# EFFECT OF TEMPERATURE AND STRAIN RATE ON THE CYCLIC PLASTICITY MECHANISMS OF AN INTERSTITIAL FREE STEEL - COMPARISON WITH ULTRA HIGH PURITY IRON

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## ABSTRACT

The cyclic plasticity mechanisms in b.c.c. materials are temperature and strain rate dependent. Below a critical temperature $T_0$, slip asymmetry (due to a reduced mobility of screw dislocations) occurs and leads to grains shape change and to intergranular short crack initiation, whereas above the transition temperature, symmetric slip is observed and the crack initiation sites are mainly transgranular.

The present study has been carried out on a b.c.c. polycrystalline interstitial free steel (IFS) at different temperatures (-80°C - +120°C) and at different strain rates ($6 \cdot 10^{-5}$ s$^{-1}$ - $10^{-3}$ s$^{-1}$), using the measurements of the activation volume V* in order to examine the cyclic plasticity mechanisms. Ultra high purity $\alpha$-iron (Fe-UHP) with controlled carbon contents between <10 and 33 wt ppm has been used as reference.

At room temperature, all the studied alloys are in the transition regime between "low" and "high" temperature. In IFS, below approximately -30°C, a pronounced increase of the maximum tensile stress (at same $\Delta\varepsilon_P/2$) and almost constant and low values of the activation volume V* indicate that the material shifts into the low temperature region.

The increase of carbon content in Fe-UHP leads to the decrease of the transition temperature. This effect is probably due to the decrease of the double kink formation energy.

At room temperature, the IFS activation volume is higher than in pure iron, whatever the strain rate, indicating a decrease of the transition temperature. The reduction of $T_0$ is attributed to the limitation of dislocation mobility by substitution atoms (Ti, Al, Mn) or by fine TiC precipitates.

## INTRODUCTION

Among steels with body centred cubic structure, the interstitial free steels (IFS) are more and more considered as material for the automotive industry. The processing design of such alloys must take into account that the cyclic damage mechanisms in body centred cubic metals are strongly temperature and strain rate dependent because of the particular behaviour of screw dislocations [1,2]. Below a transition temperature $T_0$ ($\dot{\varepsilon}$), the mobility of screw dislocations is strongly reduced and dislocation slip can occur on different planes in tension and in compression which leads to significant shape changes of individual grains [3]. As a consequence, strong stress concentrations appear along grain boundaries and favour intergranular short crack nucleation at surface as shown in $\alpha$-iron alloys by Magnin and Driver [3]. According to Sommer et al [4], the fatigue life of $\alpha$–Fe decreases strongly with temperature below the transition temperature in strain controlled low cycle fatigue tests [4].

In a high purity α-iron, the transition temperature is close to the room temperature as shown by Cottu et al. [5]. Since the overall content of alloying elements in IFS does not exceed 0.3 wt % [Table 1], it is probable that IFS as a construction material for automobiles shifts from "high" to "low" temperature region within its service temperature range, situated approximately between –40°C and +60°C.

In this study, the cyclic plasticity and the crack nucleation mechanisms of an interstitial free steel (IFS) have been examined at different temperatures and strain rates. Ultra high purity α-iron doped with manganese and carbon has been used as reference. Among different alloying elements, carbon is supposed to play an important role in cyclic plasticity mechanisms in the transition temperature range. Indeed, according to Cottu et al. [5], the flow stress of α-iron is affected by carbon under monotonic loading. In fatigue, Sommer et al. [4] investigated the influence of carbon at different temperatures. In carburised iron specimens, the fraction of transgranular short cracks at low temperatures is higher than in Fe-UHP. In parallel, the fatigue life at high temperatures is higher in carburised specimens. These results indicate that the transition temperature $T_0$ may be shifted to lower temperatures when interstitial carbon content increases.

In the present study, the cyclic plasticity mechanisms in IFS and in ultra high purity iron (Fe-UHP) with controlled carbon contents have been investigated at different temperatures and strain rates. The measurements of the activation volume V* in low cycle fatigue tests using the double strain rate change method allows to determine the transition temperature according to the chemical composition.


## MATERIALS AND EXPERIMENTAL PROCEDURE

The chemical composition of the IFS is summarised in Table 1. The estimated residual carbon content in solid solution is very low and does not exceed few ppm, due to the titanium addition and the formation of fine TiC precipitates of about 20 nm diameter.

TABLE 1
COMPOSITION OF THE INTERSTITIAL FREE STEEL (IFS) [$10^{-3}$ WT %]

| C | Mn | P | S | Si | Al | Cu | V | Ti | B | $N_2$ |
|---|-----|----|---|----|----|----|----|-----|----|----|
| 2 | 100 | <1 | 3 | 3 | 53 | 1 | <1 | 110 | <1 | 4 |

The ultra high purity α-iron-base alloys are produced at Ecole des Mines de Saint-Etienne. In order to approach the IFS composition, 500 wt ppm Mn and different carbon contents (<10, 22 and 33 wt ppm) have been added.

All the alloys are single phase body centred cubic with equiaxed grains. The mean grain size is 15 µm for IFS (with no texture), 200 µm for UHP α-iron based alloys (with Mn and C) and 800 µm for UHP α-iron.

The cyclic plasticity mechanisms have been investigated using the measurements of the activation volume V*, defined by :

$$V* = lb\Delta R \tag{1}$$

where l is the dislocation length, b the Burgers modulus, and ΔR the activation distance when the dislocation gets over an obstacle. In a single crystal with one activated slip system, V* is linked to the dislocation mobility by the following relationship:

$$\dot{\gamma} = \dot{\gamma}_0 \exp\left(\frac{\Delta H}{kT}\right) \exp\left(\frac{V* \tau*}{kT}\right) \tag{2}$$

where $\dot{\gamma}$ is the slip velocity, $\dot{\gamma}_0$ a constant, τ* the effective resolved stress in the slip plane, ΔH the activation enthalpy, T the temperature, and k the Boltzmann's constant.

V* can be measured by the double strain-rate change method [3], which consists in modifying the strain rate during a short time, with the hypothesis that no change in the dislocations configuration occurs during this strain-rate change.

In our study, the dislocation structure is first *stabilised in fatigue* (at a constant strain rate $\dot{\varepsilon}_1$), during approximately 500 cycles (see Figure 1). The dislocation structure is formed of cells of about 1,5 µm diameter.
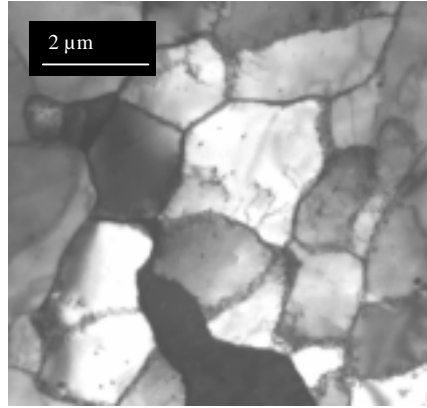


**Figure 1 :** Dislocation cells in IFS: N = 500 cycles, $\Delta\varepsilon_p/2 = 5\cdot10^{-4}$, $\dot{\varepsilon}_p = 2\cdot10^{-4}\ \text{s}^{-1}$

After stabilisation, the strain-rate is changed ($\dot{\varepsilon}_2$) during the first quarter of the fatigue loop in tension as shown in Figure 2. Strain-rate ratios $\dot{\varepsilon}_2/\dot{\varepsilon}_1$ of 10 or 5 have been applied. The activation volume is then calculated from :

$$V^* = kT\ \frac{\ln\left(\dfrac{\dot{\varepsilon}_1}{\dot{\varepsilon}_2}\right)}{M(\sigma_1 - \sigma_2)} \tag{3}$$

where $\sigma_1$ and $\sigma_2$ are the stresses corresponding to the strain rates $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$ respectively, and M is the Taylor factor for an isotropic polycrystal [3].
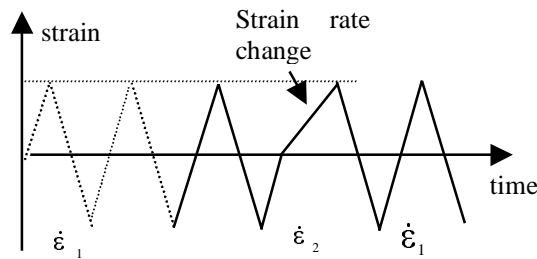


**Figure 2 :** Strain rate change in fatigue at constant strain amplitude

The measurements of V* have been made at temperatures between –80°C and 120°C at nominal strain rates $\dot{\varepsilon}_1$ between $6\cdot10^{-5}\ \text{s}^{-1}$ and $10^{-3}\ \text{s}^{-1}$ and at applied plastic strain amplitude of $3\cdot10^{-3}$. At this deformation level, both edge and screw dislocation are supposed to be mobilised to accommodate the applied strain [2]. A heat-insulated chamber is used at low and high temperatures. The desired temperature is obtained by liquid or gaseous nitrogen introduced into the chamber. At high temperatures, the chamber is heated by Joule effect. All the temperatures were kept constant during the tests within ± 1°C.

## RESULTS AND DISCUSSION

### *Transition temperature of the IFS*

#### *Short crack initiation and propagation*

Short crack initiation sites are both intergranular (Figure 3a) and transgranular at room temperature. However, the propagation is always transgranular and the fracture mode is entirely transgranular with a pronounced striation. Intergranular short cracks appear due to stress concentrations in the vicinity of grain boundaries and it does not result from intergranular embrittlement through phosphorus [6], the phosphorus content in the IF steel being not high enough (<10 ppm) to produce such effects.

The surface of specimens polished prior to fatigue tests is strongly deformed, even at room temperature (Figure 3b). The effect of shape change in b.c.c. single crystals cyclically deformed in the low temperature region is attributed to slip asymmetry between tension and compression [3,8]. In polycrystals, shape changes may result both from the above mentioned asymmetry effects but also from the deformation incompatibilities between adjacent grains.
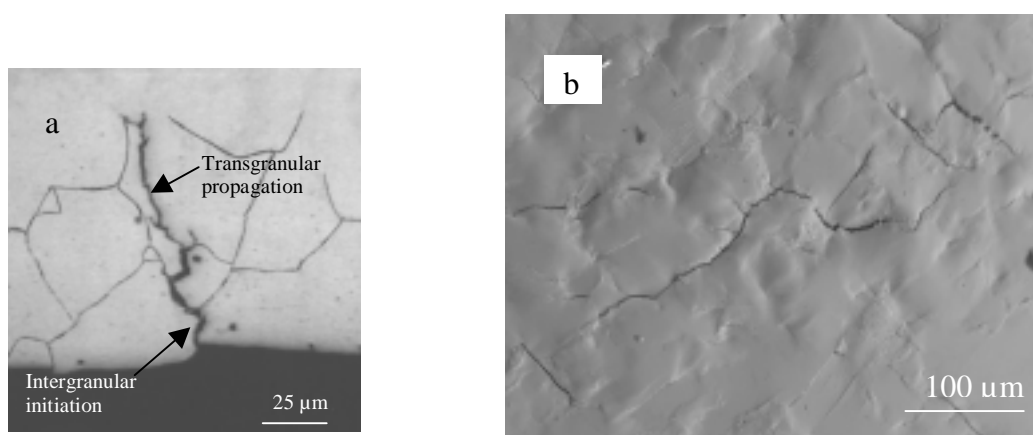


**Figure 3 :** a-Intergranular surface crack initiation and transgranular propagation in the bulk
(IFS, $\Delta\varepsilon_p/2 = 4{,}5\cdot10^{-3}$, $\dot{\varepsilon}_p = 1{,}5\cdot10^{-3}$ s$^{-1}$, N = N$_f$ = 6900 cycles, 20°C)
b-surface of an IFS specimen deformed at $\Delta\varepsilon_p/2 = 3\cdot10^{-3}$ ($\dot{\varepsilon}_p = 4\cdot10^{-4}$ s$^{-1}$, N = N$_f$ = 18500 cycles, 20°C)
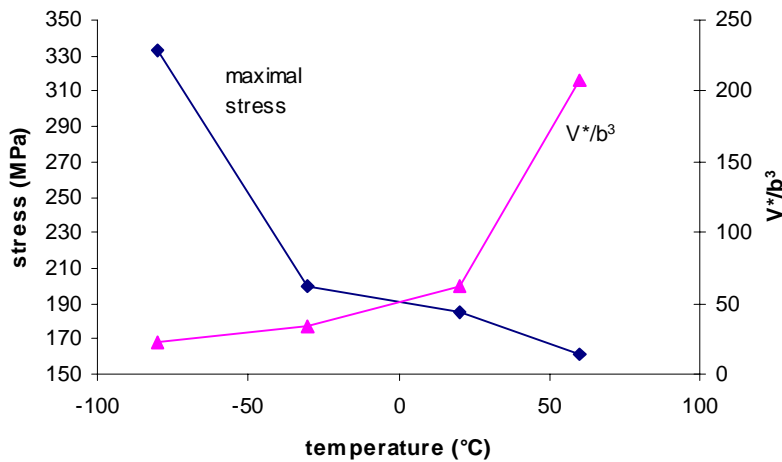
#### *Temperature dependence of the maximum tensile stress and of the activation volume*

The evolution of the maximum tensile stress in cyclic plastic deformation is strongly temperature dependent (Figure 4). A radical change of the cyclic behaviour of the material takes place between -30°C and -80°C. At 20°C and -30°C, cyclic hardening takes place all over the test and fatigue life reaches approximately 15000 cycles at ($\Delta\varepsilon_p/2 = 3\cdot10^{-3}$). At -80°C, the maximum stress is stabilised after 500 cycles and it remains stable until failure. The fatigue life is strongly reduced (5000 cycles). It is noteworthy that a temperature decrease by 50° between 20°C and -30°C leads to the increase of the maximum stress of roughly 50 MPa (from 200 to 250 MPa) while it is three times higher between –30°C and – 80°C (from 250 to 400 MPa). The results presented in Figure 4 are qualitatively equivalent to those obtained by Sommer et al. on ultra high purity iron [4].

The temperature dependence of the maximum tensile stress at applied plastic strain amplitude ($\Delta\varepsilon_p/2 = 3\cdot10^{-3}$, N = 2500 cycles) and of the activation volume is summarised in Figure 5. The maximum stress increases rapidly when the temperature decreases from –30°C to –80°C as already indicated on cyclic hardening curves of Figure 4. Above –30°C, the evolution is much slower. This indicates that the transition temperature of the IFS might be situated above –30°C, and around the room temperature. Indeed, in the "high" temperature regime, the stress is mainly composed of the internal stress, which is almost independent of temperature.
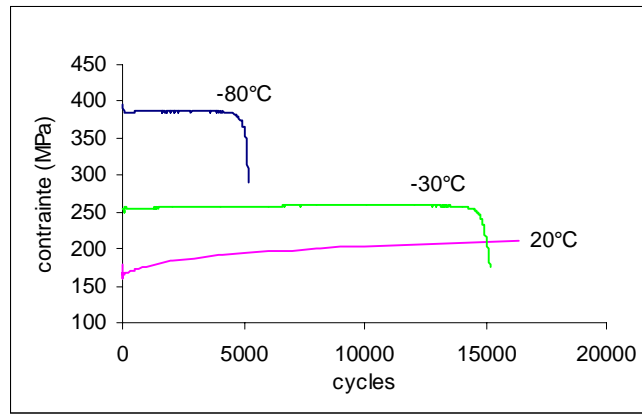
**Figure 4 :** Cyclic hardening curves of IFS at different temperatures ($\Delta\varepsilon_p/2 = 3\cdot10^{-3}$, $\dot{\varepsilon}_p = 10^{-2}\,\mathrm{s}^{-1}$)



**Figure 5 :** Evolution of the activation volume and of the maximum tensile stress (after 2500 cycles) with temperature (IFS, $\Delta\varepsilon_p/2 = 3\cdot10^{-3}$, $\dot{\varepsilon}_p = 10^{-3}\,\mathrm{s}^{-1}$)
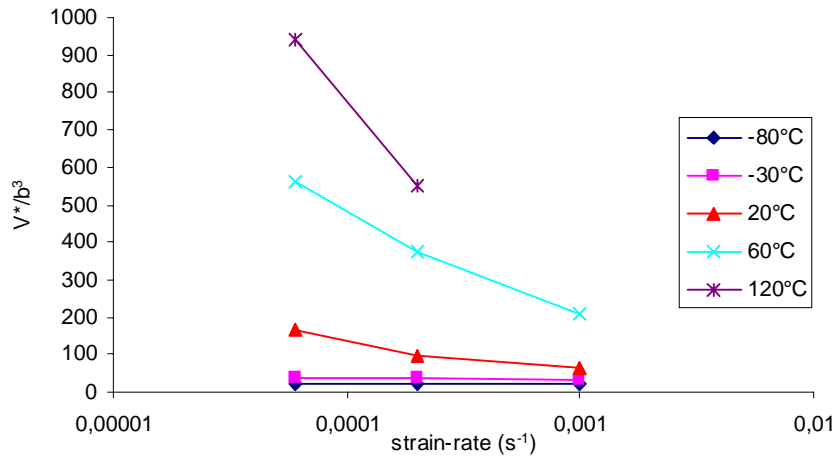
The evolution of the activation volume V* with temperature (Figure 5) completes that of the maximum tensile stress. Below the room temperature the variation of V* with temperature is low whereas it increases strongly between 20°C and 60°C. It has been shown that the evolution of the activation volume in b.c.c. is linked to the one of effective stress [5, 7]: V* decreases when the effective stress increases (i.e. at lower temperatures). For high values of the effective stress, the activation volume is quite constant, whereas the variation is important when the effective stress is low.

The above presented results indicate that the transition between low and high temperature regimes for IFS at $\dot{\varepsilon}_p = 10^{-3}\mathrm{s}^{-1}$ is situated close to room temperature.

*Evolution of the activation volume with temperature and strain rate*
Figure 6 summarises the measurements of the activation volume at different temperatures (-80°C - 120°C) and at different strain-rates ($6\cdot10^{-5}\,\mathrm{s}^{-1}$ - $10^{-3}\,\mathrm{s}^{-1}$). At -80°C and at -30°C, the activation volume is low and almost constant with respect to the strain rate, then the variation becomes more and more significant when the temperature increases. The difference between V* measured at different temperatures strongly increases when the applied strain rate decreases. The transition temperature is actually strongly strain rate dependent as it was shown by Magnin [8] in a b.c.c. Fe-26Cr-1Mo alloy in which $T_0$ increases by 60°C when the strain rate changes from $2\cdot10^{-5}\,\mathrm{s}^{-1}$ to $2\cdot10^{-3}\,\mathrm{s}^{-1}$.
The increase in the activation volume when the strain rate decreases is then related to the change from low to high temperature behaviour with respect to the dislocation mobility.

**Figure 6 :** Evolution of the activation volume with temperature and strain rate in the interstitial free steel

*Composition effect*

In tension, Cottu et al showed that the effective stress of UHP becomes close to zero at 25°C. The transition temperature of UHP $\alpha$-iron and IFS are then both close to the room temperature. This metals don't exhibit any difference in terms of crack initiation and propagation. However significant variations of the activation volume V* are observed between IFS and different UHP alloys with slightly different chemical compositions (Figure 7).
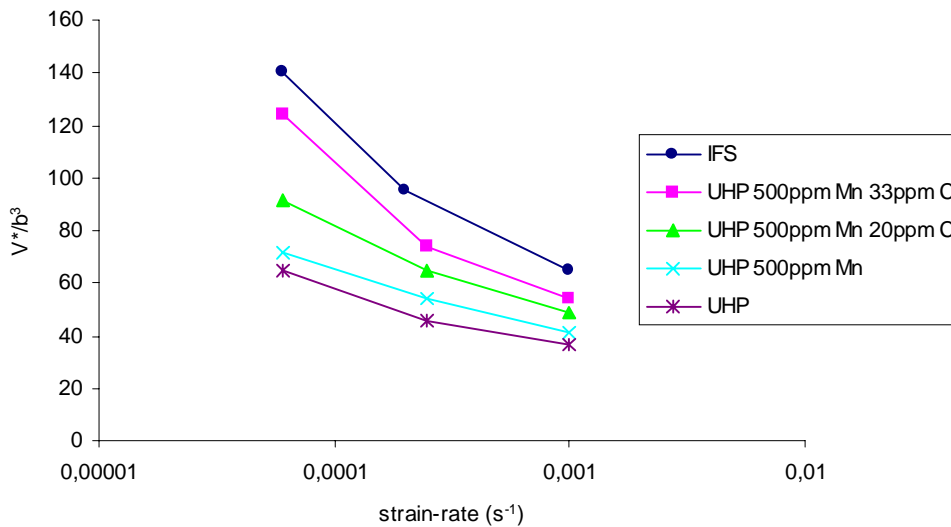


**Figure 7 :** Evolution of the activation volume with composition and strain-rate at room temperature

The addition of 38 wt ppm C in UHP $\alpha$-iron leads to an increase of nearly 100 % of the activation volume at room temperature, in accordance with literature data [5]. This effect is attributed to the decrease of the double kink formation energy by carbon atoms in solid solution [5,10]. The mobility of screw dislocations is then increased. Equivalent effect of carbon and nitrogen on dislocation mobility in $\alpha$-iron has been observed by Nakada [11] and Cottu et al. [5, 12] in tensile tests, leading to a softening in the temperature range between -120°C and -50°C.

The activation volume of the IFS at 20°C is higher than that of all UHP alloys, even if the estimated carbon content in solid solution in IFS is lower from 10 wt ppm. Therefore, the presence of interstitial atoms is not the exclusive factor which determines the activation volume. The difference between IFS and the UHP $\alpha$-iron alloys may be attributed either to the effect of TiC precipitates which act as Frank-Read sources when the strain rate changes, influencing the measurements of V*, or to the substitution elements in solid solution, which can modify the double kink formation energy. The latter hypothesis is partly confirmed by

the fact that the addition of 500 wt ppm Mn increases the activation volume of UHP by 17% at room temperature (Figure 7).

Moreover, the strain rate dependence of the activation volume is stronger for the IFS than for the Fe-UHP at room temperature, which indicates that the transition temperature of the IFS is lower than that of the Fe-UHP.

## CONCLUSION

- The evolution of the maximal tensile stress of the IFS is strongly temperature dependent in cyclic plastic deformation. At low temperature, it is stabilised after 500 cycles, whereas at high temperatures, hardening occurs until failure.
- The fatigue life of the IFS decreases strongly below the transition temperature.
- The cyclic plastic behaviour of the IFS is qualitatively equivalent to that of the ultra high purity iron with controlled C and Mn additions in terms of surface crack initiation and propagation.
- At room temperature, all investigated alloys, including IFS, exhibit a behaviour characteristic of a transition regime between "high" and "low" temperature ones.
- Carbon in solid solution slightly decreases the transition temperature in UHP alloys.
- The transition temperature of the IFS is a bit lower that the one of UHP $\alpha$-iron. The carbon concentration in solid solution in IFS being lower than this of Fe-UHP, it must be concluded that the presence of interstitial atoms alone cannot explain the shift of $T_0$ to lower temperatures.
- A comparison between IFS and C-doped Fe-UHP alloys with or without manganese addition show that, in general terms, all deviation from the ideal composition and microstructure of UHP $\alpha$-iron (interstitial or substitution atoms, precipitates, …) is susceptible to decrease the transition temperature by limiting the difference between the mobility of screw and edge dislocations in the b.c.c. lattice.

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