to creep evolution

A first indication of correlation of cavities presence to creep progress can be found in the same Neubauer et al . document of the first reported scale, where it is stated that "a noticeable cavity formation takes place at grain boundary at the end of secondary creep".

Further documents have been published representing graphs of creep curve (time exposure vs. creep strain) with the allocation of corresponding reference images of Neubauer grade damage. An example of this graphical representation is shown in figure 5, some other literature reported examples also indicates in graphs suggested actions to perform in plant. Although literature examples seem to show damage grade allocation on creep curve not completely congruent (probably due to the different creep curve represented) it can be assumed that grade 4 and 5 should be considered as representative of different stages of tertiary creep, while grade 3 should be considered as the transition point among secondary and tertiary and grade 2 should

be considered as representative of secondary creep (probably more or less advanced in according to successively introduced subclassification).



#### Figure 5 Example of Creep Damage Classification and Creep Curve

Another interesting approach for correlation of microcavities damage and residual life assessment is the method followed by some authors and based on the analysis of experimental results obtained for low alloy ferritic steels in heat affected zone of welded joint. Graph summarising the correlation of damage level and expended life fraction is presented in figure 6.

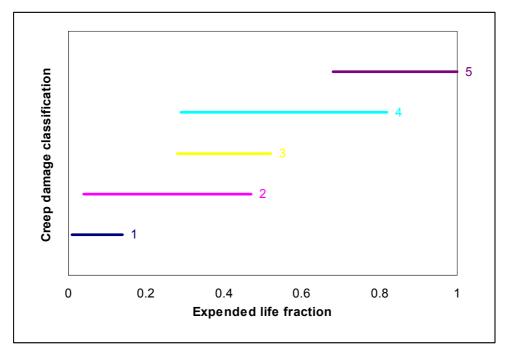


Figure 6 Creep Damage Classification and Expended Life Fraction

A critical review of these data and other in plant investigation is summarised by Sampietri et al that derived from the whole analysis the following table resuming a simplified correlation among damage grade classification and expended life fraction.

Damage	Expended	life
level	fraction	
1	0.181	
2	0.442	
3	0.691	
4	0.889	
5	1.000	

Table 5 Creep Damage and expended life fraction

Assumptions made for this correlation are quite conservative as declared by authors, but results are in agreement with other studies (Bendick et al.) stating that for 14MoV6-3 damage (first detection of cavities by light microscope) starts at an exhaustion grade of 50 to 60%.

Also derived cavitation parameter as previously mentioned A-parameter or A\*-parameter have been correlated to residual life assessment in some studies (see in figure 7 an example for rotor forging CrMoV steel) but no successive application of the concept to actual component case are known.

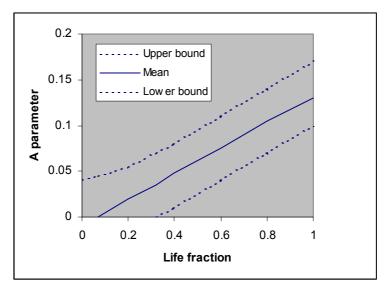


Figure 7 A-parameter and life fraction

#### 1.1.2.2 Theoretical studies on microcavities

As far as concern the theoretical aspect of cavities nucleation and growth some studies (Needham et al.) indicated that:

- for cavities nucleation
  - the number of cavities per unit grain boundary area increases approximately linearly with creep exposed time for low alloy Cr-Mo ferritic steel,
  - increasing the applied stress results in an increase in the number of cavities (exponential dependency of cavity nucleation on stress)
  - the number of cavities increases with increasing temperature for a given time and stress
  - the high purity cast materials have the lowest nucleation rate and highest rupture life
  - increase of the austenitising temperature results in an increase in nucleation rate and a decrease in rupture life
  - grain size and the type of second phase particles (MnS and Mo2C carbides) found on the prior austenitic grain affect the susceptibility of a material to cavity nucleation
  - cavities are associated with all carbide types although they form preferentially from M2C carbides
  - the presence of surface active elements as P and Sn makes cavity nucleation easier.

- For cavities growth
  - Same correlation as cavities nucleation with stress
  - Increasing of the austenitising temperature results in an increase in cavities growth rate
  - Two are the mechanisms that control cavities growth: diffusion growth and constraint growth, the first is dominating at intermediate and high stress level, the second one is dominating at the lower stresses.

Same authors proposed also an algorithm for remnant life prediction of low alloy ferritic steel based on the estimation of area fraction of grain boundary that is cavitated at time t, but it is not known a frequent application in industry of this kind of algorithm.

#### 1.1.3 Carbide evolution

Many significant studies have been conducted on the evolution of carbides present in steels due to creep exposure. Separation and coarsening of carbides is in general an index of material degradation due to creep exposure. A detailed description of the most commonly present carbides in low alloy steels with their composition and tendency to evolution with high temperature service is presented in an old paper of Woodhead et al.

The most important class of carbides that are present in low alloy creep resistant steels are:

<u>M<sub>3</sub>C</u>

Essentially cementite  $Fe_3C$  but often including other metallic elements (in particular Mn), the content of these other elements is controlled by element tendency to partitioning among ferrite matrix and affinity to carbon for carbide formation. This group of carbides has the same orthorombic structure of cementite.

 $\underline{M_7C_3}$ 

A family of chromium rich carbides with trigonal structure that can include also Mn , Mo and V.

#### $M_{23}C_6$

Typically Mo and W carbides for steels containing these elements, and Cr based carbides for other steels not containing Mo and W. These carbides have a significant tendency to include also Fe and Mn.

#### <u>M<sub>6</sub>C</u>

Essentially a ternary carbide of Fe and Mo or Fe and W with an appreciable solubility for other elements MC

Carbides with cubic structure mainly formed by V, Nb, and Ti. For vanadium carbides  $V_4C_3$  concentration is considered as pertinent to same group of VC. Mo and W carbides of the same concentration are generally considered separately due to the different kind of structure (hexagonal).

<u>M<sub>2</sub>C</u>

Usually a Mo rich carbide with face centred cubic structure.

Presence of different classes of carbides in steel is related to global composition and thermal process applied in manufacturing. Phase diagram with different parametric element concentration (binary or ternary) are available for studying equilibrium condition of carbide formation and evolution.

Very limited are the published information available (Benvenuti et al.) on the practical application of these studies in the field of maintenance investigation in industry. Some experiences are however known of trial monitoring of carbides evolution on plants through the utilisation of extractive replicas or low invasive sampling methods like those applied for small punch or indentation tests.

In this experiences of monitoring carbide evolution through extractive replica for a defined low ferritic steel, a simplified method of identification of main carbides classes ( $M_3C$ ,  $M_7C_3$ ,  $M_{23}C_6$  and  $M_6C$ ) and statistical distribution of carbides among these classes has been utilised as index of creep exposure effect on material. The analysis consists on the examination of precipitates collected through extractive replica with morphological evaluation of eutectoidic precipitates and fine particles dispersed in ferritic matrix, chemical composition determination of a defined number (statistically considered satisfactory) of carbides and representation of the detected carbides on ternary diagram where area corresponding to main classes of carbides are defined. Figure 8 shows an example of carbides ternary diagram for a low alloy ferritic steel with comparison of virgin and service exposed materials.

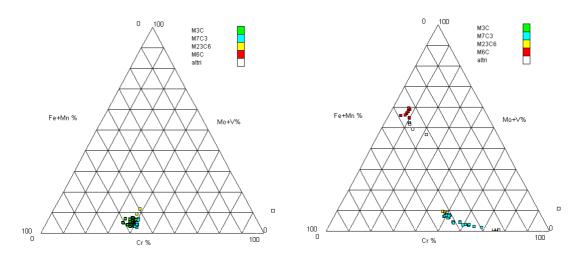


Figure 8 - Carbides ternary diagrams in virgin and service exposed low alloy ferritic steel

In the same study comparison of carbides composition in cold and hot locations of the same service exposed component have been made also on the basis of Cr/Fe ratio for  $M_{23}C_6$  and  $M_7C_3$  carbides and Mo/Fe ratio in  $M_6C$  carbides.

A large characterisation (Benvenuti et al.) of seventeen P22 steam pipes in virgin state showed that big variation in microstructure related parameters exist although the material was purchased according to the same standard specification. Among these parameter related to microstructure, carbides composition showed anyway less severe scatter.

The study of carbide composition evolution in P22 steam pipes service exposed, allowed to establish that most regular trend is the Cr enrichment in  $M_{23}C_6$  and a linear correlation has been estimated for Cr/Fe ratio (in  $M_{23}C_6$  carbides) with service time cubic root for the defined temperature of 540°C.

$$Cr/Fe = 0.32843 + 0.01653 t^{1/3}$$

The same authors in successive studies show a similar diagram of Cr/Fe ratio in  $M_{23}C_6$  carbides versus exposure time for P22 material at T=540°C. with a slightly modified equation.

$$Cr/Fe = 0.33362 + 0.01516 t^{1/3}$$

Figure 9 shows in graphical view the results of these studies summarising the two equations and the upper and lower bound referred to most refined second analysis with a confidence limit 90%.

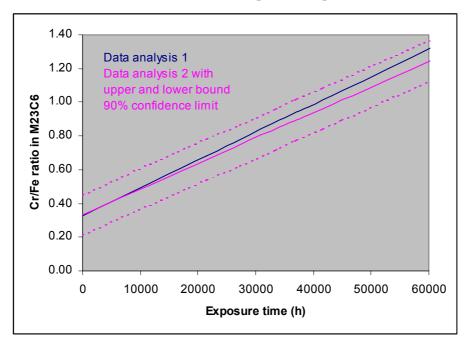


Figure 9 Cr/Fe ratio in  $M_{23}C_6$  carbides and temperature exposure time for P22 material

In the same study a graphical correlation of Cr/Fe ratio in  $M_{23}C_6$  carbides versus Larson-Miller parameter is presented for P91 steel showing the trends represented in figure 10 for gauge length and head of tested creep specimens.

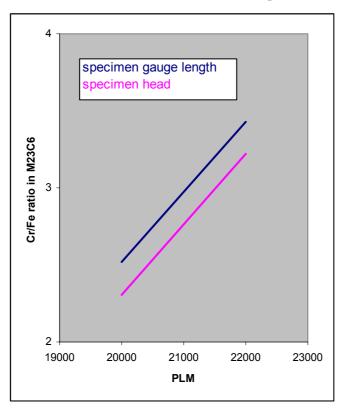


Figure 10 Cr/Fe ratio in M23C6 carbides and Larson-Miller parameter for P91 material

Alternatively to ternary diagrams, bidimensional diagram with carbide classification area in Mo/Mn ratio against Fe/Cr ratio plot have been used for carbide examination of CrMoV rotor forging steels (see generic example in figure 11); for this kind of steel a rough estimation for classification of MC and M<sub>2</sub>C in V and Mo carbides is often assumed based on V and Mo percentage (V>50% equal to VC, Mo>50% equal to M<sub>2</sub>C).

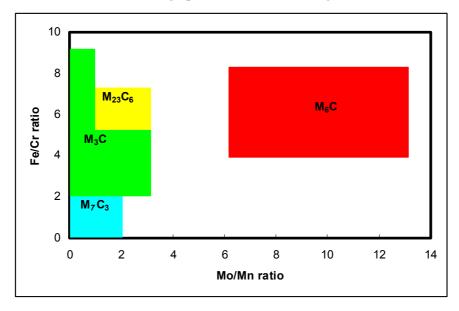


Figure 11 Carbide classification graph in bidimensional diagram for CrMoV rotor steel

Plots of Fe/Cr ratio (in carbides or in  $M_3C$  carbides) variation with time exposure to creep conditions are available in relation to study work for characterisation of ageing of CrMoV forging rotor steel (see figure 12 and 13) but it should be kept in mind that also satisfactory correlation as Fe/Cr ratio in  $M_3C$  carbides to cubic root of ageing time cannot be extrapolated to longer time as actual component service.

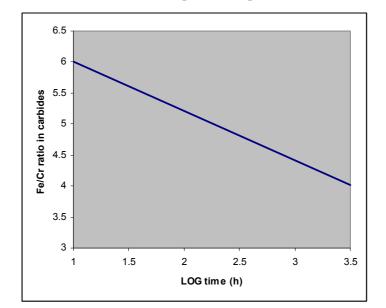


Figure 12 Cr/Fe ratio in carbides and temperature exposure time for CrMoV rotor steel

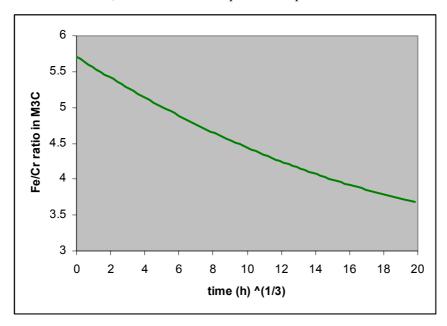


Figure 13 Cr/Fe ratio in M<sub>3</sub>C carbides and temperature exposure time for CrMoV rotor steel

It is in general well known and accepted that for low content Cr-Mo steel the increase of Mo content in precipitate either as Mo increase in eutectoidic carbide as  $M_3C$  or as  $M_6C$  formation either as Mo<sub>2</sub>C formation in fine ferritic dispersed precipitates is an index of creep exposure and microstructural degradation.

A general remark that is always stated as difficulty in correlation of microstructural evolution as carbides (or also as generic phase) to residual life assessment is the necessity to know the actual state of virgin material that for the same type of material can significantly vary from cast to cast or product to product.

#### 1.1.4 Interparticle distance

The concept is strictly related to phenomenon of carbide coarsening and grain boundary area denudated described in paragraph 2.1.1. As it can be seen from already presented figure 1 the microstructure transformation correspond in an interparticle distance growth that can be statistically (roughly or more precisely) estimated through metallogaphic investigation (especially with high magnification techniques).

Theoretical model for creep curve calculation based on interparticle distance and coarsening carbide have been studied at CEGB in early eighties and evaluated in comparison to experimental creep test data. The model are essentially based on changing of creep rate (tertiary creep) due to particle coarsening. Satisfactory results for model to experimental data fitting have been obtained for some of the models. Based on this model ENEL and TUD (Battaini et al.) started in late eighties a characterisation of interparticle distance measured with TEM in thin foils prepared from virgin and ex-service (temperature accelerated creep rupture tests) steam pipe of 12% Cr steel. Two main family of carbides have been considered based on mean (center of normal distribution) dimension and consequently two interparticle distances are estimated as

$$ID = (1/N_A)^{1/2}$$

where ID = interparticle distance and  $N_A$  is the number of carbides per  $\mu m^2$ .

The results presented in figures 14 and 15 are considered by the authors satisfactory in terms of trend, especially for the smallest size particles assimilated to small carbides precipitating during service and thus with an interparticle distance decreasing. A large scatter is in any case observed on virgin material and a lot of uncertainties contribution should be kept in mind for results analysis.

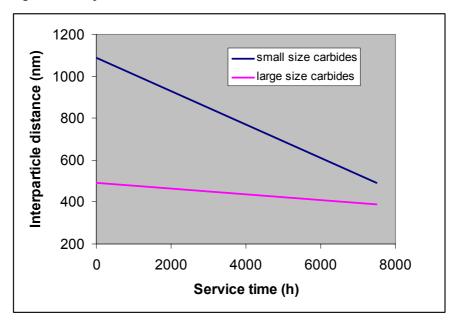


Figure 14 Inteparticle distance and service time for base material X20 steel

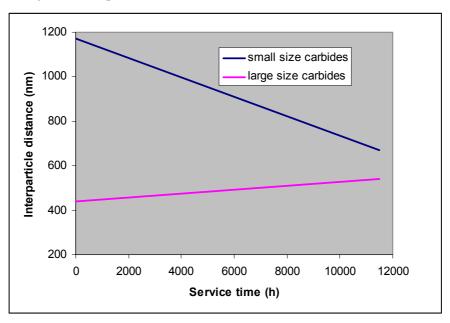


Figure 15 Inteparticle distance and service time for welded X20 steel

A successive interparticle distance evaluation (Benvenuti et al.) in high chromium (9-12%) steels show a generic interparticle distance increase with creep exposure deducted from comparison of creep specimen material in gauge length and specimen grip.

#### 1.2 Other materials

As far as concern the correlation of microstructure and life assessment in non ferrous material the info available are very limited due to the fact that in some field of application the materials are relatively new products and often still in evolution.

In the field of Nickel based alloys that are largely applied in aeronautical industry and more recently are becoming very widespread also in the power plant engineering, it is well known that the  $\gamma$ ' particles size and shape are the reference parameters used by manufacturers for detecting material degradation due to service at high temperature.

All manufacturers of gas turbines use their own algorithm based on plant service data and non destructive examination results in order to define maintenance programme for plant, most of them often perform periodical inspection with removal of some parts that are used for destructive examination including microstructural examination. More recently also the application of morphological replica is becoming more commonly adopted for microstructural investigation of in service material evolution.

#### 2 Techniques applied for investigation on in-service components

The most of the studies done on correlation of material microstructure and creep exposure have been performed by means of metallographic specimen obtained from creep specimen after tests. A large improvement of the investigation on actual in service components has been obtained with the replica technique.

The large success of this technique is correlated to the fact that replica can be assumed as a non destructive technique or at least very low invasive technique, and this is the first requirement for an investigation applied for residual life evaluation not affecting the same parameters.

Due to the criticity of weld repair the withdrawal of specimen from in service component is not recommended as approach for microstructural investigation.

The recently developed technique of small punch sampling that can be assumed as low invasive technique can be an alternative to replica especially for the wider spectrum of microstructural information that can be obtained from a massive specimen.

#### 2.1.1 Replica investigation

The technique is essentially the application of metallographic specimen preparation (grinding, polishing, and etching) to a limited area of the component that is required to investigate and the reproduction of the so prepared surface on a thin foil of polymeric material.

If microstructure evolution is the target of replica examination, the removal of a thin layer of surface material (about 0.3-0.5 mm thickness reduction) is recommended so to avoid the external layer of decarburised material.

The reproduction of prepared surface on plastic material is achieved by the softening of polymeric thin foil with adequate solvent followed by hardening of the same plastic material due to solvent evaporation.

Replica can be observed with the utilisation of an optical microscope, where standard magnification ranges from 50 to 500 x ( $1000 \times magnification$  can be achieved but often difficulties can be matched on focus optimisation of replica surface).

If properly prepared with application of a conductive support and gold thin foil application on reproduced surface, replica can be observed also with scanning electron microscope achieving the possibility to observe reproduced microstructure aspect with higher magnification. Replica examination on SEM should be performed carefully in order to take care that electron beam doesn't produce any excessive heating of the surface with possible consequent deformation of plastic material underlaying.

Major limitation of the replica is the necessity to reduce the examined area to a very limited extension if compared to actual component dimension. The preliminary correct identification of the position in

component that should be replicated is thus a very critical phase, in order to be sure that the replica examined is representative of the most probabilistic damaged area.

Location of area to be examined by means of replica is usually based on the analysis of plant operating condition correlated to component design dimensions and material type. Pre-replica measurement of actual thickness component parts by means of UT is also recommended in order to detect eventually existing defined area where local corrosion or other actions can cause thickness reduction and consequently higher stresses with more probabilistic creep damage.

Replica can be prepared mainly on the external surface of components, industrial experience of creep damages or failure has however showed that in the most cases creep damage is generally most evident in this area although some cases of internal surface creep damages have been detected.

As already stated although replica is a low invasive technique a limited reduction of thickness is necessary in replica preparation and this represents thus also a limit for application.

#### 2.1.2 Extractive replica

Following the same procedure of surface preparation used for morphological replica, it is possible to take from material also extractive replica. Applying a specific etching of the prepared surface oriented to remove the metal matrix and separate carbides and other precipitates, it is then possible by means of specific time hardening polymeric resins to remove from material surface the precipitates that can be later analysed with electron microscope (scanning and transmission). Alternative techniques for carbon extraction replica are available (not using polymeric resins) but mainly applicable on laboratory specimens (from mechanical or metallographic testing)

All limitations previously described for morphological replica are in principle extended also to extractive replica, in addition to these it is very critical for the extractive replica to apply the proper etching that enables to remove the metal matrix without any perturbation on carbides and other precipitates. The necessity to examine this replica through electron microscope is an additional limitation due to the difficulty to evaluate on plant the good quality of produced replica. Preliminary examination by light microscope can give just a rough idea of replica quality. Examination of extractive replica with scanning electron microscope doesn't require any particular additional preparation except than taking care of polymeric resin conductivity. In the case of transmission electron microscope, the replica sampled in plant should then be prepared for final examination by transferring the carbides and precipitates from resins (dissolved) to a thin layer of graphitic carbon. The examination of these replica in transmission electron microscope where the thin samples that can by analysed are small square foils of a few millimetres size, enhances more than in the previous case the problems related to representativity of sampling area for component.

#### 3 Hardness

Since the first study developed for the assessment of residual life in high temperature serviced components, attention has been paid to hardness value in order to find numerical correlation of the parameter with service and expected time of the component. In the research studies hardness has been measured through standard instruments as Vickers or Brinell indenter based, but for in plant direct monitoring of serviced component some instruments based on energy absorption during impact or indentation combined with ultrasonic measurements are available as correlation for standard unit conversion. Due to this fact it is thus possible to obtain also results directly from in-service components. Actually at the moment although hardness measurement in plant is a commonly applied technique during maintenance inspection (especially in combination with replica) the most of the published studies are based on laboratory measurement made on test specimens with standard hardness measurement techniques.

Gooch et al. in the already mentioned study relevant to remanent life assessment of rotor forgings evaluated in addition to microstructural aspect also correlation to hardness remarking that:

- hardness changes with ageing is time, temperature and stress dependent,
- although the confidence limits are very wide, a relationship of hardness with Larson-Miller can be described,
- hardness decreases with exposure to creep conditions service,
- softening under applied stress is greater than in the case of thermal ageing without stress,
- softening under applied stress is proportional to stress,
- hardness evaluation can be used for estimating true operating temperature but statistical confidence limits suggests to evaluate results generated from a database carefully case by case,
- hardness evaluation is not a practicable means of estimating the creep rupture strength and ductility of a
  previously uncharacterised rotor.

The general proposed equation for hardness changes and life assessment correlation can be espressed as:

$$H_v/H_{v0} = a \bullet PLM + b \bullet PLM^2 + c$$

where Larson-Miller parameter is calculated by

$$PLM = T \bullet (A + Log t)$$

"t" is time in hours

while a, b, c and A are parameters depending on the assumed database in the following ranges

<sup>&</sup>quot;T" is temperature in °K

"a" from 1.25•10<sup>-3</sup> to 1.6•10<sup>-3</sup>

"b" from 3.91•10<sup>-8</sup> to 4.97•10<sup>-8</sup>

"c" from -8.94 to -11.99

"A" from 7.8 to 15.82 as best fit values or 20 as conventional value.

Graphical representation of the correlation for various databases and with a unified analysis based on conventional value of A in Larson-Miller parameter is presented in figure 16 with lower and upper bound limit curves for a 95% confidence limit.

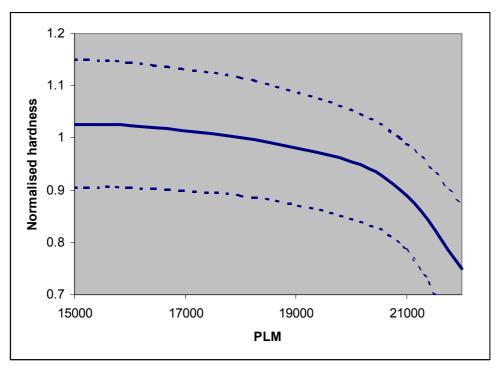


Figure 16 Hardness and creep exposure through Larson Miller parameter

#### 4 Comments

VGB Guideline for the assessment of microstructure and damage development of creep exposed materials for pipes and boiler components is currently considered in large part of Europe as the most updated reference document for microstructure and residual life assessment correlation. The still in progress status of the art on this subject is however confirmed by the following extracts from VGB guidelines.

"The knowledge of the structure and degree of damage could be essential for the assessment of residual service life and a damage analysis respectively. It should however be pointed out that the above mentioned knowledge alone does not allow a prediction of the residual service life ... it is therefore understandable, that the presence of cavities after 40000 hours requires different and short term measures as the same degree of damage after 200000 h."

It's important to remark that all other (but cavities) microstructural observed parameters need to be evaluated on the basis of the as received material correspondent status in order to avoid any misleading deduction from generic or recommended microstructure variation not actually correlated to service exposure.

## **5** Final recommendations

Evaluation of microstructural evolution in exposed to service materials is a key tool for a correct evaluation of material status and allowable service extension. A reliable life assessment should be made not only by means of microstructural inspection but it's preferable that together with other inspections the same is included. Among the different aspects that can be observed the evaluation of microcavitation presence and creep damage evolution seems to be, for the widely applied ferritic low alloyed steels, the most consolidated approach and the evolution of Neubauer classification with subclassification in particular of grade 2 and 3 (that corresponds to the longest part of a component life) should be continued and encouraged. At present it seems that singular countries are working independently in this field and an essay for a common effort of a unified revision of Neubauer classification should be welcomed.

For every other microstructural aspect (except microcavities), whatever is the monitored one it is very important that the evaluation is made by comparison with the original (virgin material) status.

From the view point of the techniques applied for monitoring microstructure on in-service components, replica are surely a consolidated and reliable technique, attention should be paid in any case also to alternative low invasive technique for sampling as the ones applied for small punch or impression creep specimen preparation.

## 6 Bibliography

- Microstructural examination of several commercial <sup>1</sup>/<sub>2</sub>Cr<sup>1</sup>/<sub>2</sub>Mo<sup>1</sup>/<sub>4</sub>V casts in the as received and service exposed conditions (K.R.Williams) CEGB research laboratory report 1979
- 2) Note on VC coarsening in ferritic steels (J.M.Silcock) CEGB confidential note 1981
- 3) VC coarsening in <sup>1</sup>/<sub>2</sub>Cr<sup>1</sup>/<sub>2</sub>Mo<sup>1</sup>/<sub>4</sub>V steel (T.P.Crispin-J.M.Silcock) CEGB confidential note 1981
- Metallographic examination of <sup>1</sup>/<sub>2</sub>Cr<sup>1</sup>/<sub>2</sub>Mo<sup>1</sup>/<sub>4</sub>V creep specimens from ERA project 2021 (M.Askins-M.S.Shammas-J.M.Silcock) CEGB confidential note 1982
- 5) Summary of calculations of creep curves from coarsening equations (J.M.Brear-S.Bruce-B.J.Cane-J.M.Silcock) CEGB research internal document 1982
- Metallographic techniques for residual life assessment of 1CrMoV rotor forging (J.Maguire-D.J.Gooch) CEGB unrestriced but not published document 1988
- Techniques for remanent life assessment of 1CrMoV rotor forging Cavitation assessment (G.Lyell-A.Strang-D.J.Gooch-J.Maguire-S.M.Beech-W.R.Callender-C.E.Raynor) National Power Confidential note 1990
- Techniques for remanent life assessment of 1CrMoV rotor forging Hardness evaluation (G.Lyell-A.Strang-D.J.Gooch-J.Maguire-S.M.Beech-W.R.Callender-C.E.Raynor) National Power Confidential note 1990
- The effect of oxidation specimen size on the creep rupture of low alloy ferritic steels (D.J.gooch-M.S.Shammas) National Power confidential note 1990
- 10) Methods for remaining life prediction of steam pipes and boiler tubes (Castani-D'Angelo-Michelizzi / ENEL) presented at seminar on "Remanent life of high-temperature plants" London UK October 1983
- 11) Application of image analysis techniques and scanning electron microscopy to the study of cavitation in creep resistant materials (Anelli-Faccenda-Franzoni / Centro sperimentale Metallurgico) Elsevier 1981
- 12) Microstructure and creep crack growth behaviour in low alloy steels (Airoldi-Bianchi-D'Angelo / ENEL) presented at International Symposium on "Microstructure and mechanical behaviour of materials" Xi'an China October 1985
- 13) Reliability of quantitative metallography approach for material evolution assessment (D'Angelo-Regis / ENEL; Franzoni /CSM; Rossi/Dalmine) proceedings of Second International Conference on "Creep and fracture of engineering materials and structures" Swansea UK September 1984
- 14) How the microstructure affects the high-temperature properties of low-alloy steels strengthened with vanadium carbide (Prnka /FMRI; Foldyna-Sobotka/RIVS) presented at International Conference "Properties of creep resistant steels" Dusseldorf may 1972

- 15) Carbide changes in Cr-Mo-V steels in the course of long service (Bologov-Lanskaya-Bulanov-Tolstov) published by Thermal Engineering 1981
- 16) Prediction of service characteristics of steam pipes made of 12Cr1MoV steel after their design service life (Bologov-Lanskaya-Novitskaya-Rushchits) published by Thermal Engineering 1979
- 17) Macroscopic measurements of creep damage in metals (Brathe/University of Technology Goteborg) published by Scandinavian Journal of Metallurgy 1978
- 18) Role of carbides in low-alloy creep resisting steels (Woodhead-Quarrell / University of Sheffield) published on Journal of the iron and steel institute 1965
- 19) Structural integrity assessments and lifetime predictions of operating thermal power pipelines (Billi-D'Angelo-Maciga-Livraghi /ENEL)
- 20) Application of Structural Surface Examination for the testing of power plant components subjected to creep stress (Arnswald/VKR;-Blum/FV-Neubauer/TUV-Poulsen/JTI ) reprinted from VGB Kraftwerkstechnik July 1979
- 21) Determinazione del periodo di ulteriore esercibilità prima del successivo controllo per un componente soggetto a scorrimento viscoso (Fossati-Sampietri/ CESI) seminar in Siracusa I 2001
- 22) Criteria for prolonging the safe operation of structures through the assessment of the onset of creep damage using non-destructive metallographic measurements (Neubauer/TUV) reprint from "Creep and fracture of engineering materials and structures" march 1981
- 23) R. Viswanathan Damage mechanisms and life assessment of high temperature components ASM International 1989
- 24) Metallographic techniques for estimation of residual creep life (Roberts-Ellis-Henry/CE; Askins-Shammas-Townsend/CEGB; Cane/ERA; Viswanathan/EPRI)
- 25) Remanent life assessment of process plant (Cane-Bell/ERA)
- 26) Residual life techniques for plant life extension (Viswanathan-Dooley-Manfred/EPRI)
- 27) Creep life assessment techniques for fossil power plant boiler pressure parts (Viswanathan-Dooley/EPRI)
- 28) Experiences with remanent life evaluation testing (De Witte-Stubbe/Laborelec)
- 29) Cavitation and rupture life prediction in low alloy ferritic steels (Needham-Gladman/BSC)
- 30) Microstructural changes in long term aged steels (A.Benvenuti-N.Ricci/CISE D.D'Angelo-G.Fedeli/ENEL) Microstructures and mechanical properties of ageing material –The mineral, metals, & materials society 1993
- 31) Evaluation of microstructural parameters for the characterisation of 2½ Cr 1Mo steel operating at elevated temperature (A.Benvenuti-N.Ricci/CISE G.Fedeli/ENEL) Proceeding of Swansea conference 1990

- 32) Interparticle distance evolution on steam pipes 12%Cr steel during power plants' service time (P.Battaini-D.D'Angelo-G.Marino/ENEL J.Hald/TUD Lyngby) Proceeding of Swansea conference 1990
- 33) Evoluzione microstrutturale di acciai 9-12%Cr sottoposti a scorrimento viscoso (A.Benvenuti-N.Ricci/CISE P.Bontempi/ENEL) La metallurgia italiana 1995
- 34) Assessment of material thermal history in elevated temperature components (A.Benvenuti-S.Corti-N.Ricci/CISE P.Bontempi/ENEL) Material characterisation Elsevier science 1996
- 35) Reliability problems in methods of residual creep life assessment (L.Kloc-V.Sklenicka/IPM Brno) Proceedings of 10<sup>th</sup> conference on creep & fracture of engineering materials and structure Prague 2001)
- 36) Creep properties affected by morphology of MX in high Cr ferritic steels (K.Yamad-S.Muneki-F.Abe/NMIR M.Igarashi/Sumitomo) Proceeding of Swansea conference 2001)
- 37) Microstructure change during creep deformation of modified 9Cr1Mo steel (F.Yoshida-D.Terada-H.Nakashima/Kyushu university H.Hayakawa/Kyushu electric power S.Zaefferer/TU Darmstad) Proceedings of Swansea conference 2001
- 38) The use of light and electron microscope investigations to characterise the creep behaviour of welded joints in modern power station materials (E.Letofsky-H.Cerjak-I.Papst-P.Warbichler/Graz University) Proceedings of Swansea conference 2001
- 39) Linee guida per la valutazione di vita residua per componenti in regime di scorrimento viscoso ISPESL (Istituto Superiore per la Prevenzione e la Sicurezza del Lavoro) Circ. 48/03
- 40) Critical stress for transition of creep deformation behaviour in virgin and long serviced material of 2.25Cr-1Mo steels (T.Ohba, E.Baba, K.Kimura, F.Abe, K.Yagi/NIMS; I Nonaka/IHI) Proceedings of the 6<sup>th</sup> international Charles Parsons turbine conference
- Reliability assessment of 2.25Cr-1Mo steel after prolonged exposure in plant (T.Ohba, E.Baba, F. Abe/NIMS; I.Nonaka IHI) 27<sup>th</sup> MPA-Seminar 2001
- 42) A cost effective maintenance approach for a steam system subject to creep conditions (H.J. Bouwman,A. Helkoop) International Conference "Life Assessment and Extension" 13-15 June 1988
- 43) Optimisation of lifetime of creep loaded structures Results of projects of the Netherlands Institute of Welding (C.F. Etienne, J. Prij) International Conference "Life Assessment and Extension" 13-15 June 1988
- 44) Utility survey and inspection for life assessment of elevated temperature headers (Roberts-Ellis/CE; Viswanathan/EPRI) American Power conference