

Mechanism of the fatigue life increment due to dynamic strain ageing during hold time

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The effect of hold time on the low-cycle fatigue behaviour of AISI 4140 steel at 303 to 473 K was investigated in air and argon gas. All tests were conducted under a constant total strain amplitude of 0.5, 0.65, 1.0, 1.3 and 1.6×10^{-2} with zero mean level. Experimental results show that dynamic strain ageing is occurring during the hold time period and that piled-up dislocations near the carbides are locked by the Cottrell atmosphere. Formation of the Cottrell atmosphere around dislocations near the carbides induces the redistribution of carbon or carbides in the matrix. These randomly dispersed grown up carbides behave like the increased number of dislocation pile-up sites from which fatigue cracks may initiate. Therefore, as the hold time is applied in fatigue cycles under constant strain amplitude the dislocation density at each site will be lowered to retard the fatigue damage or improve the fatigue life.

1. Introduction

Under low-cycle fatigue condition, the relation between the number of cycles to fracture, N_f , and the plastic strain range, $\Delta\epsilon_p$, is known to be $(N_f)^\alpha \Delta\epsilon_p = \text{constant}$, which is the Coffin–Manson relationship [1, 2]. Rie [3] has shown that, instead of using N_f in the Coffin–Manson relationship, the critical number of cycles to failure, N_{cr} (the number of cycles leading to the formation of crack which noticeably weakens the specimen) can be used for the better fitting of the Coffin–Manson relationship.

When a period of strain or stress hold is applied at the tensile or compressive peak of loading cycles, the degradation of fatigue endurance is frequently observed on stainless steel, Cr–Mo–steels and superalloys [4, 5].

In the ASME Code Case 1592, a “linear damage summation rule” has been suggested on the basis of assumptions that fatigue and creep do not interact with each other, and if the linear sum of

each damage reaches a critical value, then the material fails. The linear damage summation rule has been applied to 304 and 316 stainless steel, $2\frac{1}{4}$ Cr–Mo steel and Incoloy 800, but it is proved to be inadequate for life prediction especially when the creep and the fatigue interact non-linearly [6–9].

Three phenomenological models have been proposed for prediction of fatigue life under combined creep-fatigue damage. The first is the “frequency-modified Coffin–Manson law” by Coffin [10, 11], the second is the “strain range partitioning method” by Manson [12, 13] and the third is “damage functions based on hysteresis energy” by Ostergren [14, 15]. In addition to the above three phenomenological models, there are two more models suggested on the basis of the micromechanistic considerations for life prediction under combined creep–fatigue damage. One is proposed by Tomkins and Wareing [16] and the other is “interactive damage rate equations”

by Majumdar and Maiya [17–19]. In the practical application of these models, however, a lot of difficulties lie in obtaining various parameters and constants in the equations.

Applied hold time on loading cycles usually reduces fatigue life, however there are some exceptions e.g. hold time applied at compressive loading extends fatigue life in 18–8 type stainless steel [2], applied hold time for 2¼ Cr–Mo steel accelerates the precipitation of Mo₂C thus increasing fatigue life [21], and Plumtree *et al.* [22] and Antolovich *et al.* [23] have tested Fe–Ni–Cr alloys and Rene 80, respectively, to observe less damage in combined creep–fatigue than that of the summation of creep and fatigue damage independently.

Some of the microstructural changes during the fatigue of steels with different ageing treatments were reported by McGrath and Bratina [24]. They observed that metastable carbide platelets smaller than 30 nm obtained by ageing at ≤ 333 K were reduced below a critical size by the shearing action of dislocations and eventually became small enough to be dissolved into the matrix, giving precipitate-free channels of easy slip. Carbides larger than 300 nm obtained by ageing at 473 K were stable and acted as dislocation sources giving a rather uniform dislocation distribution throughout the matrix. The effects of strain ageing on fatigue behaviour in differently aged specimens were reported by Wilson *et al.* [25–27]. They found the amount of cyclic hardening in an as-quenched low carbon steel increased with the concentration of dissolved carbon atoms which contributed to dynamic strain ageing. Cyclic strain ageing led to the precipitation of fine carbide particles in the fatigue aged matrix and to the abnormal growth of cementite particles at the slip band/matrix interfaces and at slip band intersections. Hence, when hold time is applied at the fatigue loading cycles, the effect of strain ageing during the hold time may occur and it may affect the fatigue life. To achieve a better understanding of the effect of hold time on low-cycle fatigue, a typical tension–com-

pression fatigue test was conducted at the low temperature range with the low alloy carbon steel.

2. Experimental procedures

2.1. Experimental set-up

To see the effect of hold time in low-cycle fatigue, an Instron Model 1362, creep-fatigue testing machine was used. To control the constant total strain amplitude and to prevent possible bending of the specimen during compression, two extensometers (HBM Co, type DD1) were installed outside the gauge length, because the gauge section was sealed by the specially designed heating chamber.

2.2. Specimen preparation

AISI 4140 steel was used as the specimen, and its chemical composition (wt%) is as follows: 0.42 C, 0.93 Cr, 0.17 Mo, 0.21 Si, 0.74 Mn, 0.022 P and 0.011 S.

A round bar with a diameter of 19.4 mm was machined into unnotched hour-glass specimens having a shoulder radius of 10 mm, a nominal gauge length of 20 mm and a diameter of 11 mm. The specimens were annealed at 1150 K for 1 h in a vacuum and quenched in oil at room temperature and then tempered in a vacuum at 873 K for 1 h. To make the surface of the gauge length smooth, the specimens were mechanically polished and then chemically polished using a solution of a mixture of 5 HF: 70 H₂O₂.

2.3. Experimental conditions

The following experimental parameters were chosen to study the effect of hold time on low-cycle fatigue.

(a) Atmosphere: air and argon gas of 1 atm.

(b) Temperature: 303 to 473 K.

(c) Total strain amplitude ($\Delta\epsilon/2$): 0.5, 0.65, 1.0, 1.3 and 1.6×10^{-2} with zero mean level.

(d) Hold time, strain rate and frequency: to see the effect of these parameters the following loading schedules are applied for each cycle:

F_n	Straining raising time (sec)	Maximum strain hold time (sec)	Strain lowering time (sec)	Minimum strain hold time (sec)	
F_1	3.0	0.1	3.0	0.1	without hold time
F_3	3.0	30	3.0	30	with hold time
F_6	3.0	60	3.0	60	with hold time

From the above test schedules, the data for the hysteresis curve, the cyclic flow curve, N_f , N_{cr} and the stress relaxation curve are obtained, and the transition modes of fatigue without hold to that with hold and with hold to without hold are also observed.

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used for observation of the fracture surfaces, and the carbide morphology and dislocation structures, respectively.

3. Experimental results and discussion

The microstructure of the heat treated test specimen is the tempered martensite with average prior austenite grain size of $30\ \mu\text{m}$. Fig. 1 shows the TEM structure of the tempered martensite. Directionally precipitated carbide particles whose size is in the order of 10 to 100 nm are shown in the ferrite matrix in which dislocations are observed. Observation of similar carbide distribution and dislocation was also reported by Thielen *et al.* [28] for AISI 4140 steel after similar heat treatment to this investigation.

The tensile properties of the specimen at 303 and 473 K are shown in Table I and the SEM fractographs for the fractured surfaces are shown in Figs. 2a and b respectively. As one can see from the fractographs and the results of tensile tests, the specimens have a significant amount of ductility, showing ductile failure. The effect of testing temperature on the tensile properties

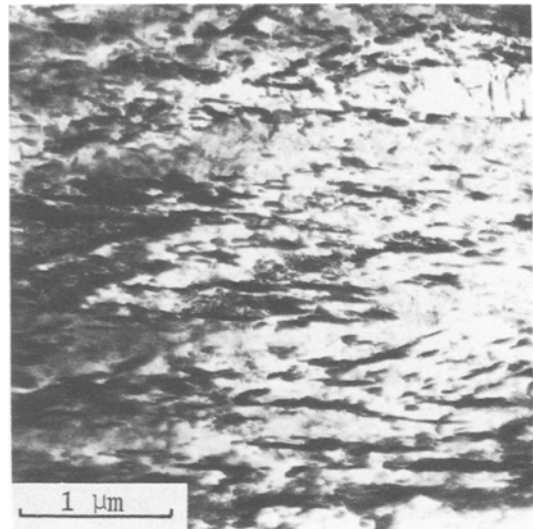


Figure 1 Transmission electron micrograph of the tempered martensitic structure.

is not significant but at the higher temperature the specimens have slightly greater ductility.

In the frame of the suggested experimental conditions, the planned fatigue tests have been performed and their results are plotted in Fig. 3 for plastic strain range ($\Delta\epsilon_p$) against critical number of cycles to failure (N_{cr}), where $\Delta\epsilon_p$ is the plastic strain range at $N = \frac{1}{2}N_{cr}$. From Fig. 3 one can realize many interesting phenomena:

(i) For a given strain range at low temperature (303 K), tests performed in air show a similar life to those performed in argon. However, at

TABLE I Tensile properties and various parameters for the Coffin–Manson plot in Fig. 3

Temperature (K)	Atmosphere	σ_{ys}^1 (MPa)	σ_{uts}^2 (MPa)	σ_f^3 (MPa)	ϵ_f^4	F_n	σ_f^5 (MPa)	ϵ_f^6	b^7	c^8	N_t^9
303	Ar	1070 ± 10	1190 ± 10	800 ± 40	0.20	F_1	1092	1.66	-0.076	-0.717	1828
	Ar					F_3	1016	0.45	-0.066	-0.510	3155
	air					F_1	1117	2.21	-0.080	-0.757	1807
	air					F_3	1125	0.77	-0.082	-0.583	3045
473	Ar	1000 ± 10	1050 ± 10	650 ± 40	0.25	F_1	-	2.32	-	-0.760	-
	Ar					F_3	-	0.54	-	-0.536	-
	air					F_1	-	1.75	-	-0.741	-
	air					F_3	-	0.68	-	-0.578	-

Elastic modulus, $E = 8.1 \times 10^4$ MPa for Ar 303 K, 473 K and H_2 303 K; and $E = 7.6 \times 10^4$ MPa for H_2 473 K.

¹ σ_{ys} equals 0.002 offset monotonic tensile yield stress (strain rate = 1.07×10^{-2} sec⁻¹).

² σ_{uts} is the ultimate tensile strength.

³ σ_f is the fracture strength.

⁴ ϵ_f is the fracture strain.

⁵ σ_f is the fatigue strength coefficient, intercept of $\log \Delta\sigma$ against $\log N_{cr}$ plot at $N = 1$.

⁶ ϵ_f is the fatigue ductility coefficient, intercept of $\log \Delta\epsilon_p$ against $\log N_{cr}$ plot at $N = 1$.

⁷ b is the fatigue strength exponent, slope of $\log \Delta\sigma$ against $\log N_{cr}$ plot.

⁸ c is the fatigue ductility exponent, slope of $\log \Delta\epsilon_p$ against $\log N_{cr}$ plot.

⁹ N_t is the transition fatigue life cycles where $\Delta\epsilon_p = \Delta\epsilon_e$.

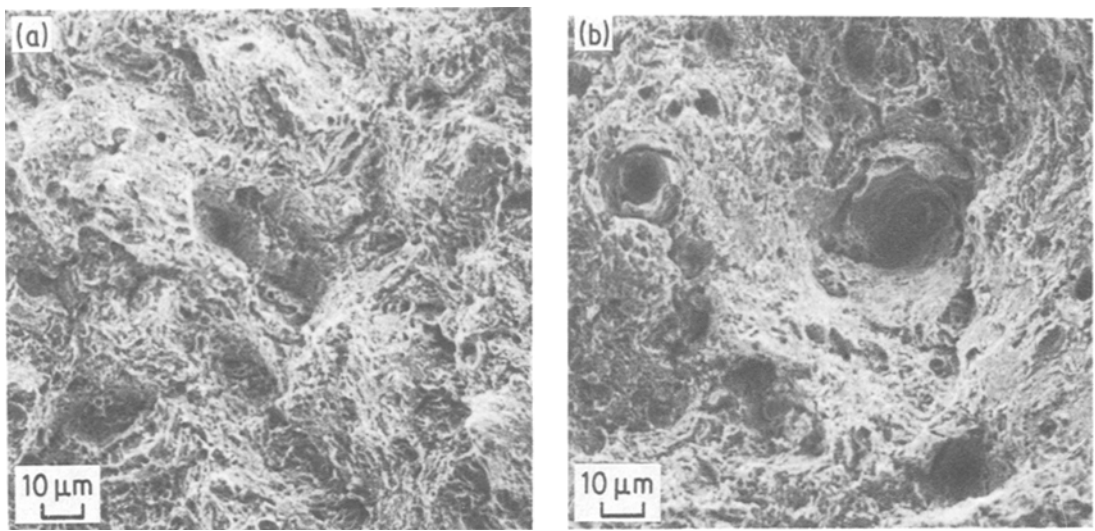


Figure 2 Scanning electron micrographs for the fractured surfaces by tensile test in argon gas (a) at 303 K and (b) at 473 K.

higher temperatures (473 K) N_{cr} is reduced in air but not in argon,

(ii) As the hold time at the maximum and minimum strain is applied, the life is extended in both air and argon for the two temperature ranges.

One can easily understand the reason for the reduced life in air, since it is well known that the oxidation at the surface may assist crack initiation and N_{cr} may have a lower value at the higher temperature when the increment of temperature is not high enough to promote ductility but just

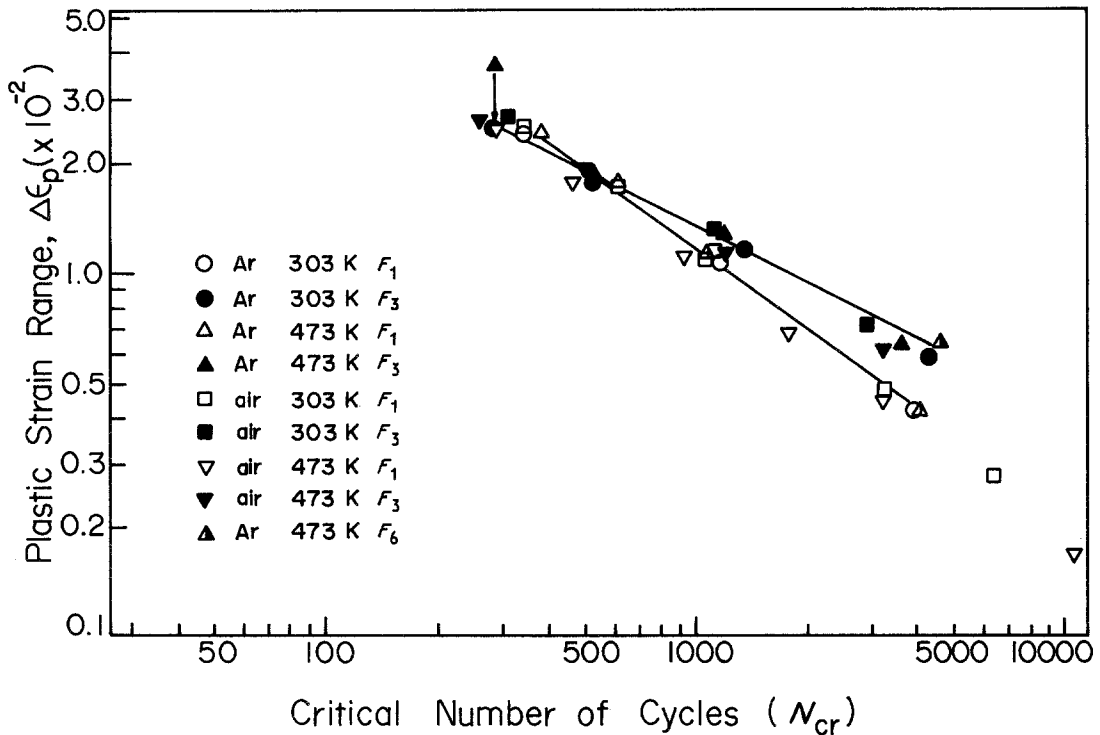


Figure 3 Modified Coffin-Manson plot of plastic strain range against critical number of cycles in air and argon at 303 and 473 K with or without hold time.

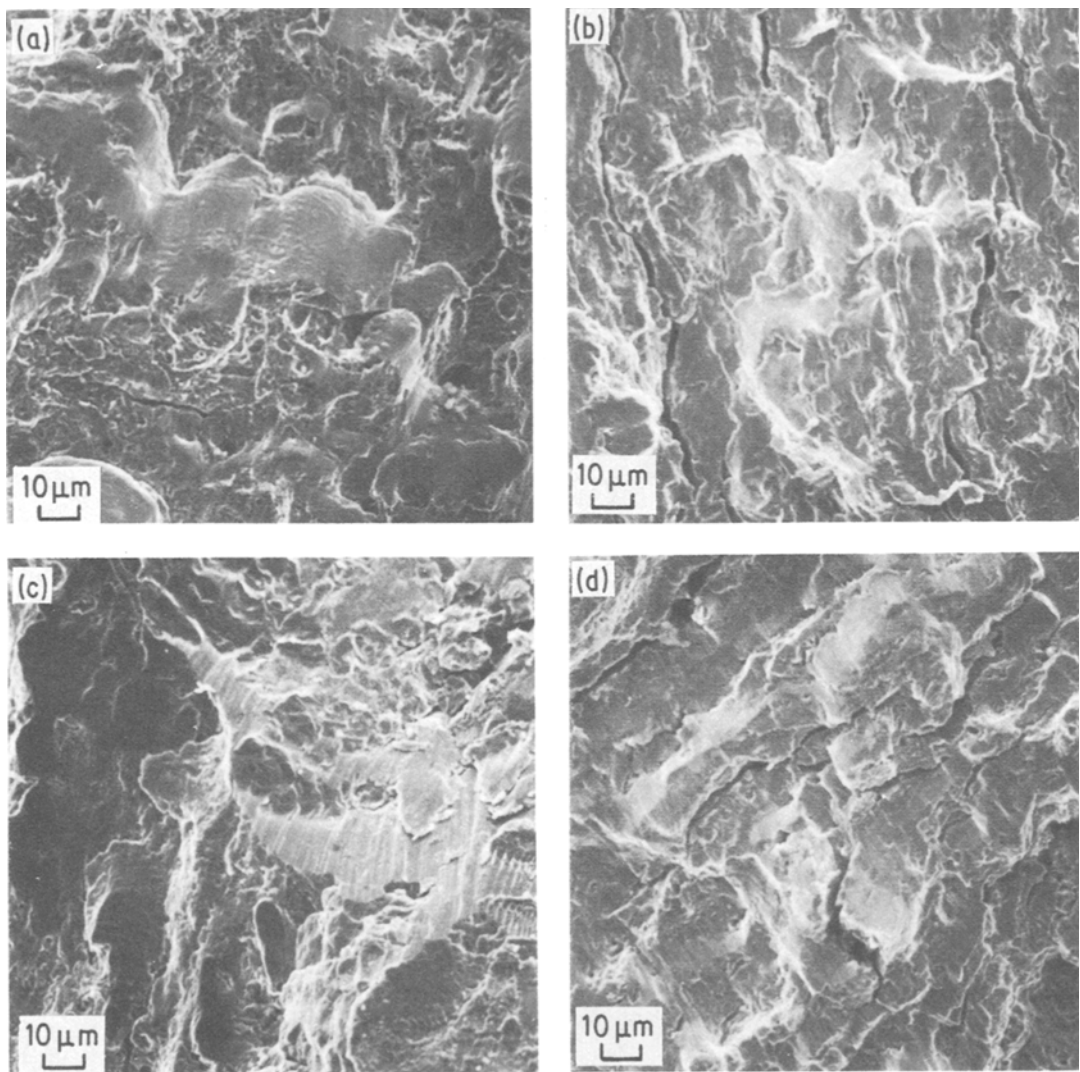


Figure 4 Scanning electron micrographs of the fatigue fracture surfaces in an argon atmosphere with a plastic strain range of 6.5×10^{-3} . (a) Without hold time (F_1) at 303 K, (b) with hold time (F_3) at 303 K, (c) without hold time (F_1) at 473 K, (d) with hold time (F_3) at 473 K.

enough to enhance oxidation during fatigue. However, it is not easy to understand the reason why the N_{cr} is extended with hold time. One possible consideration for the investigation of this phenomenon is that there will be some processes which overcompensate the reduction of N_{cr} due to oxidation or creep damage during the hold time period. Among the possible processes, an increase in ductility due to recovery during hold time period is considered. To investigate this possibility, the fracture surfaces of the specimens fractured with or without hold time have been observed, but no sign of improved ductility with hold time is observed (Figs. 4 and 5).

To find an alternative interpretation, we started the test without hold time and monitored the peak stress variations. Applying the hold time after a certain number of cycles of a simple fatigue test, the peak stress was increased, removing the hold time, the peak stresses were recovered for the original trend (Fig. 6). To see what is happening during this hold time period, the related stress during hold time period has been plotted against the test temperature (Fig. 7). In argon, the stress relaxation decreases with increasing temperature.

To see the mode of crack propagation more clearly, SEM photographs have been obtained for

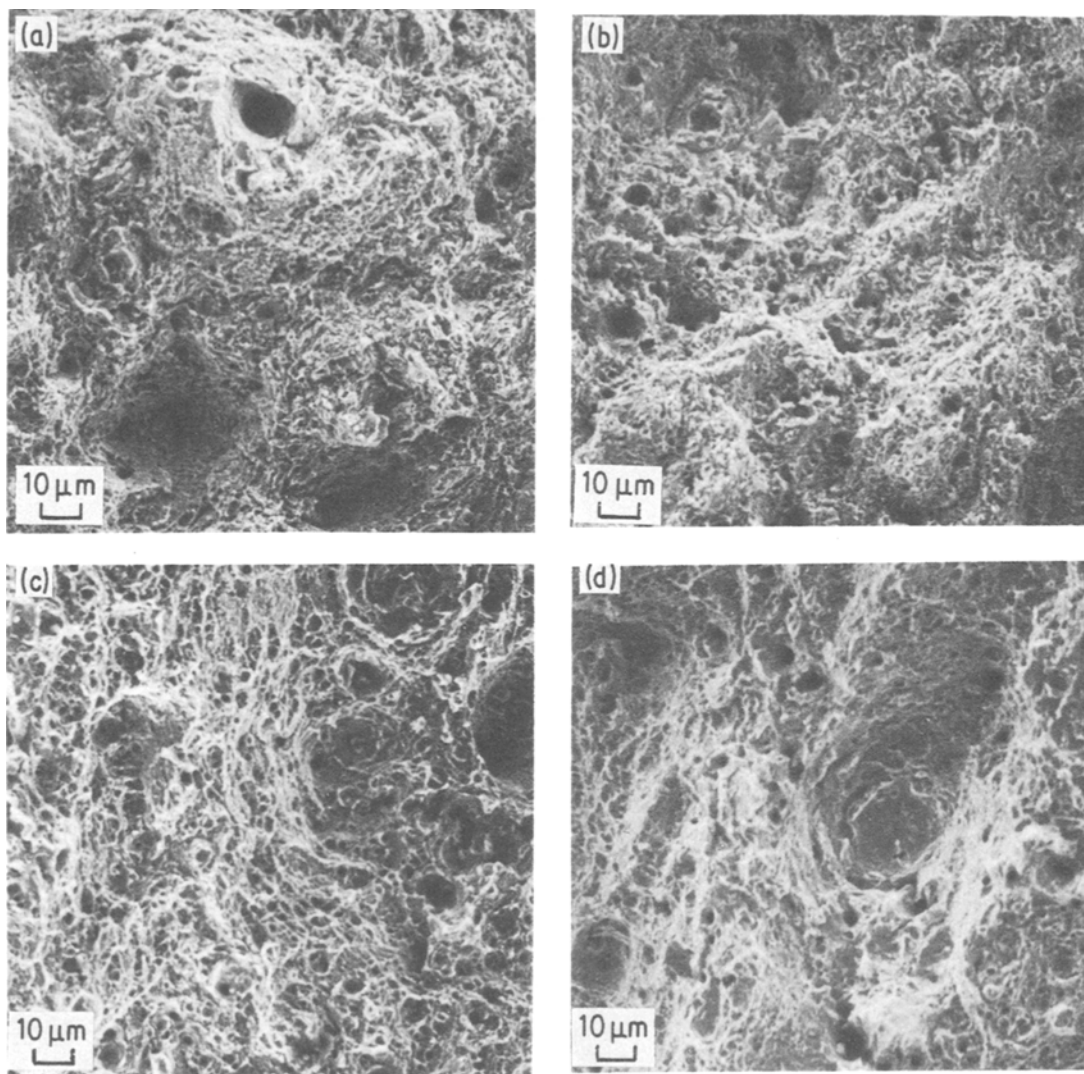


Figure 5 Scanning electron micrographs of the fracture surfaces by an overload tensile test during fatigue testing in an argon atmosphere with a plastic strain range of 6.5×10^{-3} . (a) Without hold time (F_1) at 303 K, (b) with hold time (F_2) at 303 K, (c) without hold time (F_1) at 473 K, (d) with hold time (F_2) at 473 K.

the fractured surfaces of the specimens tested under a plastic strain range of 6.5×10^{-3} simply because at this low strain range the difference in fatigue life with or without hold time was observed to be very significant. In Fig. 4 the mode of crack propagation is shown for two test temperatures with or without hold time. Figs. 4a and c show the fractured surfaces tested at 303 and 473 K, respectively, without hold time. These indicate that the main crack is propagating in a mixed intergranular and transgranular mode. Figs. 4b and d show the fractured surfaces at 303 and 473 K, respectively, but with hold time. In these cases many secondary cracks are observed,

i.e. secondary cracks are developed when hold time is applied.

During the applied strain hold period, the load is observed to be relaxing with time and this may be influenced by time dependent dislocation movement, cavity formation, crack growth, solute rearrangement, etc.

The ratios of N_{cr}/N_f against hold time in argon are plotted in Fig. 8. The ratio of N_{cr}/N_f is increased with increasing hold time both at 303 and 473 K, and the ratio at 473 K is higher than that at 303 K with the same hold time. A higher value of the ratio means that the initiation of the unstable growth of the main crack is harder,

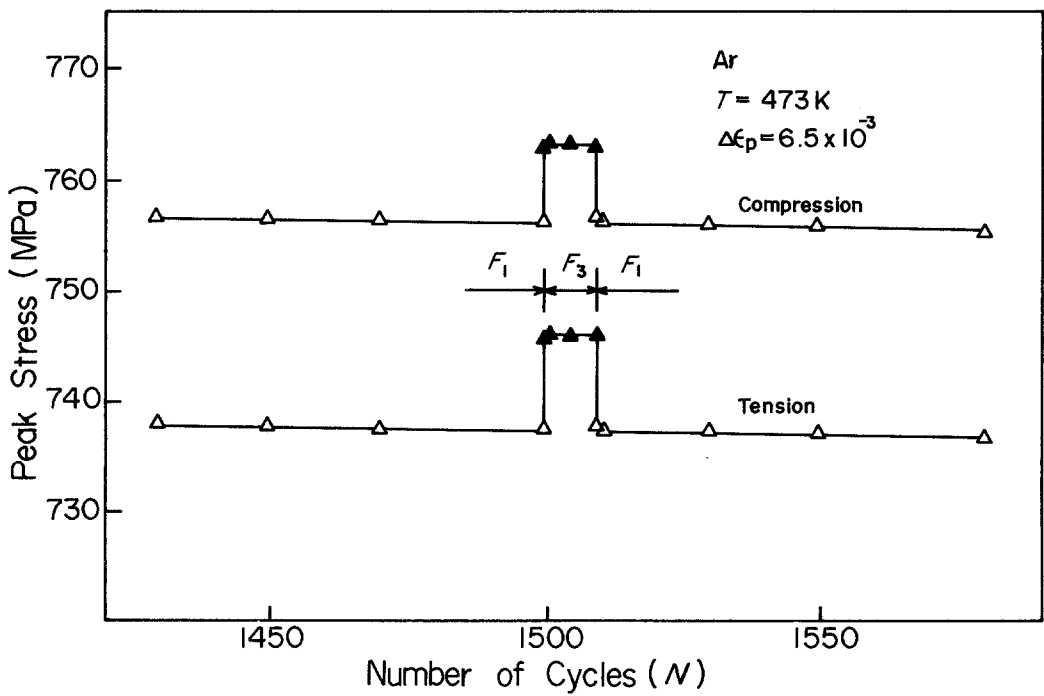


Figure 6 Increment of tensile and compressive peak stress due to strain holding in argon with the plastic strain range of 6.5×10^{-3} .

and this seems to be related to the formation of many secondary cracks.

The question of why hold time increases fatigue life, encourages development of secondary cracks and increases the ratio of N_{cr}/N_f may be answered after consideration of the effect of dynamic strain ageing during hold time.

As previously mentioned, one can see from Fig. 3 that hold time increases the fatigue life. However, there are numerous experimental obser-

vations which indicate that hold time reduces the fatigue life [4, 5]. Therefore, it is very interesting for us to investigate the mechanism by which the fatigue life is extended with hold time. At the higher plastic strain range (1.2×10^{-2}), it is observed that hold time does not affect the life significantly. However, at a low plastic strain range (6.5×10^{-3}) hold time increases the life significantly.

For a mechanistic investigation of the reason

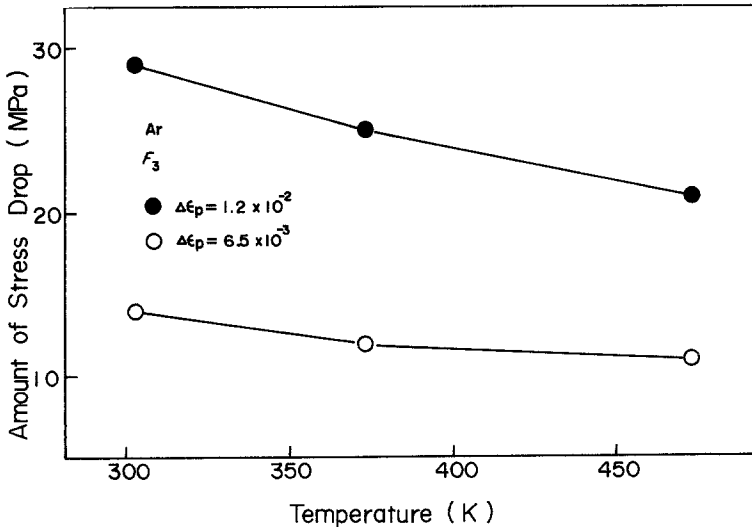


Figure 7 Variation of the amount of stress drop during tensile strain holding time of 30 sec at three different temperatures in argon with plastic strain ranges of 6.5×10^{-3} and 1.2×10^{-2} .

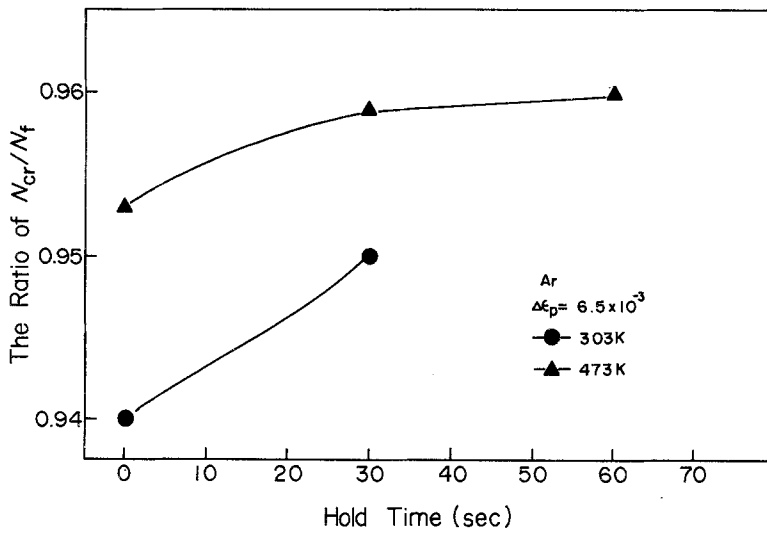


Figure 8 The ratio of N_{cr}/N_f against hold time in argon with a plastic strain range of 6.5×10^{-3} .

why the peak stresses are increasing, we have conducted two sets of tests such that simple fatigue test (F_1) has been interrupted at zero stress for a certain length of time and restarted. Hold time (ageing time) is applied at zero stress level to reduce the prestrain effect on peak stress increment, which may occur as the form of stress relaxation during the hold time period. At the first quarter of one cycle, the tensile peak stress has been observed to increase with increasing ageing time. In Figs. 9 and 10, the increment of tensile peak stress after ageing under zero stress level has been plotted against the ageing time for the total strain amplitudes of 6.5×10^{-3} and 1.0×10^{-2} (in terms of plastic strain range these correspond to 6.5×10^{-3} and 1.2×10^{-2} , respectively). As one can see from the figures, the stress

increase is time dependent and at a given time the increment of stress becomes higher with higher temperature. This may mean that the process which is responsible for the increment of stress due to ageing, is a thermally activated time dependent process. According to Friedel's treatment [29] which is based on the report of Cottrell and Bilby [30] and to the results of Harper [31], Ham [32] and Wycliffe *et al.* [33], the stress increment due to ageing, $\Delta\sigma$, is such that

$$\Delta\sigma = C_1(Dt/T)^{2/3}$$

or

$$\Delta\sigma = C_2 \left[\exp\left(-\frac{Q}{RT}\right) t/T \right]^{2/3}$$

where C_1 and C_2 are constants, D is the diffusion coefficient, t is ageing time, Q is the activation

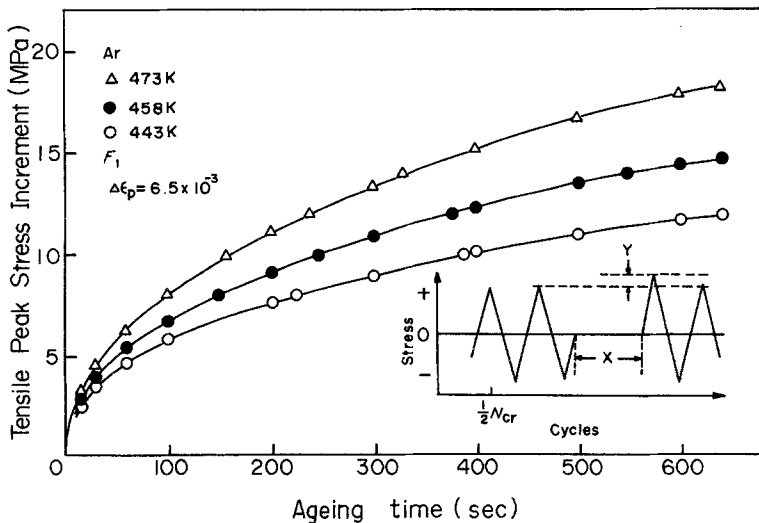


Figure 9 Increment of tensile peak stress due to holding at zero stress against ageing time in argon. The plastic strain range is 6.5×10^{-3} .

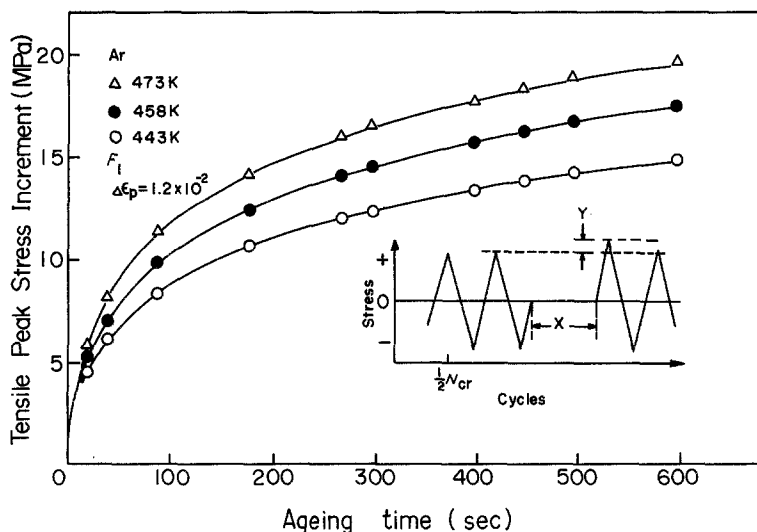


Figure 10 Increment of tensile peak stress due to holding at zero stress against ageing time in argon. The plastic strain range is 1.2×10^{-2} .

energy and T is the ageing temperature. Therefore, by taking the times at which the tensile peak stress increment reaches 4, 6, 8, 10, 12 and 14 MPa for three different temperature ranges, respectively, the activation energies for the process, which is responsible for the increasing peak stress after ageing, can be calculated. Each activation energy at the given peak stress increment level has been calculated from the slope of the plot of $\ln(T/t)$ against $1/T$ in the above equation. The results are plotted in Fig. 11 for plastic strain ranges of 6.5×10^{-3} and 1.2×10^{-2} , respectively. As the peak stress increment increases or the ageing time increases, the activation energy is also increasing to approach about 65 kJ mol^{-1} . This activation energy is believed to be that of the process responsible for the increasing peak stress after ageing.

In reaction kinetics, it is well known [34] that the apparent activation energy, Q_{app} is the difference between thermal and athermal activation energy and it is expressed as follows.

$$Q_{app} = Q_{thermal} - Q_{athermal}$$

The athermal energy can be expressed at [35]

$$Q_{athermal} = \sigma V^*$$

where σ is the applied mechanical stress and V^* is the activation volume. Lord and Beshers [36] have reported that the measured activation energy for carbon diffusion in stress-free iron is about 80 kJ mol^{-1} . Gilifalco and Welch [37] showed that the activation energy for diffusion is reduced with the straining. The measured activation energy for the process is about 40 kJ mol^{-1} with short ageing time. During this short ageing period the internal stress may be high enough to

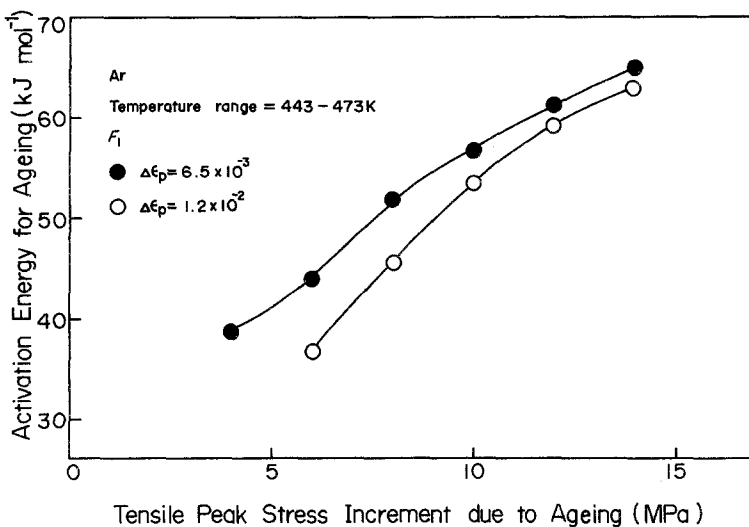


Figure 11 Variation of the activation energy for the tensile peak stress increment in argon. The plastic strain ranges are 6.5×10^{-3} and 1.2×10^{-2} .

influence strongly the athermal component of the activation energy. One also can see from the plot that the measured activation energy is always lower for the specimen tested with higher strain amplitude. This may also mean that the athermal effect is more significant in the more deformed specimen. On the other hand, the increasing activation energy with increasing ageing time may mean that the athermal effect is decreasing with the help of recovery by the extended heating under zero stress. Therefore, if the athermal effect disappears the apparent activation energy may be approaching the value of thermal activation energy, 80 kJ mol^{-1} .

The experimental observations shown in Fig. 11 may mean that the measured apparent activation energy is indicative of the activation energy for carbon diffusion in iron. In other words, carbon diffusion is one of the possible processes for the increment of the peak stress during the hold time period. The next question then is how the carbon diffusion increases the peak stress. During hold time period, piled-up dislocations at the carbides may not be moving and carbon diffuses to the dislocations to form Cottrell atmospheres. For the next cycle, these locked dislocations may need a higher stress to move away from the atmosphere, which is observed experimentally as the peak stress increment.

We now have another question why the applied hold time increases the fatigue life, and how this extended fatigue life is related to the peak stress increment. For the answer to this question the work by McGrath *et al.* [24] will be referred to. They quenched a Fe-0.08 wt% C alloy and aged it at temperatures of 333 and 473 K. After this heat treatment they fatigue tested by bending. From this test they observed that carbide platelets smaller than 30 nm, which were formed at 333 K, were cut by the dislocation and became small enough to be dissolved into the matrix, giving precipitate-free channels of easy slip. Carbides larger than 300 nm, formed at 473 K, behaved as dislocation sources throughout the matrix giving a rather uniform dislocation distribution. Wilson *et al.* [25–27] also reported that small carbides were dissolved into the matrix and this dissolved carbide was reprecipitated at the slip band/matrix interfaces and at slip band intersections. Thielen *et al.* [28] also observed carbides and locally tangled dislocation structures in fatigued 4140 steel at room temperature. Kalish

et al. [38] reported that, in strain-tempered martensitic steel, uphill diffusion of carbon atoms at dislocations in the severely deformed region is responsible for the increasing yield stress. Nakagawa *et al.* [39] reported that dynamic strain ageing due to solute carbon in carbon steel was responsible for the increasing fatigue limit.

According to our experimental observations and the results from the references mentioned above, during hold time it can be hypothesized that piled-up dislocations at the carbides in the ferrite matrix behave as Cottrell atmosphere forming sites. As the fatigue stress is reapplied at the end of hold time, the dislocations will move away leaving high carbon concentration behind them. Repeating this process, the high carbon content around the carbide will be diffused to the carbide to make it grow. Therefore, the carbides are growing to be redistributed in the ferrite matrix.

To see if our hypothesis for the above mentioned phenomenon is appropriate, we have observed the distribution of carbides after fatigue. TEM micrographs were taken from those specimens fatigued in argon in a plastic strain range of 6.5×10^{-3} at 303 and 473 K, with or without hold time. The photographs are shown in Fig. 12. These photographs show quite a different morphology of the carbide distribution compared to that of the as-tempered specimen (see Fig. 1), i.e. in the as-tempered specimen the carbides are precipitated along a certain direction and distributed fairly uniformly, however, after fatigue the distribution of carbides has changed depending on the test condition. As one can see from Fig. 12, the mode of carbide distribution is not influenced by the test temperature. But it is clearly seen that hold time makes marked differences in the mode.

Without hold time, lath-type carbide free regions have been formed so that the mean free path of the dislocation is long and so fewer dislocation blocking sites exist, leading to more piled-up dislocations in one site under the strain controlled condition. On the other hand, with hold time, carbide particles of a larger size are fairly randomly distributed. In this situation the dislocation mean free path will be very short and there will be many barriers to dislocation motion leading to fewer piled-up dislocations in one site. Therefore, as the hold time is applied on fatigue cycles, the fatigue life is increased due to the retarded main crack initiation. The model expressing these obser-

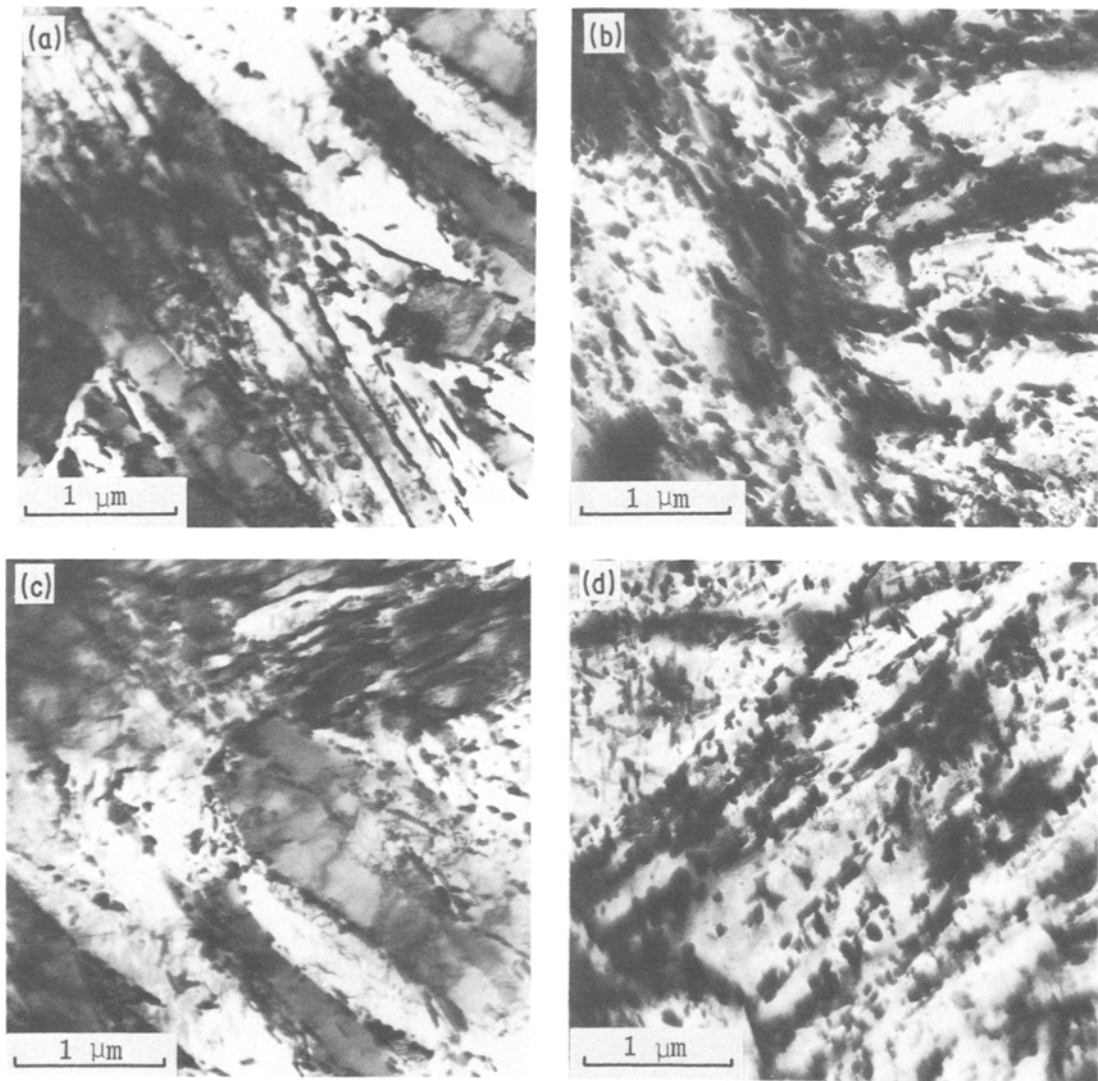


Figure 12 Transmission electron micrographs of the fatigue specimens in an argon atmosphere with a plastic strain range of 6.5×10^{-3} . (a) Without hold time (F_1) at 303 K, (b) with hold time (F_3) at 303 K, (c) without hold time (F_1) at 473 K, (d) with hold time (F_3) at 473 K.

vations and interpretations is shown in Fig. 13, and it can explain why as hold time is applied on fatigue cycles, secondary cracks develop and the ratio of N_{cr}/N_f is increased as shown in Fig. 4. That is, as the hold time is applied on fatigue cycles, carbides grow-up and become dispersed so as to behave as an increased number of dislocation pile-up sites. As a result, the fatigue life is increased, the formation of the main crack becomes harder and the ratio of N_{cr}/N_f is increased. Fatigue cracks may initiate from the increased number of dislocation pile-up sites. As the number of cycles reaches N_{cr} , fine cracks develop as the secondary cracks are randomly distributed, and after

N_{cr} , the main crack propagates across the matrix with many fine secondary cracks. Therefore, the fatigue life could be extended by the hold time to spend more energy for the formation of many small secondary cracks.

4. Conclusions

1. When hold time is applied on fatigue cycles, the peak stress is increased, and this increased peak stress is believed to be related to the formation of Cottrell atmospheres around dislocations by dynamic strain ageing due to solute carbon diffusion.

2. Applying hold time on fatigue cycles at

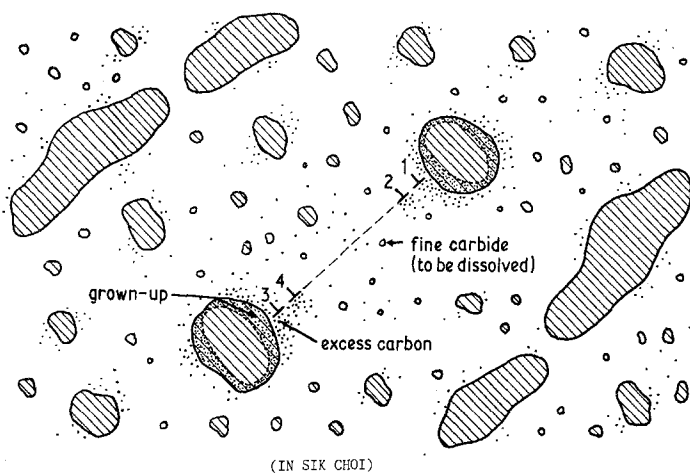


Figure 13 A model for the redistribution of carbide due to dynamic strain ageing during hold time.

temperatures of 300 to 470 K, dynamic strain ageing occurs even more actively during the hold time period, and the piled-up dislocations near the carbides are locked by Cottrell atmospheres to redistribute the carbides randomly and to grown-up carbides in the matrix.

3. Randomly dispersed grown-up carbides act as a barrier to dislocation motion leading to an increasing number of pile-up sites (reducing the number of piled-up dislocations in one site) and in turn to an increase of the fatigue life of 4140 steel under strain controlled condition in air and argon gas.

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