## CRACK GROWTH BEHAVIOUR OF P92 AT TEMPERATURES ABOVE 500°C IN DIFFERENT ATMOSPHERES

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#### ABSTRACT

The ferritic/martensitic steel P 92 is one of the best creep resistant material at temperatures about 600°C. This material may replace steels like the 12 % Cr-steel X20CrMoV121 for application temperatures above 550°C. Thick walled components are more und more designed with fracture mechanic assessments. This paper provides a set of experimental results with fracture mechanic specimens of P 92 concerning the impact of proof temperatures, proof atmospheres and structural stability (over ageing).

## **KEYWORDS**

Creep resistant steel P 92, proof temperatures (RT, 500 to 600°C), proof atmospheres (vacuum, air, simulated steam).

#### **INTRODUCTION**

Safety relevant thick walled components in power plants, such as turbine rotors, steam-pipes and pressure vessels for high temperature operational temperatures are dimensioned and designed by using time dependent properties, ascertained by safety margins, by these procedures the components may be protected against plastic and creep deformations (compare AD[1] or TRD[2] guidelines, ASME CC N47[3] or RCC-MR[4]). These design procedures help to guarantee the integrity of a component through the operational life, postulating that the component has been made of a continuous without any flaw. But all the components might contain pre-existing flaws, like metallurgical inhomogeneity, which are preferential local sites for the initiation of voids and microcracks. During all the operational history, these structural inhomogeneity may grow and coalesce and may finally leading to unexpected rupture or failure of the component. Therefore failure analyses become more and more acceptable for the design and for the estimation of the allowable operational life. In most cases, the initiation of a crack at a structural flaw is introduced by repeated operational loads (fatigue), the developed crack may additionally grow slowly under steady state loadings (creep crack growth). By the growth and/or coalescence of microcracks the critical defect size may be resulted which can be lead to a spontaneous (say catastrophic) failure of the component.

Therefore information on crack growth behaviour under all kind of loading conditions are needed to estimate the long term structural strength of the component. The knowledge of high temperature fracture mechanic concepts is required. This requirement can be satisfied by experimental work with fracture mechanic specimens of the alloy exposed at working temperatures under cyclic (fatigue), steady state (creep) loadings and/or under combined loading cycles (e.g. fatigue with hold time) in the working fluids.

The experimental examinations in open air represent the events initiated by inhomogeneity close to the surface of a component, the examinations in vacuum should help to understand what could happened in the internal of a component. Examinations in simulated working fluids, (like  $Ar+50\%H_2O$ ) may additional help to introduced the effect of the fluid/metal interactions on the crack growth behaviour.

# 1. EXPERIMENTAL DETAILS

Significant increases in creep strength have been achieved during the last twenty years by the development of advanced 9 to 12 % Cr steels. The steel named P 92 provides the highest creep resistance in the temperature range about 600°C. The basic strengthening mechanisms and the long term stability are understood [5]. For temperature above 550°C the questions concerning steam oxidation resistance is still under considerations. But there are very limited results concerning the crack growth behaviour of this steel.

Compact tension specimens (1/2" CT) were manufactured from a wrought ring of a thick wall pipe, the crack branch direction was in the rolling direction of the semifinished product (direction T-L according ASTM E 399) as shown in Fig. 1.



Fig. 1: Location of take samples and dimensions of a CT-specimens

The test specimen has been sidewise grooved with a leading notch in the ligament. P92 was heat treated by austenitization at 1070°C/2h and tempering at 775°C/2h. The as-received

microstructure is presented in Fig. 3. The nominal chemical composition of P92 and two materials for comparison are given in table 1.

Material	С	Si	Mn	Р	s	Cr	Мо	W	v	Nb	В	N	Ni	Al	Ti
P92	0.115	0.028	0.46	n.b	0.006	8.85	0.42	1.85	0.2	0.065	0.008	0.042	0.068	0.02	-
X22CrMoV12 1	0.24	0.38	0.53	0.031	0.008	12.0	1.21	-	0.31	-	< 0.02	0.034	0.63	0.006	-
Inconel 706	0.014	< 0.02	< 0.02	< 0.02	< 0.02	16.0	-	-	-	3.1	< 0.02	0.0014	43.0	0.18	1.69

Heat treatments P92 X22CrMo V12 1 Inconel 706

austentization:  $1070^{\circ}$ C / 2h, air-cooled to RT, annealing  $775^{\circ}$ C/2h austentization:  $980^{\circ}$ C / 2h, air-cooled to RT, annealing  $690^{\circ}$ C / 2h annealing:  $980^{\circ}$ C / 2h, air-cooled, aged:  $720^{\circ}$ C /  $8h_{-}^{1K/min}$   $620^{\circ}$ C / 8h / AC

Table 1: Nominal chemical composition of P92, X22, Inconel 706 and heat treatments

As demonstrated by tensile tests at 600°C the stress strain behaviour of both of the ferritic steels is very similar, where as the stress strain behaviour of the Ni(Fe)-base alloy Inconel 706 is of a very other kind (Fig. 2).



Fig. 2: Tensile test at 600°C of different materials



Fig. 3: Microstructure (as received) of P92

All CT specimens were prepared according the ASTM standard E 647. The notched CTspecimens were pre-cracked at room temperature by fatigue with a frequency of 85-90Hz to obtain a sharp crack tip. The crack growth rate examinations were carried out under load control mode in servo-hydraulic or electro-mechanical test facilities. The creep fatigue crack growth test (CFCG) were performed with trapezoidal wave (compare Fig. 4) with loading and unloading times of 1s and a dwell duration of t=300s = 5 min. The R-value was kept constant equal 0.1. The crack length was monitored online by means of electrical potential technique (DC). At the end of each test, the potential drop signal was calibrated with the final crack length of the fractured path on the broken specimens measured optically.



Fig. 4: Schematic illustrations of the used loading cycles

The heating system was either by an induction coil or resistance furnace (compare details in [6]). The experiments carried out in vacuum used a vacuum chamber with a pressure as low as  $2x10^{-5}$  mbar. For the simulation of the influence of steam, a heated recipient is used containing a mixture of Ar+50% H<sub>2</sub>O (as steam: experimental details compare [6]).

To prove the influence of the proof atmosphere first estimation can be made with CER – tests (Constant Extension strain Rate) with circumferential notched rod specimens help to evaluate very simple the susceptibility of metallic materials to environmentally assisted cracking.

# 2. RESULTS

The main emphasis has been put on the impact of test atmosphere on crack growth behaviour.

# 2.1 EXAMPLES FOR THE IMPACT OF PROOF ATMOSPHERES

In Fig. 5 the stress/strain curves of P92 of tensile test with notched CER-specimens and a constant strain rate of  $\dot{\varepsilon} = 10^{-5} mms^{-1}$  in open air, simulated steam (Ar+50%H<sub>2</sub>O) and vacuum (10<sup>-5</sup> mbar) are given. The results in air and simulated steam are very similar, where as those in vacuum provided a shorter life time and a smaller yield point.

These results provide the impression that the crack propagation in vacuum may be faster compared to oxidizing atmospheres.



Fig. 5 : CER-tests at 550°C

With fracture mechanic examinations the influences of proof temperatures, proof atmospheres and of over ageing are tested.

It is known from the LEFM-theory [7], that the crack propagation under cyclic loading (fatigue) can be divided in three stages, the first one is increasing crack length with continuously reduced crack growth rate, the second is a steady state crack growth with fairly constant crack growth rate, the Paris-Erdogan crack growth and the third, mostly not extensively observed, is the continuous increase of the crack length up to structural failure.

The comparison of results of FCG experiments in the different atmospheres (vacuum and open air) is mainly concerned with the first and second stage of crack growth. At RT the FCG

seemed to be easier in vacuum compared to that in open air (Fig. 6). Fig. 7 compares the behaviour at 550°C under pure cyclic loading (FCG).



FCG tests at RT

Fig. 6: Comparison of FCG tests at RT



FCG tests at 550°C

**Fig. 7:** Comparison of FCG at 550°C in vacuum and on open air  $(da/dN=f(\Delta K_I))$ 



CCG test at 550°C

**Fig. 8:** Comparison of CCG at 550°C in vacuum and on open air ( $F_0=7$  kN) and in simulated steam ( $F_0=6.5$  kN)

Fig. 8 compares the CCG-behaviour of P92 at 550°C in air, in vacuum and simulated steam. At that proof temperature the crack growth in air started with low threshold  $K_{I0}$  compared to the behaviour in vacuum, whereas under static loading the crack growth started earlier in vacuum (Fig. 9) compared to air. The results are given according to an assessment with the  $K_{I}$ -method, da/dt=f( $K_{I}$ ). Using simulated steam atmosphere for testing, the accelerated oxidation in the crack tip in this environment seemed to retard again the beginning of crack growth (Fig. 8 and 9).



Fig. 9: Fracture surfaces without (tested in vacuum) and with oxidation layer (tested in  $Ar+50\%H_2O$ )

The micrograph appearance of the crack tip (Fig. 9a and 9b) demonstrats that the growing oxide layer in the crack under static loading (CCG) retards the crack growth in steam compared to that in inert atmosphere (vacuum).

It was assumed before this experimental work that fatigue crack growth experiments with hold time should be demonstrate more clearly the influence of the test atmosphere, but it was found, that the effect of oxidation processes in the crack tip area under cyclic and static loading is reversed for this kind of creep resistant, highly oxidation sensitive material.



Fig. 10a)



Fig. 10b)

Fig. 10: Comparison of CFCG-behaviour of P92 in different atmospheres at different proof temperatures

Fig. 10 a), b) compares the crack behaviour da/dN= $f(\Delta K_I)$  for 500 and 600°C in the atmospheres open air, simulated steam and vacuum. Evaluating the experimental results as a fatigue crack growth experiment (da/dN =  $f(\Delta K_I)$ ), there is no difference to be observed in the Paris-Erdogan state. Due to oxidation during the dwell period, the enhanced propagation in vacuum due to pure cyclic loading seemed to be retarded by the formation of oxide layers in the crack branches. This principal observation is also true for the 550°C proof temperature.

# 2.2 EXAMPLE FOR THE IMPACT OF PROOF TEMPERATURE

There is a clear observation that increasing the test-temperature from 500 to 600°C, the tendency for crack growth increases (compare Fig. 11,  $da/dN = f(\Delta K_I)$  in simulated steam).



**Fig. 11**:  $da/dN = f(\Delta K_I)$  in simulated steam

Metallographic examinations of the area around the crack branches (Fig. 12a),b),c)) indicated that in this test atmosphere, the number and extension (length) of secondary (sidewise) micro-cracks increased with increasing test temperature.



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Fig.12: Microstructure of crack branches of the specimens tested according Fig. 11
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# 2.3 INFLUENCE OF MICROSTRUCTURE DUE TO OVERAGEING

The stability of the microstructure has been proved by 3000h overageing at 650, 700 and 800°C in air. The biggest change in the microstucture is to be expected after the 800°C treatment. Nevertheless the Paris-Erdogan behaviour at RT under fatigue loading is fairly similar. The threshold value seemed to be influenced by the oxidation products within the crack tip. At a proof temperature of 550°C the overageing at 650°C (3000h) does not change the threshold behaviour and Paris-Erdogan growth but with increasing aging temperature the threshold behaviour is shifted to higher values (Fig. 13).



Fig. 13: FCG test at 550°C in air

## **3. DISCUSSION**

The use of the new creep resistant steel P 92 is sometimes restricted by the poor oxidation resistance of this 9-10% Cr steel compared to the 12% Cr-steels. In the temperature range above 550°C the oxidation rate of this steel becomes increasingly higher [8]. Fracture mechanic tests, where load are cycled at elevated temperature are identified as fatigue crack growth experiments and are sensitively depending on frequency and R-ratios. The analyses of the crack growth behaviour in this case can generally be dealt with a LEFM-approach to evaluate the function  $da/dN = f(\Delta K_I)$ . The failure due to fatigue cracking at frequencies high enough (>0,1 Hz) is usually identified by transgranular failure mode. At low frequencies and in the range of loading cycles in which time dependent reactions may occur, e.g. fatigue creep crack growth experiments (CFCG), that means fatigue with hold time, the failure mode may change and atmosphere/metal reaction becomes of importance. At static loading (creep crack growth experiments, CCG) the time dependent reaction like oxidation and creep behaviour becomes of main importance.

At the beginning of these documented crack growth experiments, it was supposed that with this material the influence of atmosphere should be clearly to be documented by creep crack growth and combined loading experiments. From Ni-base alloys the SAGBO-Effect (Stress Accelerated Grain Boundary Oxidation) is very well known [9]. The reaction along the grain boundaries in these austenitic materials have a great often negative effect on time dependent crack growth behaviour. It was one idea to proof this phenomena for the ferrtic/martensitic steels. At 550°C the observed crack growth behaviour under cyclic loading (FCG) can be derived as da/dN =  $f(\Delta K_I)$ . The crack growth rate in air under this cyclic loading is higher than in vacuum. Although the crack propagation path of the main crack in both cases is transgranular the number of secondary small microcracks increase after the test in open air.

Under static loading (CCG) the samples of the results with P 92 documented a totally inverse tendency. The crack growth rate in vacuum is much higher compared to that observed in air and in the oxidizing atmosphere: simulated steam. The time dependant oxidation within the branches of the crack and of the crack tip retard the crack growth. The results of CFCG experiments documented the overlapping of both observations. For the temperature range of 500 to 600°C the results may be summarised by schematic evaluation given in Fig. 14.



**Fig. 14:** Schematic overview of some parameter influencing the crack growth behaviour of P92 at 500 to 600°C

In this ferritic steel P92, the rapidly growing of an oxide layer within the crack protected the oxidation reaction in the grain boundary. There is a big difference between the kind of grain boundaries in a ferritic steel and an austenite structure (steel or Ni-base) alloy. The missing SAGBO-effect to be observed in ferritic P92 seemed to improve the resistance of P92 against CFCG at 600°C compared with results of specimens of the austenitic alloy Inconel 706.

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