INFLUENCE OF ENVIRONMENT AT 500°C ON FATIGUE BEHAVIOUR IN A Ti6246 ALLOY

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ABSTRACT

The present work considers environmental Fatigue Crack Propagation in a Ti6246 alloy at 500°C. The environments are normal air, humidified argon, low vacuum and high vacuum. There is a general trend of higher crack growth rates in the order : humidified argon, air, low vacuum and high vacuum. This corresponds to increase crack growth rates at a given ΔK when the partial pressure of water vapor is increased and the frequency decreased. A fatigue-corrosion mechanism is identified in the three tested environments and the active specie determined as water vapour. Microfractographic observations show that cracked surfaces aspect depends on the nature of the tested environment. A mechanism involving a humidity dependent resistance of Titanium alloys by water vapour adsorption is found to be plausible.

KEYWORDS

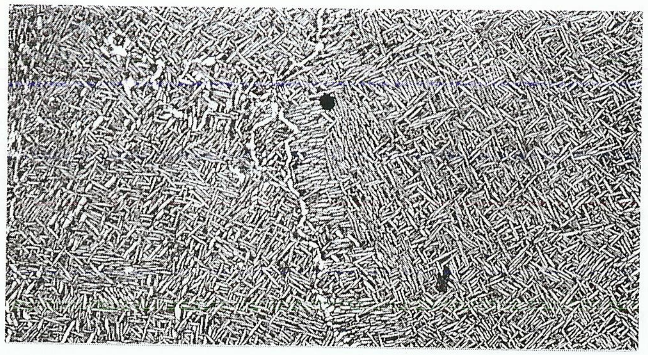
Fatigue Crack Propagation, Titanium alloy, temperature, environment, water vapour, frequency, oxidation, adsorption.

INTRODUCTION

Titanium alloys are interesting to aerospace structural designers because of their corrosion resistance, high specific strengh and low density. They are often used under cyclic loading conditions in aggressive environments such as humid air and at high temperature (up to 600°C). Consequently, attention is concentrated on the Ti6246 at 500°C specifically on the influence of environment. Fatigue in Titanium alloys at high temperature in air is believed to be controlled by oxygen action by many authors (Ruppen, 1983; Foerch, 1993). In this study, considering environments "free" of oxygen with various partial pressures of water vapour at different frequencies, makes the distinction between these two gaseous species. Results of this investigation deal with the effects of environment at 500°C, on the near-threshold and the mid-rate Fatigue Crack Propagation (FCP) behaviour of a Ti6246 alloy.

MATERIAL CHARACTERIZATION

The Ti-6%Al-2%Sn-4%Zr-6%Mo (in wt%) used in this investigation is β-forged at 950°C. The specimen was subjected to heat treatment consisting of 930°C for two hours, followed by water quenching, 900°C for one hour and air cooled, held at 595°C for a total aging time of eight hours and air cooled.
It displays a Widmanstätten structure consisting of aligned alpha platelets and basketweaved structure contained in large prior beta-grains (200 µm-300 µm). The size of α grains does not exeed 50 µm. An illustration of the microstructure is presented in figure 1 and the mechanical properties are listed in Table1.



50 µm

Fig.1 : Optical microfractography of the Ti6246 alloy

Table 1 : Mechanical properties of the Ti6246

	20°C	300°C	425°C	465°C	500°C
E (GPa)	125	112	106	104	102
A %	10.2	10.9	14.6	-	-
σ_e (MPa)	985	753	727	-	-
σ_{max} (MPa)	1098	901	859	-	-

EXPERIMENTAL PROCEDURE

FCP tests are performed using CT 40 compact tension specimens (W = 40 mm, B = 10 mm) designed in accordance with ASTM recommandations, (standard E647, 1986). A sinusoïdal waveform is used at frequencies varying from 35 Hz to 10^{-3} Hz with a load ratio R of 0.1 and at variable R. Some additional dwell waveforms are used at 500°C with 10 seconds rise and fall time and hold periods of 90 seconds and 180 seconds or no hold time (triangular signal of 10s-10s). Precracking for all specimens is carried out at room temperature at R = 0.1. Crack lengths are tracked using a DC (electrical) potential drop technique. Crack closure is detected using a capacitive displacement gauge and determined by means of the compliance variation method (Elber, 1970; Kikukawa, 1977). In addition, some tests are performed at variable R (constant Kmax) so as to eliminate any closure contribution in the near threshold range.
The environmental effect at 500°C is studied with complementary experiments performed in various gaseous atmospheres controlled by a mass spectrometer and by hygrometers. On one hand, the partial pressure of waper vapour can be lowered by reducing the total pressure : high vacuum, low vacuum; on the other hand, an atmospheric environment saturated with water vapour and with a very low amount of oxygen is obtained with a controlled leak on humidified argon (50 pct relative humidity).
For all the tested frequencies conditions, the threshold is determined using a load shedding procedure down to the threshold. The load was then progressively increased by step of 3 to 5 %, to determine the crack growth data at increasing ΔK.

RESULTS AND DISCUSSION

Air and high vacuum

The FCP determined in high vacuum, supposed to be representative of the intrinsic behaviour of the Ti6246 at 500°C, is compared to air environment in figure 2a) and b) with and without closure correction.

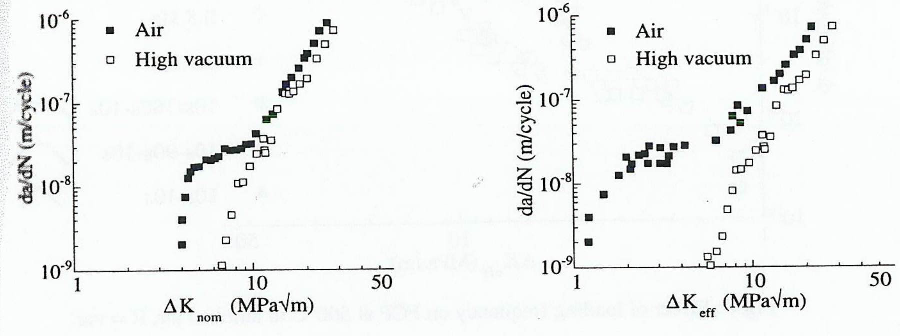


Fig.2 a) and b) : Influence of environment, R = 0.1, 35 Hz.

For rates higher than $4x10^{-8}$ m/cycle, similar behaviour is revealed for the nominal curves and a little difference appears on the effective curves due to the influence of environment. But the most significant effect of environment is obtained for lower rates with nominal and effective thresholds respectively at 7 MPa√m in air and 4 MPa√m in high vacuum, 1 MPa√m in air and 6 MPa√m in high vacuum. An enhanced crack propagation is encountered at 500°C in air after crack closure correction due to a well defined plateau at $3x10^{-8}$ m/cycle. At this growth rate, the propagation is quite independent of the applied stress, suggesting that the controlling parameter is mostly governed by the time. This result indicates a great sensitivity to environment of the material at 500°C. The FCP behaviour of the Ti6246 alloy at temperatures ranging from RT to 500°C in laboratory air described elsewhere (Lesterlin, 1994), shows that the Crack Growth (CG) rate at a given ΔK range increases with increasing temperature specially at 500°C.
Microfractographic observations of the cracked surfaces of the specimen tested at 500°C show yellow and purple oxide bands corresponding to the different steps of crack propagation. By mean of RBS analyser (Lesterlin, PHD-1996), coloration of oxidized fracture surfaces has been related to the oxide thickness and the oxide has been identified as TiO_2 (rutile) whatever the coloration. Any direct correlation can not be etablished between the surface oxidation and the embrittling mechanism. These observations do not support any oxygen embrittling role.

Influence of test frequency in air

In a previous paper (Lesterlin, 1996) it has been shown that the influence of frequency increases with temperature and appears to be much more pronounced at 500°C. This result supports the existence of an enhanced effect of environment which becomes more accentuated at 500°C. Such influence of frequency is consistent with a time dependent mechanism and to identify such a synergical effect between a time dependent environmental process and the mechanical loading, three tests were performed at 500°C at constant K_{max}, respectively at 3.5 Hz, 0.5 Hz and 0.1 Hz (Fig.4) and compared to the effective measurements obtained at 35 Hz. Complementary growth rate data were performed with dwell waveforms (10s-90s-10s, 10s-180s-10s) and a triangular waveform without hold time (10s-10s).

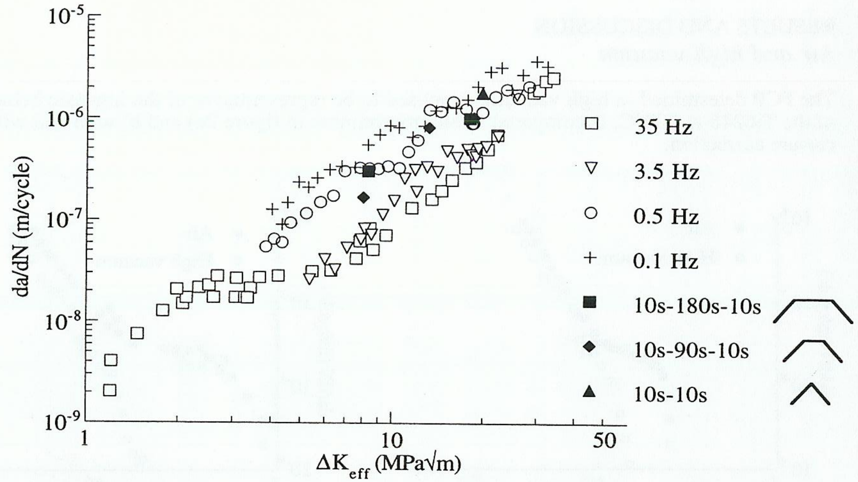
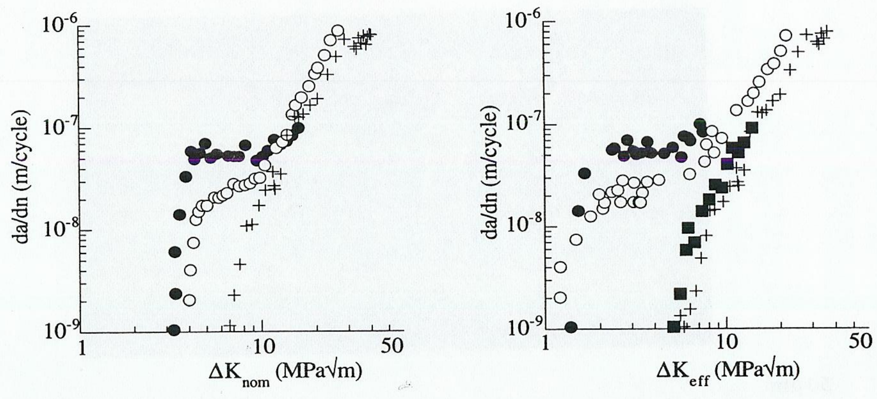


Fig.4 : Effect of loading frequency on FCP at 500°C in ambient air, R = var.

For rates lower than 3×10^{-6} m/cycle, lowering frequency from 35 Hz to 0.1 Hz induces an increase in FCP which becomes more pronounced as the frequency decreases. At a given ΔK_{eff} the growth rate at 0.1 Hz can be ten times higher than at 35 Hz.
Even if at 0.1 Hz and 35 Hz, the fracture surfaces present a yellow coloration, a substantial increase of the growth rates is observed when the frequency is lowered : i.e. 10^{-6} m/cycle at 0.1 Hz and 10^{-7} m/cycle at 35 Hz, at a fixed ΔK of 10 MPa√m. Such observations support the existence at low frequency of a more embrittling mechanism which is not related to significant change in surface oxidation (Lesterlin, 1996).
The triangular and dwell loadings give growth rates comparable to that of the sinusoïdal loading at 0.5 Hz and 0.1 Hz supporting a saturation in the environmental influence at very low frequencies. These results suggest that a creep-fatigue type of damage does not seem to be operative at 500°C. Therefore, the loss in the FCP resistance of the material at this temperature level can be primarily attributed to a corrosion-fatigue mechanism.
The nature of the active species, which is suspected to be oxygen in the literature (Ruppen, 1983; Foerch, 1993) has to be determined. Furthemore, on the basis of previous experiments conducted at 300°C in various environments with controlled partial pressures of water vapour and oxygen, the detrimental influence of ambient environment at this temperature has been attributed to an embrittling effect of water vapour particularly enhanced in the near threshold domain (Sarrazin, 1995).

ENVIRONMENTAL ASSISTED FATIGUE

To get more informations on the behaviour of Titanium alloys at 500°C and on the related mechanisms, tests were carried out in selected controlled environments ; i.e. humidified Argon, containing the same amount of water vapour as in ambient air but with a partial pressure of oxygen 10^4 times lower, and low vacuum, containing a very low partial pressure of water vapour. Results are compared to the above presented data in air and high vacuum.



a) b)

● Humidified argon : P_{H2O} = 1.3 kPa; P_{O2} < 1 Pa
O Air : P_{H2O} = 1.3 kPa; $P_{O2} = 2\times10^4$ Pa
■ Low vacuum : P_{H2O} = 1 Pa; $P_{O2} < 4\times10^{-4}$ Pa
+ High vacuum : $P_{H2O} = 2\times10^{-4}$ Pa; $P_{O2} < 10^{-6}$ Pa

Fig.5 : CG rates in humidified argon, air, low vacuum and high vacuum, 35 Hz.

The corresponding nominal and effective crack propagation data obtained at 35 Hz for the different environments are presented in figure 5 a and b. The propagation curves in humidified argon are characterized by a plateau, like in air, at ΔK_{nom} ranging from 4 MPa√m to 8 MPa√m and ΔK_{eff} from 2 MPa√m to 7 MPa√m. This plateau phenomenom in the mid-rate range is particulary pronounced and occurs at higher rates in humidified argon (about 5×10^{-8} m/cycle) than in air (about 2×10^{-8} m/cycle) in both diagrams. This result supports that at 500°C, water vapour constitutes the active specie responsible of the great enhancement observed in the mid-rate range compared to high vacuum or to low vacuum which both presents the same behaviour. Indeed, different rates of FCP for the humidified argon and air emphasize that oxygen has no embrittling effect on crack tip fatigue damage and at least presents a protective role (Sankaran, 1994). The behaviour in low vacuum, comparable to that in high vacuum shows, that a partial pressure of water vapour of 1 Pa at 35 Hz is not sufficient to embrittle the material. Finally, all these results indicate the predominant importance of moisture in air.

Water vapor partial pressure and test frequency interaction

Frequency can have a great influence in air predicting a direct relation between frequency and the partial pressure of water vapor, as previously observed (Gao, 1984; Sarrazin, 1996) : high frequencies allow less time for H_2O interaction in the crack tip, requiring a greater environmental pressure to produce a given growth rate. What appears to be a behaviour corresponding to a saturation effect of environment has been determined in low vacuum and in humidified argon, like in air, at fixed frequencies respectively of 0.1 Hz in argon and 0.01 Hz in low vacuum (fig.6). The dashed line faired through the data in high vacuum is used as a reference to evaluate the environment effect.

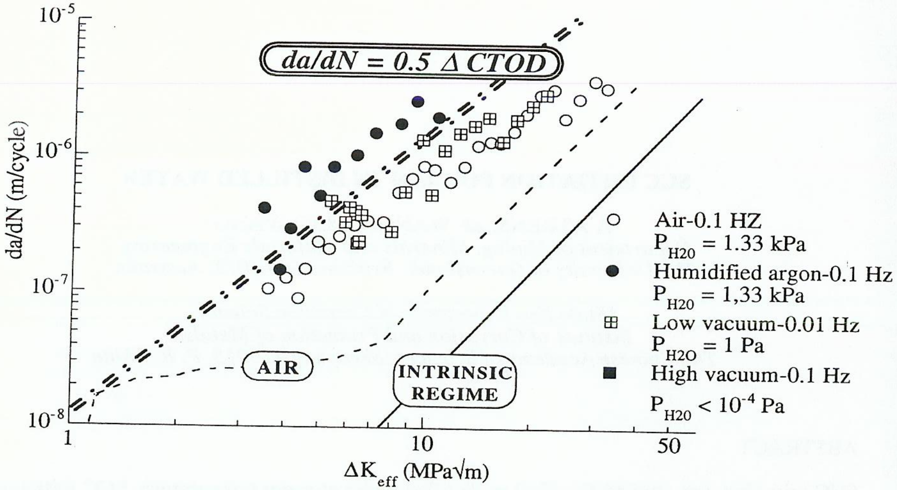


Fig.6 : Influence of frequency at 500°C in the four tested middles.

The most demonstrative result is the very large difference in FCP rates between high vacuum and low vacuum when the frequency is lowered. In high vacuum, lowering the frequency at 0.1 Hz leads to the same rates than at 35 Hz, and confirms that this environment is representative of the intrinsic behaviour of the Ti6246 at 500°C. But in low vacuum, when the frequency is decreased from 35 Hz to 0.01 Hz, FCP rates like in air are obtained, showing that a partial pressure of 1 Pa of water vapour is sufficient to cause accelerated cracking at a such low frequency, while at 35 Hz, the same curves are obtained in high and low vacuum. Similar result has been reported by other authors for Titanium alloys, but only at room temperature (Wanhill, 1976; Gao, 1984). The low pressure of water vapour in low vacuum is sufficient to induce a crack acceleration. It only needs more time than in air. In humidified argon at 0.1 Hz, rates are slightly higher than in air in a similar way as at 35 Hz.
The comparison of the propagation data for different partial pressures of water vapour and different frequencies suggests an embrittling process controlled by the exposure of the fresh surfaces at the crack tip as suggested by Wei (Gao, 1984), and support the existence of a fatigue-corrosion mechanism in air which is also operative in low vacuum and in humidified argon. In the cases the active specie appears to be water vapour. The substantial enhancement of the saturated regime can be attributed to the adsorption (Langmuir, 1918; Achter, 1966; Rehbinder, 1971; Piascik, 1991) of water vapour on freshly created cracked surfaces at the crack tip and subsequent hydrogen assistance. The saturated regime for the three active environments, illustrated on the figure 6 by a dashed line, can be described by the model proposed by Mc Clintock (Mc Clintock, 1968) :

$$da/dN = 0.5\ \Delta CTOD$$

The microfractographies given in figure 7 are representative of the features found on the Ti6246 fractures surfaces in humidified argon, air and low vacuum at low frequencies. For the three environments, FCP produces highly faceted and flat surfaces, corresponding to a transgranular stage II propagation. The high density of faceted areas and the flat surfaces support a mechanism mainly insensitive to microstructure. Microcracks, which are present in all the cases, seem associated to the coupled action of temperature (Foerch, 1993) and water vapour. The most significant feature is the difference of the microcracks morphology with respect to environment. In low vacuum, the microcracks are parallel to the propagation direction while they are perpendicular in humidified argon and in air. Specimen have been sectionned, in the direction parallel to the propagation to reveal the correlation between the microstructural features and the subsurface microcracks, fig.7. In air, the section plane shows that microcracking

proceeds along α laths and seems to result from oxidation, while in humidified argon, are observed microcracks independent on microstructure. In low vacuum, microcracks can not be observed because they are parallel to the section plane. In humidified argon and in air, both surfaces are respectively covered with blue and yellow oxide layers but are generally free of oxide debris. In low vacuum, few oxide is present. But whatever these differences in the surface morphology, the FCP rates are quite similar (specially in low vacuum and in air). This is consistent with previous studies (Allison, 1985) which establish that surface oxide films are not damaging. All these results support a detrimental influence of water vapour in each case but with some specific characteristics.

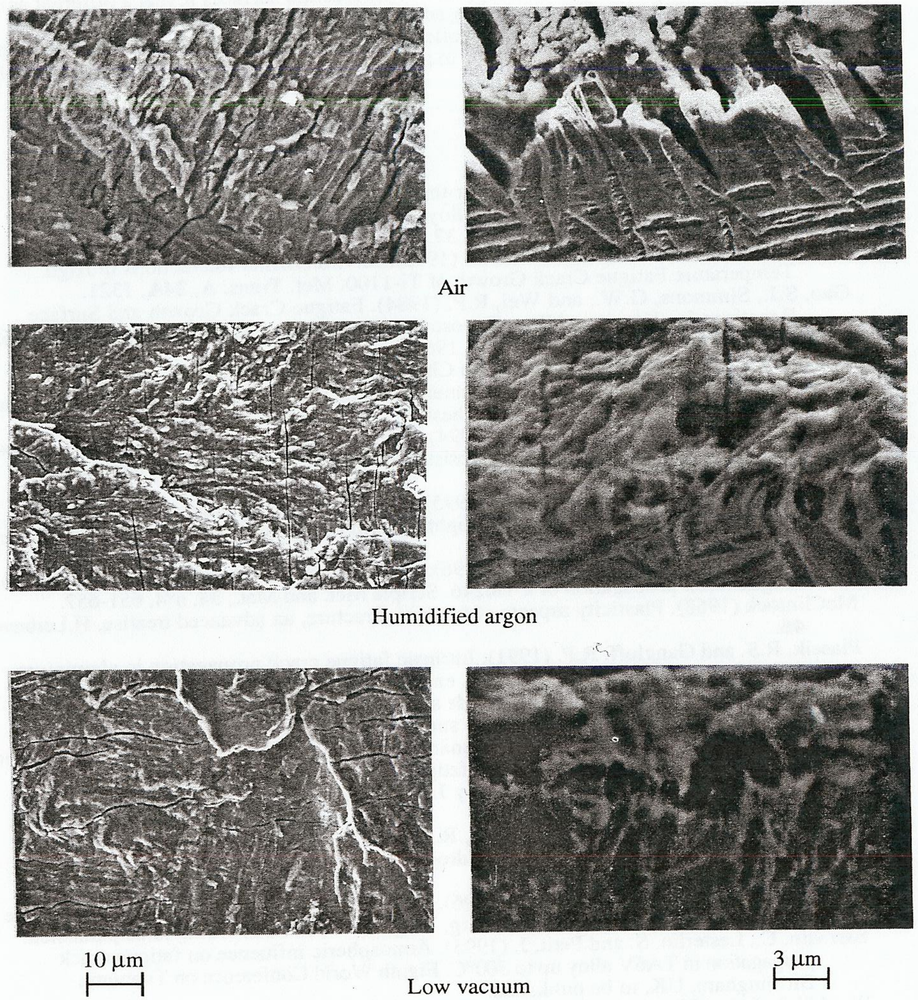


Fig.7 : SEM images showing both microstructure and fracture surfaces for the saturated regime at 500°C, 10^{-7}-10^{-6} m/cycle. (Propagation : left - right)

CONCLUSION

The study of the Fatigue Crack Propagation behaviour in the Ti6246 alloy at 500°C in various gaseous environmental conditions leads to the following conclusions :

- A substantial Crack Growth rates enhancement is obtained in air at 500°C on the Ti6246 attributed to a temperature-environment interaction.
- By mean of tests at various frequencies and in different partial pressures of water vapour, a saturated environment effect is determined supporting a fatigue-corrosion mechanism. The active specie is identified as water vapour, but the fracture surfaces reveal a different action of environment in each case. Oxygen mainly adds a protective role.
- This mechanism appears to be controlled by the exposure which depends on the partial pressure of water vapour and on the time to achieve surface coverage by adsorbed molecules.

REFERENCES

Achter, M.R. (1966). ASTM, STP, 181.
Allison, J.E., Williams, J.C. (1985). Near-threshold fatigue crack growth phenomena at elevated temperature in titanium Alloys. Scripta Met., **19**, 773-778.
Elber, W. (1970). Eng. Fract. Mech., **2**, 37.
Foerch, R., Madsen, A. and Ghonem, H. (1993). Environmental Interactions in High Temperature Fatigue Crack Growth of Ti-1100. Met. Trans. A, **24A**, 1321.
Gao, S.J., Simmons, G.W., and Wei, R.P. (1984). Fatigue Crack Growth and Surface Reactions for Titanium Alloys Exposed to Water Vapor. Mat. Sc. and Engng., **62**, 65.
Kikukawa and al (1977). J. of Mat. Sci., 1964.
Langmuir, I. (1918). Journal of American Chemical Society 40, 1361.
Lesterlin, S. (1996). Influence de l'environnement et de la température sur la fissuration par fatigue des alliages de titane. PHD thesis.
Lesterlin, S., Sarrazin, C. and Petit, J. (1994). In : Structures des véhicules spatiaux et essais mécaniques. Fatigue crack growth resistance of Ti alloys at elevated temperature. CNES, 905.
Lesterlin, S., Sarrazin, C., and Petit, J. (1995). Temperature-environment interactions on fatigue behaviour in Ti6246 alloy. Eighth World Conference on Titanium, Birmingham, UK, to be published.
Lesterlin, S., Sarrazin, C. and Petit, J. (1996). Temperature and environment interactions on fatigue crack propagation of a Ti6246. Scripta Met. and Mat., **34**, n°4, 651-657.
McClintock (1968). Plasticity aspects of fracture. Fracture, an advanced treatise, H.Leibewitz, 48.
Piascik, R.S. and Gangloff, R.P. (1991). Intrinsic fatigue crack propagation in aluminium-lithium alloys : the effect of gaseous environments. Met. Trans., **22A**, 2415.
Rehbinder, P.A. (1971). Les phénomènes de surface dans la déformation et la fracture des solides. Séminaire de mécanique des surfaces, ISMCM.
Ruppen, J.A., Hoffman, C.L., Radhakrishnan, V.M. and McEvily, A.J. (1983). The effect of environment and temperature on the fatigue behavior of Titanium alloys. In : Fatigue, Environment and T effects. Edited by J.H. Burke et V. Weiss, Plenum Press, New York, 265.
Sankaran, S.N., Herrmann, R.K., Outlaw, R.A., and Clarck, R.K. (1994). Barrier-Layer Formation and Its Control during Hydrogen Permeation trough Ti-24Al-11Nb Alloy. Met. et Mat. Trans.A, **25A**, 89.
Sarrazin, C., Lesterlin, S. and Petit, J. (1996). 27th ASTM National Symposium on Fatigue and Fracture Mechanics, Williamsburg, Virginia, ASTM STP 1296, to be published.
Sarrazin, C., Lesterlin, S. and Petit, J. (1995). Atmospheric influence on fatigue crack propagation in TA6V alloy up to 300°C. Eighth World Conference on Titanium, Birmingham, UK, to be published.
Wanhill, R.J.H. (1976). Environmental Fatigue Crack Propagation in Ti-6Al-4V Sheet. Met. trans. A, **7A**, 1365.