Oxidation and Corrosion Effects on Thermal Fatigue Behaviour of Hot Work Tool Steel X38CrMoV5 (AISI H11)

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Abstract. Effects of atmosphere and specimen geometry on thermal fatigue (TF) crack initiation and propagation in a low Si content hot work tool steel X38CrMoV5-47HRC were investigated. The TF specimen's geometry enhances the uni-axial TF loading conditions. A high frequency induction heating (3 to 4 MHz) is used. A new TF rig, working under air and/or inert atmosphere with reduced P_{O2} has been set up. The reduction of P_{O2} results in localized oxidation sites. Whatever geometry and atmosphere conditions, TF cracks initiate exclusively in the oxide layers. Damage mechanisms are environment dependant. Under laboratory air, parallel macroscopic cracks initiate perpendicular to the hoop stress. Under argon and nitrogen, SEM surface observations show that initiated cracks coalesce by zigzagging along crystallographic paths between non-oxidized zones. In-depth crack propagation mechanism is mainly trans-granular. TF crack initiation life under air and in presence of Fe-Al intermetalllics is decreased in comparison to inert atmosphere.

Introduction

Hot forming tools (rolling [1]. forging, die-casting, in particular high pressure die casting etc) works under transient thermal cycling. They are prone to thermal fatigue (TF), heat-checking ("crack") initiation and propagation. Such cracking is basically related to macroscopic and microscopic local multi-axial loadings. In fact, heat flux density and subsequent temperature distributions change from one region to another in a die inducing different thermal fatigue strains and stress histories [2]. TF damage may be activated and/or coupled with environmental effects (oxidation, corrosion). Investigations on TF crack propagation on tubular specimens are rather difficult mainly because of very complex crack propagation mechanisms under multi-axial loadings. Procedure for determination of crack propagation curves, cracks depth vs. number of thermal cycles is very time consuming. The single edge wedged cross-section specimens [3,4,5] or thin circular-shaped discs match better to TF crack propagation investigations under quasi-uni-axial thermal fatigue loading.

This contribution deals with investigations on TF crack initiation and propagation under various experimental conditions of a low Si double tempered martensitic X38CrMoV5 (47 HRC) steel. TF experiments are conducted both in air and under argon or nitrogen. In order to evaluate the influence of intermetallic phases on cracks initiation, some specimens were pre-aluminized prior to be tested under TF loadings. New TF specimens are used.

Experiments and material

Material and specimens. A quenched and double tempered martensitic tool steel, X38CrMoV5 with low Si content is investigated with a 47HRC hardness (Table 1). Heat treated forged bars are delivered by Aubert & Duval France.

Table.1 Chemical composition of X38CrMoV5 (AISI H11) (% weight).

C	Cr	Mo	V	Si	Mn	P	Sn	Sb	Fe
0.36	5.06	1.25	0.49	0.35	0.36	0.006	0.0022	0.0005	bal

The isothermal oxidation behaviour of this hot work tool steel was previously studied [6]. The oxide scales, obtained from 600°C to 700°C are formed of an external iron-rich scale (hematite α-$Fe_2 0_3$) and an internal scale enriched in Cr (spinel oxide $(Fe, Cr)_3O_4$) (Fig. 1).

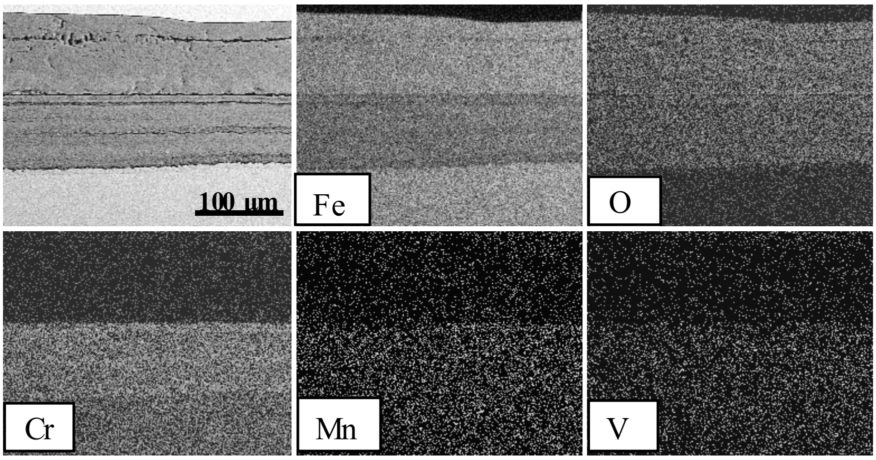


Fig. 1. X-ray maps of the cross section of an oxide scale grown during exposure at 700°C for 90 hours in wet air (pH20 = 198mbars)

TF specimens are machined from these bars. The specimen edges are polished along the circumferential direction. Details of TF specimens are given in Fig. 2.

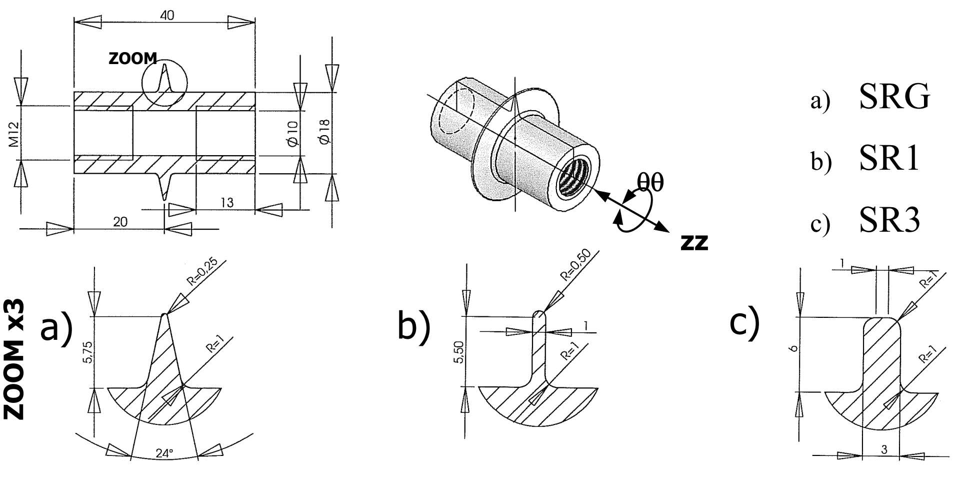


Fig. 2. Typical thermal fatigue specimens (dimensions: mm)

Different TF specimen geometries were used. The following results concerned only the specimen called SR1 (Fig. 2). Details of the selection of this geometry are given in [7].

Thermal fatigue reference cycle and test conditions. The edges of TF specimens are heated by an Hüttinger high frequency induction facility (25kW, 3 to 4 MHz) and cooled by natural convection air, while they are internally cooled by a permanent water-circulation.

Temperature-times (T-t) cycles are measured by spot-welded thermocouples type-K on the edge. An in-house LabView data acquisition system collects these datas. TF experiments are performed at different T_{max} (600 and 650°C) under identical heating period (about 1s). The minimum temperature (T_{min}) is set at 100°C (Fig. 3). Experiments are regularly interrupted for SEM examinations of crack initiation and crack propagation inwards from specimen edges.

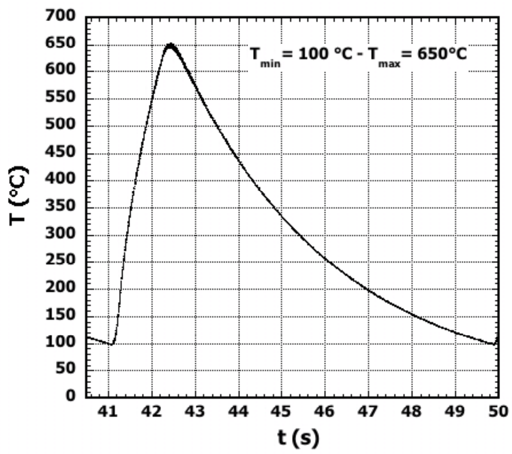
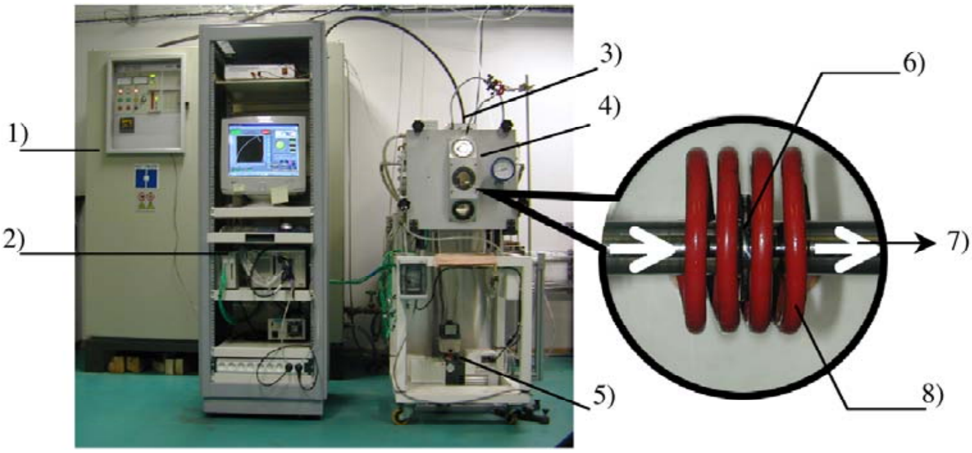


Fig. 3. Typical TF cycle at edge tip of specimens. Heating and cooling period are about 1s and 7s respectively

A vacuum chamber is used for TF experiments under inert gas (Fig. 4). The chamber is first put under a secondary vacuum ($5x10^{-5}$ mbars), prior introducing inert gas (argon or nitrogen). Table.2 shows the gas impurities contents.

Table.2 Gas composition

	H_2O	O_2	C_nH_m
Argon (Alphagaz Ar)	< 3 ppm	< 2 ppm	< 0,5 ppm
Nitrogen (Alphagaz N_2)	< 3 ppm	< 2 ppm	< 0,5 ppm



1)Hüttinger induction facility - 25kW, 3 to 4 MHz frequency

2) Data acquisition system (National Instrument PXI-1002 – Soft Labview)

3) Gas pipe

4) Chamber with controlled atmosphere

5) Vacuum pump

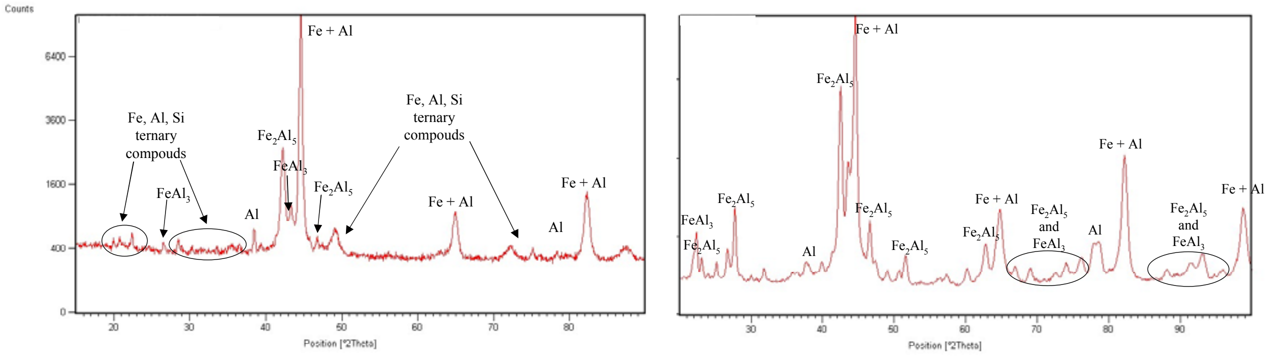
6) SR1 Specimen

7) Water cooled direction through the TF specimen

8) Induction coil

Fig. 4. Thermal fatigue rig under controlled atmospher.

Aluminium diffusion coating applied by pack cementation process. X-ray diffraction analyses of the surface of an industrial high-pressure die-casting die (after 20 000 shuts) reveal the presence of iron-aluminium intermetallic phases. The injected aluminium alloys was AS9U3 alloy with high level of silicon content. The X-ray diffraction analyses reveal the presence of binary iron-aluminium intermetallic phases and ternary iron-aluminium-silicon intermetallic phases (Fig. 5(a)) [8, 9, 10, 11]. In order to estimate the role of iron aluminium intermetallic phases on crack initiation, some specimens were pre-aluminizing by pack cementation. This process is usually widely used to confer oxidation resistance on ferrous or nickel alloys. In order to maintain the initial steel microstructure and hardness, it was decided to perform the pack cementation at 550°C, that is lower than the second tempering temperature. The specimen is immersed in a powder's mixture containing pure aluminium (source), a halide salt (activator) and alumina as inert diluent (filler) [12]. This pack cementation process allows to form the same binary iron-aluminium intermetallic phases on the surface specimen as industrial die (Fig. 5(b)). It should be emphasized that on the industrial die, different ternary iron-aluminium-silicon intermetallics are observed (Fig. 5(a)).



a) b)

Fig. 5. X-ray diffraction pattern on X38CrMoV5 coupons :

a) cut from the industrial die

b) after aluminizing by pack cementation (8h at 550°C)

Fig. 6 shows the intermetallic layers obtained by pack cementation with an external layer of $FeAl_3$ and an inner layer of Fe_2Al_5.

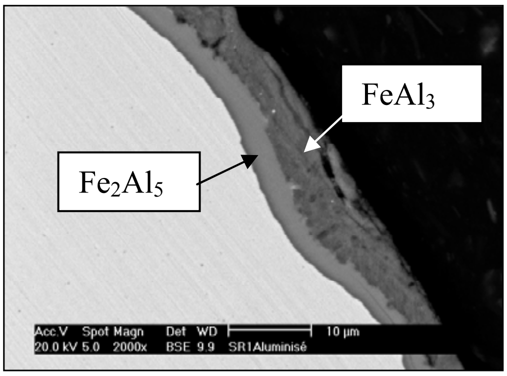


Fig. 6. Cross section of X38CrMoV5 steel aluminized at 550°C for 8h.

Results and discussion.

Crack initiation and propagation mechanisms While the oxide morphology depends upon T_{max}, the oxide scale is always duplex with one layer rich in Cr in contact with steel and one poor in Cr in direct contact with air. These layers have quasi-identical thicknesses [13] alike under isothermal oxidation tests (Fig. 1). Under air, uniform oxide layer is formed on the edge of TF specimens (Fig. 7(a)). Under inert atmosphere, very early, parallel-localised oxide scales (alike an “atoll”) are formed perpendicular to the hoop stress ($\sigma_{\theta\theta}$). The “non-oxidised” regions surround these localized oxide-atolls.

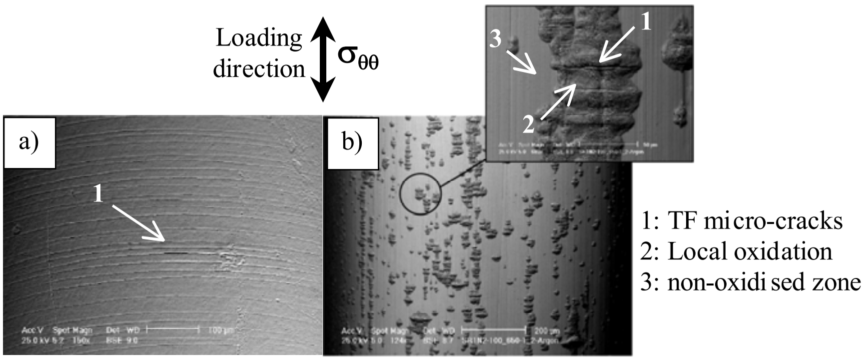


Fig. 7. Thermal fatigue oxidation mechanisms (T_{max} = 650°C):

a) experiment under air after 2500 cycles (generalized oxidation)

b) experiment under argon with reduced P_{O2} after 5000 cycles

Edge surface SEM observations at Tmax=650°C under air shows that crack initiation and early propagation are relatively perpendicular to the hoop stress direction (Fig. 8). Under argon, short cracks initiate on localised oxides atolls and then coalesce by zigzag propagation in between.

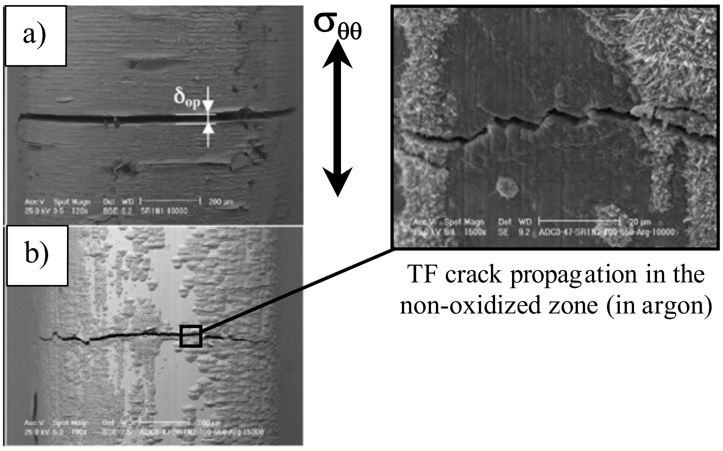


Fig. 8. Typical Uni-axial TF crack propagation (Tmax = 650°C):

a) Sample tested under air (10000 cycles)

b) Sample tested under argon with reduced P_{O2} (15000 cycles)

Fig. 9 presents the cross section of a TF specimen tested at 650°C under air showing the parallel cracks initiated on oxide layer corresponding wedged type inward penetration to the steel. These perturbations are privileged region for further cracks propagations.

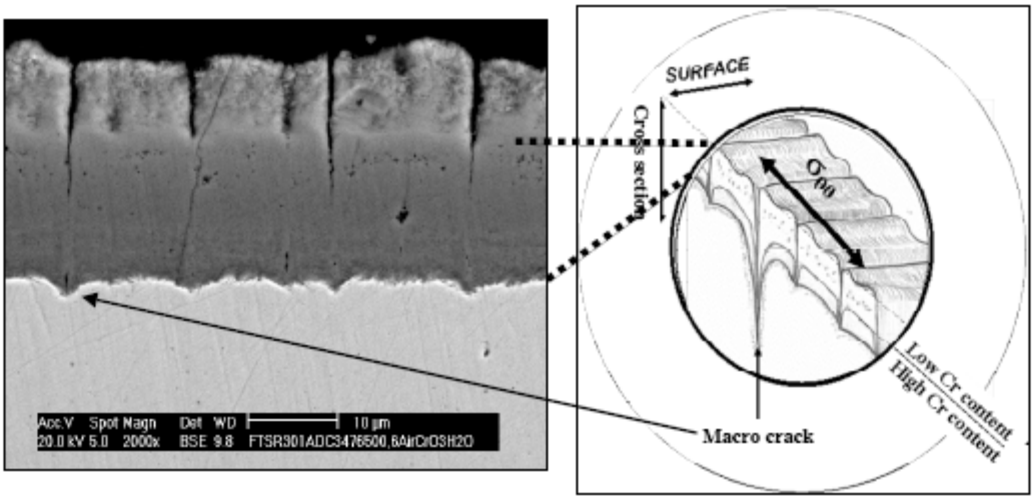


Fig. 9. Cross section of the edge tip of TF specimen tested at 650°C under air.

This specimen geometry is a very promising for in-depth crack propagation investigations. When the crack progresses beyond about the edge tip radii, it becomes a through thickness crack, leading to follow the crack progress via measurements on both disc faces. The TF crack depth vs. of number of cycles curves are reported in Fig. 10. As can be observed, TF crack initiation life under air is decreased in comparison to the inert atmosphere. Similar trend is observed for pre-aluminized specimen tested under inert atmosphere revealing definitively the effect of the binary iron-aluminium intermetallics. In both cases, no clear effects on crack propagation rate are observed.

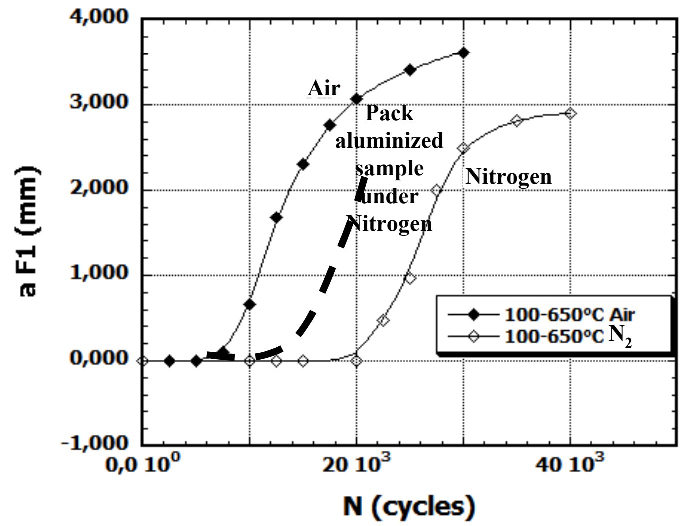


Fig. 10. Variation of cracks depth vs. number of thermal cycles under air and inert atmosphere for X38CrMoV5 with and without a pre-aluminized layer.

Conclusions

Thermal fatigue behaviour of a low-Si content X38CrMoV5 (AISI H11)-47 HRC is investigated. New thermal fatigue specimen geometries and a new high frequency induction heating under controlled atmosphere (air, inert gas, vacuum) are used. It is observed that oxidation decreases the thermal fatigue crack initiation life. Under test conditions reported here, no clear effect of oxidation on crack propagation rate is observed. Under inert atmosphere, early localized oxidation is observed. Whatever geometry and atmosphere conditions, TF cracks initiate exclusively in the oxide layers. Under inert atmosphere, short cracks coalesce by zigzagging along crystallographic paths between non-oxidized zones. In depth crack propagation mechanism is mainly trans-granular both under air and inert atmosphere.

Thermal fatigue experiments performed under inert atmosphere reveal definitely that iron-aluminium intermetallic phases decrease the thermal fatigue crack initiation life of low-silicon X38CrMoV5 die steel.

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