

High-temperature low-cycle fatigue of a nickel-based MAR-M200 + Hf alloy in Ar and Ar + 20% O₂ environment

E. AGHION

Department of Mechanical Engineering, University of Natal, King George V Avenue, Durban, 4001, Republic of South Africa

M. BAMBERGER, A. BERKOVITS

Technion, Israel Institute of Technology, Haifa, Israel.

A systematic study was made on the environmental influences of inert gas and oxygen on the low-cycle fatigue failure of MAR-M200 + Hf unidirectionally solidified nickel-based alloy at 975 °C. The cyclic load was constant and comprised creep tension and plastic compression. Under an inert argon atmosphere, a pure transgranular fracture free of environmental influences was obtained. Under an Ar + 20% O₂ atmosphere the fracture was intergranular along a path through the interdendritic microsegregation zone. In the area close to the propagating crack tip, a uniphase layer was detected, resulting from a process of internal oxidation which developed a preferred oxide-type bonding with the elements making up the principle precipitating phase, γ' , namely titanium and aluminium. The internal oxidation led to changes in the boundary layer between the matrix and γ' phase, as a result of which the original coherence between them was lost. In order to reduce the surface energy between the precipitate and the matrix, a process of growth and coarsening of γ' phase took place in these conditions. The internal oxidation and the formation of the uniphase layer increased the brittleness over a comparatively wide area adjacent to the crack tip. As a result, crack branching and blunting took place during propagation in the oxygen-containing atmosphere. The branching and the blunting were instrumental in lengthening the active life of the alloy in the oxygen-containing atmosphere compared with that in the inert atmosphere.

1. Introduction

Unidirectionally solidified MAR-M200 + Hf alloy is used for turbine blades in the hot region beyond the combustion chambers of jet engines. The service life of these turbine blades depends on a number of factors, namely fatigue, creep, oxidation hot corrosion, erosion and microstructural changes.

The purpose of the present study was to characterize the influence of argon and Ar + 20% O₂ atmospheres on MAR-M200 + Hf alloy under a constant high-temperature low-cycle fatigue (HTLCF) loading regime.

There are relatively few reported investigations on this subject. Duquette and Gell [1] reported that at 760 °C the service life under fatigue loads of single-crystal MAR-M200 alloy in air was nearly the same as in vacuum, whereas at 927 °C the service life in air was longer than in vacuum. Sadanada and Shahinian [2] examined the effects of the enveloping atmosphere (air and vacuum) on the rate of fatigue crack propagation in cast superalloys at 927 °C. They found that the presence of air causes considerable increase in the rate of crack propagation compared with that in vacuum. Manson and Halford [3] studied the character of the fracture obtained in the MAR-M200 alloy under

cyclic loading in air with a creep component. They found that the fracture obtained was intercrystalline. According to these authors, this type of fracture results from the presence of the creep component, which causes creep deformation through a sliding of grain boundaries.

Thus it can be stated that the influence of the environment on the mechanism responsible for fracture under a combination of different loading conditions can be very complex, and very little understanding presently exists on this subject.

2. Experimental procedure

The material was received in the form of unidirectionally solidified 16 mm diameter bars of MAR-M200 + Hf nickel-based alloy, also known as PWA1422. The chemical composition of the material is shown in Table I.

The original bars were turned to produce fatigue specimens in the middle of which there was a constriction 6.35 mm diameter and 25 mm long. Specimen strain was measured with an extensometer in the form of strain transfer rods attached at one end to gauge-length collars on the specimen, and fitted at the other

TABLE I Chemical composition of MAR-M 200 + Hf alloy

	Al	Hf	W	Nb	Ti	Cr	Co	Ni
wt %	4.0	2.4	12.6	1.2	2.5	7.0	9.0	61.3
at %	9.0	0.8	4.2	0.8	3.2	8.5	9.5	64.0

end with a pair of Linear Variable Differential Transducers (LVDTs) located outside the elevated temperature environment. The two LVDT outputs were averaged by a conditioning unit before the signal was fed into the strain-control function of the testing machine. Both strain and load measured by the testing machine load cell were recorded as related functions or as function of time on a two-channel x - y recorder.

In order to isolate parameters and to track the influence exerted by the atmosphere on the failure process in conditions of a combination of parameters, all the experiments were conducted in a constant and uniform loading regime and at a constant temperature of 975 °C. The loading regime was of the creep plasticity (CP) type and followed the strain-range partitioning (SRP) method, the strain range being constant at $\pm 6 \times 10^{-3}$ ($\Delta\epsilon = 0.012$), as shown in Fig. 1. From it a loading cycle that comprised tensioning at the rate of $2.4 \times 10^{-4} \text{ sec}^{-1}$ and compression at the rate of $60 \times 10^{-4} \text{ sec}^{-1}$ is seen to be obtained in the given strain range. This slow tension in effect provides the creep component (C) of the cycle, while the compression delivers its plasticity component (P). It should be pointed out that this loading regime was selected because it contains creep under tension, a fact that exerts great influence on the failure processes in HTLCF.

The thermo-mechano-chemical system serving the HTLCF tests in controlled atmospheres was composed of two principal parts, one comprising the gas mixing subsystem, the other the mechanical subsystem for loading at high temperature in the required atmosphere. With the aid of the subsystem gas mixing, a mixture of different gases at varying concentrations could be obtained. The gas mixture was then fed to a cylinder that was mounted round the specimen constriction. The mechanical subsystem set up was composed of a standard MTS system and an ATS electric furnace. Also, in order to trace the influence of only the gases on the environmental behaviour of the alloy at 975 °C, a corrosion chamber was built.

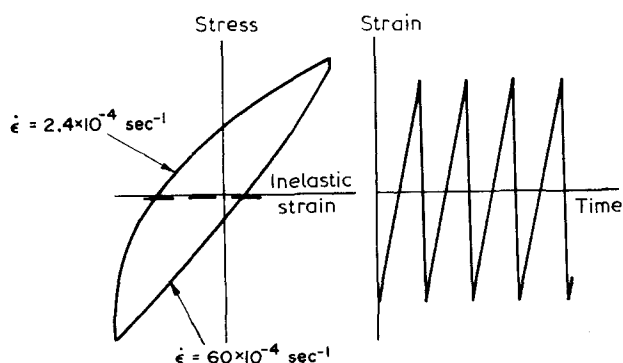


Figure 1 Hysteresis loop for $\Delta\epsilon = 0.012$ at CP = 0.04 Hz in accordance with the SRP method.

The tests were carried out in pure argon and in Ar + 20% O₂. The oxygen concentration in the test, which was higher than that customary in combustion chambers, was set so as to increase the active cross-section for the oxidation processes in the atmosphere being investigated.

3. Results

The microstructure of the unidirectionally solidified MAR-M200 + Hf alloy consisted of long dendrites in the direction of solidification and exhibited dendritic protrusions normal to it (Fig. 2a). Within the space of the dendritic grains was a fine phase of the γ' type (Fig. 2b). The latter was cubic in shape and constituted in all a 95% volume fraction of the alloy. X-ray diffraction analysis obtained from a cross-section normal to the direction of solidification (Fig. 3) showed that there is a rich presence of the principal precipitating phase, γ' , namely Ni₃ (Al, Ti). In addition modular γ' phase of Ni₃Nb and Ni₃Hf and also carbides, especially Cr₂₃C₆, W₂C and WC, were present.

HTLCF experiments at 975 °C in a pure argon atmosphere showed that the average life in that atmosphere is about 620 cycles (each experiment was an average of at least four measurements). A metallographic representation of a cross-section adjacent to the fracture that occurred after HTLCF in an argon atmosphere is given in Fig. 4. This shows that the fracture obtained in argon is transcrystalline; Fig. 4a illustrates the fact that the crack has propagated through the grain and parallel to the region of interdendritic segregation. Fig. 4b, a magnification of a characteristic region in the path of the crack's propagation, exemplifies the microstructure of the area making up the fracture edge, from which it is clear that the crack, propagating through the grain, did not entail any interaction between the atmosphere and the alloy or micro-structural changes in the region close to the crack.

In order to identify and to determine the relative quantities of light elements, e.g. carbon and oxygen, as well as to recognize the chemical state of the elements constituting the alloy, an Auger electron spectroscopic (AES) analysis was made. The pertinent specimens were taken from the fracture region of the fatigue specimen. The specimens were ground at an angle of approximately 10° relative to the fracture surface, as schematically shown in Fig. 5.

The AES analyses were made in three main areas adjacent to the fracture surface of the fatigue specimen. The first, marked "F" (free surface), represents the fracture surface itself. The second area, marked "I" (internal phase), represents the region underneath the fracture surface and closest to it. The third region, marked "B" (bulk), is from an area at some distance from, and unaffected by, the fracture surface. It serves mainly as a reference for the analyses of the other regions. The purpose of this analytical method is to track the changes in the concentrations of the elements and their chemical state as functions of their locations relative to the fracture surface. With this method the interaction between the gaseous

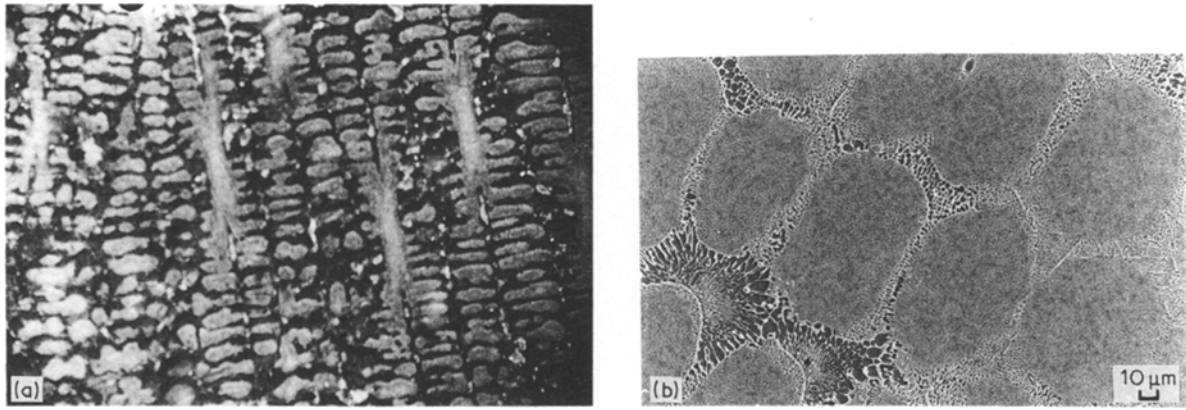


Figure 2 Typical cross-section microstructure of unidirectionally solidified MAR-M200 + Hf alloy: (a) longitudinal, $\times 34$, (b) transverse.

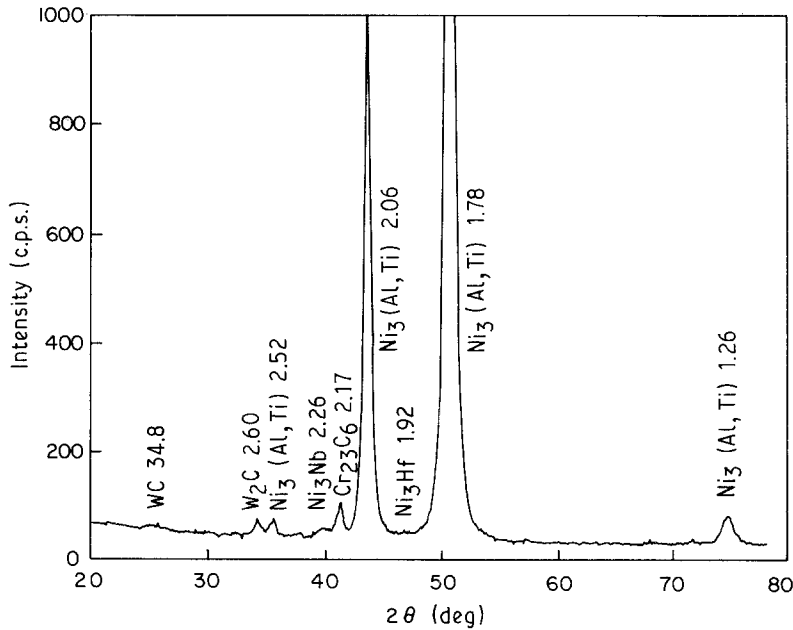


Figure 3 X-ray diffraction using $\text{CuK}\alpha$ radiation, of the bulk materials (MAR-M200 + Hf) on cross-section transverse to the direction of solidification.

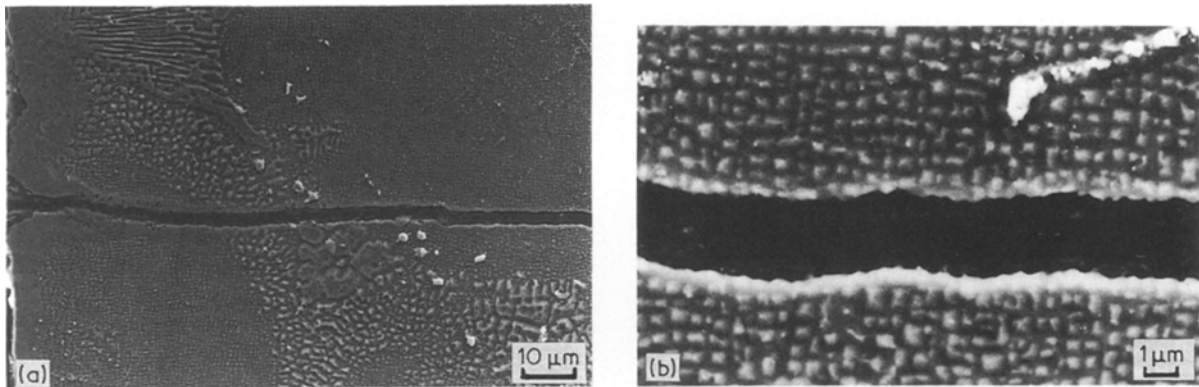


Figure 4 HTLCF crack in an argon environment at 975°C .

atmosphere and the surface at high temperature can be studied [4, 5].

AES analyses of the compositions of the layers examined in the cross-section affected by the fracture following HTLCF in an argon atmosphere at 975°C , are given in Fig. 6. The main and most conspicuous finding, as shown by this diagram, is that practically no differences are detectable in the composition and in the relative quantities of the elements making up the

various zones of the section (B, I and F, as defined above).

In tests made in an $\text{Ar} + 20\% \text{O}_2$ atmosphere it was found that the average service life after HTLCF at 975°C is 765 cycles. (Each experiment was an average of at least four measurements). Fractographic examination made in this atmosphere shows that no single central crack could be detected but, rather, a number of parallel cracking processes took place

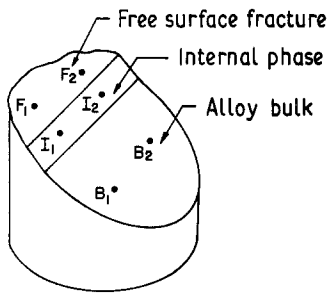


Figure 5 Points on fracture surface examined by Auger analysis.

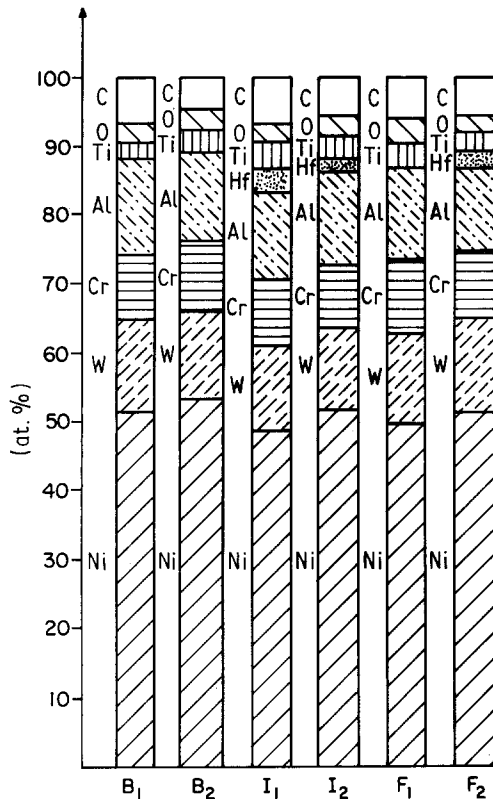


Figure 6 Auger analysis of element concentrations in different layers of the cross-section of the fracture surface after HTLCF in an argon environment at 975 °C. (F = free surface, I = internal phase, B = bulk.)

(Fig. 7a). These multiple processes are characteristic of the atmosphere under study and may indicate that it provides conditions favourable to cracking.

The results of the metallographic examination of

the section adjacent to the fracture after HTLCF at 975 °C in an Ar + 20% O₂ atmosphere are given in Fig. 7b. This shows that the crack produced in that atmosphere exhibited a tendency to propagate in the zone of interdendritic segregation and was mainly of the intercrystalline type. The preferred region for crack initiation was the area in which the zone of interdendritic microsegregation encountered the surface. Another result evident from these figures is the fact that the cracks propagating in the Ar + 20% O₂ atmosphere tended to branch. It is also seen that within the space of the crack there is a strong presence of porous oxides, which were instrumental in the blunting of the crack tip.

In order to ascertain the nature of the layers composing the fatigue crack edges, energy dispersive spectroscopic (EDS) analyses were made in the zones close to the edges, as shown in Fig. 8, from which it is seen that the edges of the fatigue crack obtained in the Ar + 20% O₂ atmosphere were made up of three main layers. The first, the closest to the crack edge, consisted of porous oxides rich mostly in reactive elements, such as aluminium and hafnium (as compared with the original composition, shown in Table I). Below the first layer is the second, a uniphase one, in which processes of growth and coarsening of γ' phase had taken place. In this layer the concentration of elements such as aluminium and titanium which make up the γ' phase, is seen to have been largely preserved as it had been in the bulk. The third layer's microstructure is characteristic of the areas of interdendritic segregation. This fact points to the segregation areas constituting a preferred path for crack propagation in the subject atmosphere.

The quantitative results of the AES analyses made in the crack-affected cross-section following HTLCF in an Ar + 20% O₂ atmosphere at 975 °C are given in Fig. 9. The outer fracture surface (zone F) is seen to be oxygen-rich, the average oxygen concentration therein being about 35 at. %, compared to an average content of 9 at. % in zone I and at 3 at. % in zone B. This finding shows that the fracture surface had been exposed to oxygen at a high temperature, leading to an interaction between the oxygen and the constituent elements of the alloy. A further important conclusion that can be drawn is that the internal phase, which is parallel with the uniphase detected in the crack edge

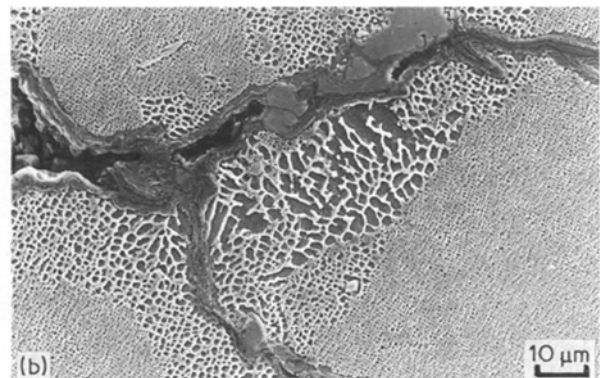
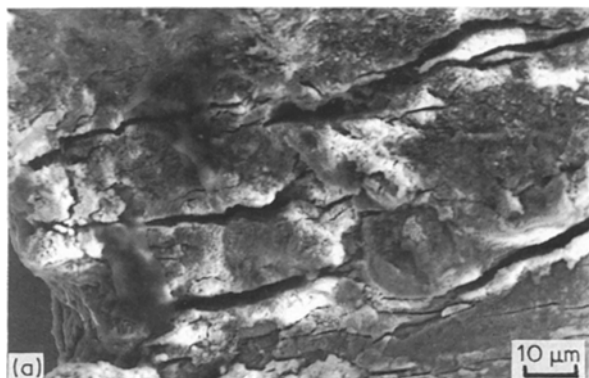


Figure 7 Scanning electron micrograph after HTLCF failure in Ar + 20% O₂ environment at 975 °C. (a) Fractographic examination, (b) metallographic examination.

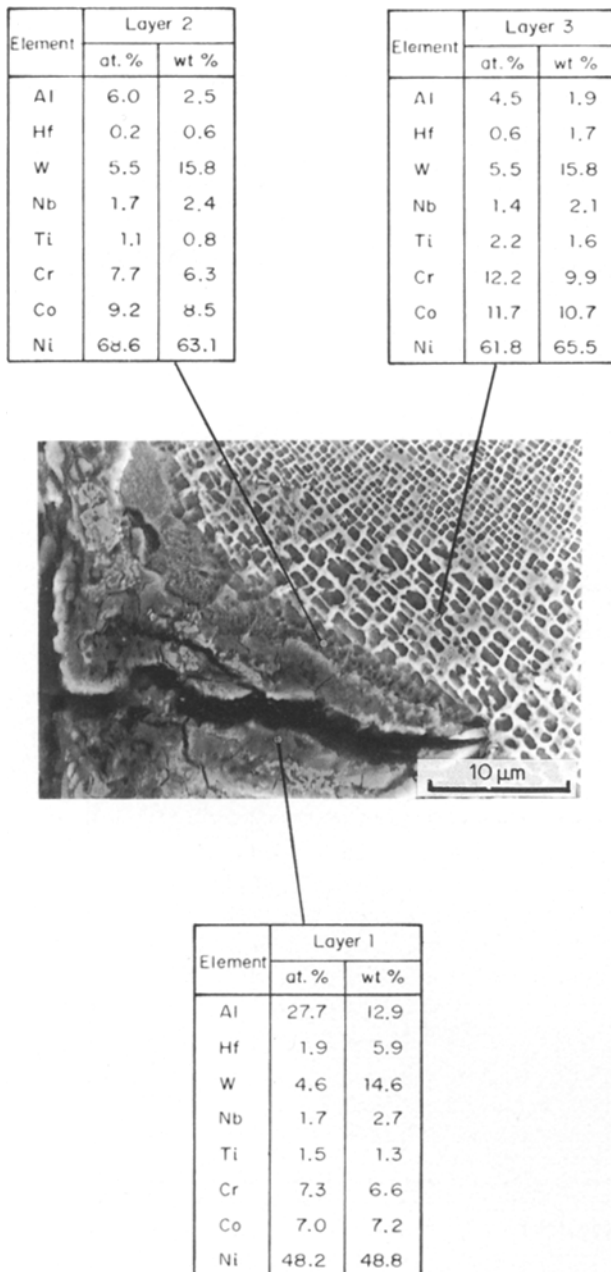


Figure 8 Microstructures and chemical compositions of the different layers beneath the crack surface after HTLCF in an Ar + 20% O₂ environment at 975 °C.

by metallographic examination, had dissolved a relatively large part of the oxygen. This oxygen uptake by the internal phase is termed “internal oxidation”. The uniphase that may develop at the crack tip during crack initiation and subsequent propagation is one of the important factors in determining the character of the failure mechanism and that of the crack propagation.

Fig. 10 shows the characteristic spectra obtained by AES analysis of a cross-section close to the fracture surface after HTLCF in an Ar + 20% O₂ atmosphere. This serves to describe the nature of the chemical bonds of the various elements constituting the alloy in that region. In Fig. 10a a prominent oxygen peak testifies to that element’s abundant presence, while the nature of the nickel and titanium peaks indicate that these elements are present in the form of oxides [6].

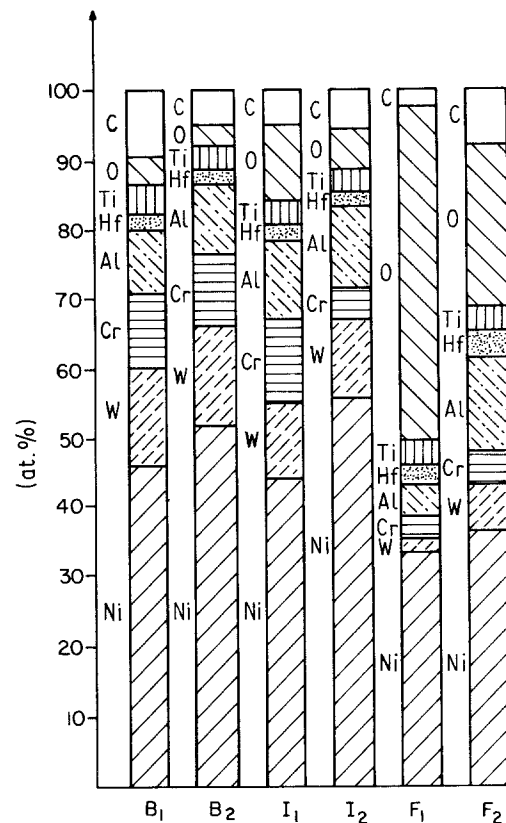


Figure 9 Element concentrations by Auger analysis of different layers on a cross-section of the fracture surface after HTLCF in an Ar + 20% O₂ atmosphere at 975 °C. (F = free surface, I = internal phase, B = bulk.)

Fig. 10b shows a characteristic spectrum taken in zone I, in which a clear oxygen peak stands out, again signifying the rich content of that element in the region. Furthermore, it is seen that chromium, aluminium and titanium exhibit a predominantly oxide bond, whereas the other elements bear the character of a metallic bond. The carbon in this region shows a carbide bond, stemming from the carbides present in the original alloy. Hence it follows that the zone of the internal phase, the “uniphase”, mainly contained oxides of the reactive elements, titanium and aluminium, which belong to the γ' phase, as well as oxide of the other reactive element, chromium, which is one of the constituents of the matrix. The spectrum obtained in zone B is shown in Fig. 10c. It indicates a relatively low presence of oxygen, while the elements of which the alloy is composed exhibit the original metallic bond. The carbon shows a carbide bond.

The X-ray analysis of a specimen made from a section perpendicular to the direction of solidification and exposed to an atmosphere of Ar + 20% O₂ in the corrosion chamber at 975 °C elicited the following facts (Fig. 11). There is a strong presence of the main precipitate phase, γ' , namely Ni₃(Al, Ti), as well as of the nodular γ' phase, Ni₃Nb, and characteristic carbides of the types Cr₂₃C₆ and Cr₃C₂. In addition the oxides of the elements making up the precipitate γ' phase are strongly represented: Al₂O₃, NiAl₂O₄, HfO₂ and NbO₂. Also there is a presence of oxides of the elements composing the matrix, namely Cr₂O₃, NiCrO₄, and NiWO₄.

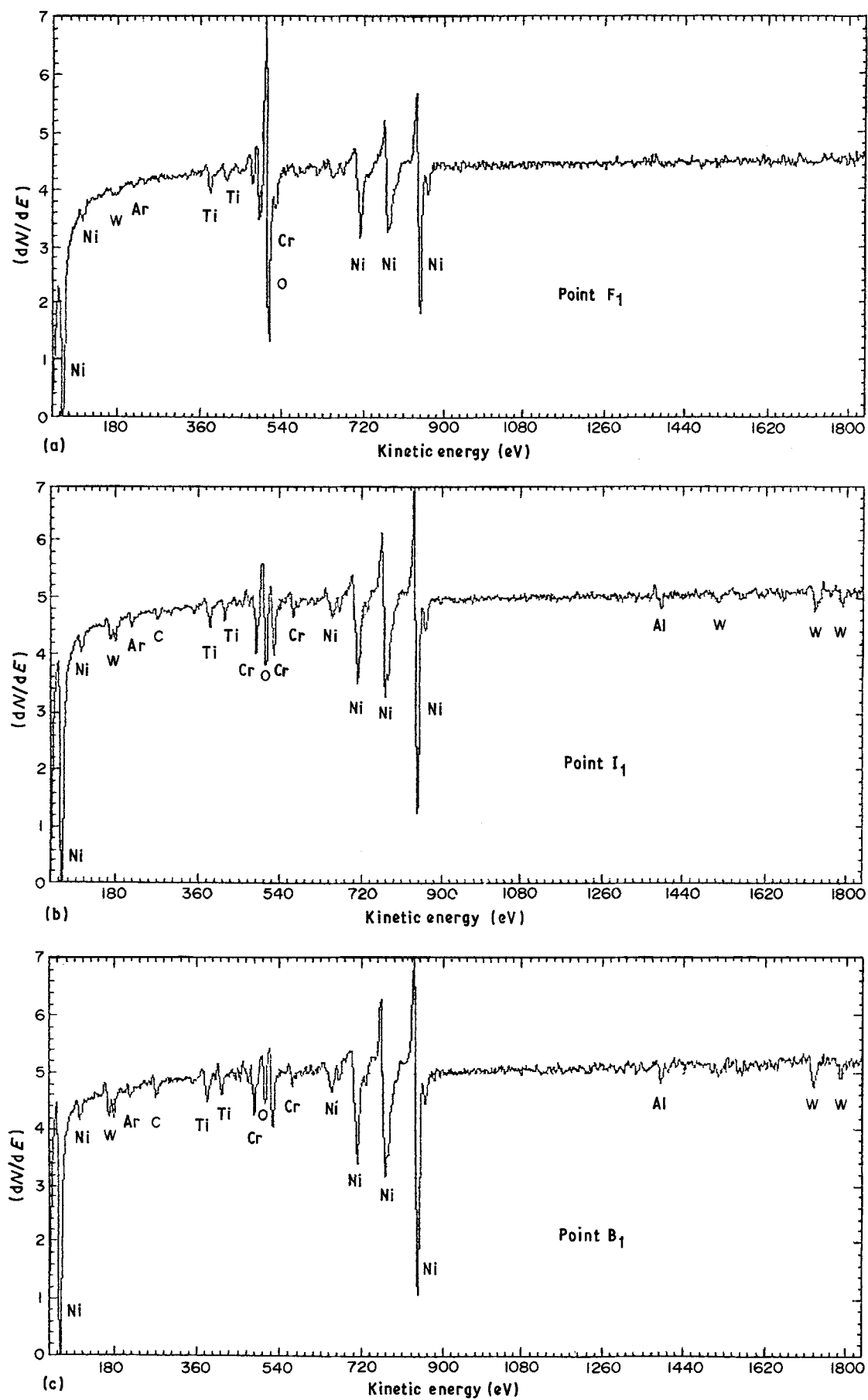


Figure 10 Auger spectra of the different layers in a cross-section of the fracture surface after HTLCF in Ar + 20% O₂ at 975°C. (a) Free surface, (b) internal phase, (c) bulk.

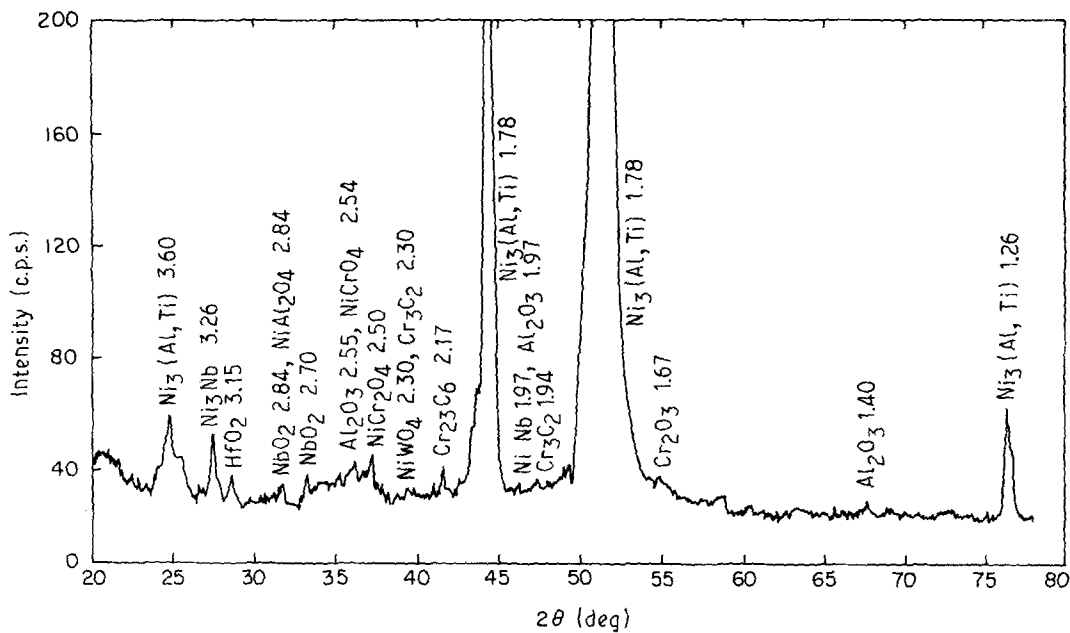


Figure 11 X-ray diffraction using $\text{CuK}\alpha$ radiation of MAR-M200 + Hf exposed to an Ar + 20% O_2 environment in the corrosion chamber at 975°C.

From the above it can be deduced that most, but not all, of the constituent elements of the alloy appear in the outer layer in their oxidized state, the exception being the oxides of titanium and nickel, of which no peaks figured in the X-ray analysis, although the presence of these oxides in an ostensibly parallel region, the F region, was shown by the AES analysis. This discrepancy may stem from differences in the experimental conditions of the specimens before the analyses: the specimen for the AES analysis had previously undergone HTLCF in an Ar + 20% O_2 atmosphere at 975°C, so that it represented results obtained by composite parameters and in kinetic conditions. The specimen subjected to X-ray analysis had resided in the corrosion chamber in the same atmosphere and at the same temperature, but without having undergone mechanical loading, so that it was, in effect, received in a state closer to thermodynamic equilibrium. It follows that the products obtained in kinetic conditions may not be the same as those that are closer to thermodynamic equilibrium. This dissimilarity is caused mainly by differences in the rate and the kinetics of growth dictated by the experimental conditions.

4. Discussion

The discussion will focus mainly on the crack tip in combined conditions, because it is these that are responsible for the rate and the nature of crack propagation and in effect determine the resulting failure mechanism. Two main basic assumptions underlie this discussion. One rests on the fact that a concentration of stresses exists at the crack tip and on the conditions thereof, the other, which bears some relation to the first, holds that the reaction taking place at the crack tip is kinetically directed and not at equilibrium.

According to this assumption relaxation phenomena occur at the crack tip in reaction to the

developing stress concentration. These phenomena are divided into two main types, namely chemical relaxation and mechanical relaxation. The chemical relaxation is manifested in the interaction between the alloy and the constituents of the gas of the surrounding atmosphere. Mechanical relaxation finds expression in the development and growth of the crack. Other manifestations are possible as, for example, the growth and coarsening of phases in the boundary layer between the bulk of the material and the zone adjacent to the crack edge.

The conditions applying in this research included cyclic loading with creep fatigue of the creep plasticity (CP) type by the strain range partitioning (SRP) method within a constant range of $\pm 6 \times 10^{-3}$ at a constant temperature of 975°C in an atmosphere of either argon or Ar + 20% O_2 .

The failure in the pure argon atmosphere was marked by the absence of any chemical reaction between the atmosphere and the alloy, that absence being made manifest by the fact that no layers of a different microstructure were formed at the crack edge and by the further fact that the AES analyses of the sloping section of the fracture were almost completely identical. The preferred path of the crack propagation in the pure argon atmosphere was clearly transdendritic. These results in a pure argon atmosphere differ substantially from those obtained by Manson and Halford [3] as well as from those of Bill *et al.* [7]. The addition of 20% oxygen to the argon atmosphere was reflected in the resulting failure mechanism characteristic of oxidizing conditions, which differs substantially from that in inert environments. Under oxidation the failure mechanism is a complex one that encompasses a number of different processes.

Generally speaking it can be seen that the failure which occurred in the Ar + 20% O_2 atmosphere involved intercrystalline cracking in the region of interdendritic microsegregation. That type of failure may

be explained on the basis of the strong chemical affinity between Hf and oxygen, namely HfO_2 , in the form of a porous oxide layer. The removal of Hf from the zones of interdendritic segregation through oxidation causes a reduction in the cohesion among the grain boundaries, exposing the alloy to creep deformation, e.g. by grain-boundary sliding. Hence it may be concluded that the presence of oxygen weakens the interdendritic zones. The cracking process advances preferentially through these zones, leading to an intercrystalline fracture.

Underneath the layer of loosely packed oxides is the uniphase layer, which was not found in the experiment carried out in pure argon. The formation of the uniphase layer can therefore be attributed to the internal oxidation process and to the preferred bonding of the oxygen to the reactive elements, titanium and aluminium, making up the γ' phase, as shown by AES analysis. The internal oxidation processes also leads to changes in the nature of the bonds between the precipitate and the matrix, whereby the interface between them is weakened. In more extreme cases of internal oxidation the precipitates may be entirely decomposed with an attendant material change in the interfacial microstructure [8]. These assumptions explain the second phenomenon connected with the uniphase layer, namely the growth and coarsening of its principal precipitating phase γ' .

All these processes and changes together are the cause that the uniphase layer does not become etched during the metallographical examination but appears as a uniform and continuous layer. From this it may be concluded that the uniphase is not depleted of γ' , as is claimed in the literature [9, 10]. It is, on the contrary, a layer in which changes in the morphology of the principal precipitating phase have taken place as a result of internal oxidation and coarsening. This conclusion is strengthened by the fact that the concentration of the main elements composing the chief precipitating phase, γ' , is preserved. Additional characteristics of the failure in an Ar + 20% O_2 atmosphere are the branching and the blunting of the crack. The branching, too, may be explained as being a result of the oxidation of the elements composing the alloy by a mechanism of internal oxidation in the zone adjacent to the crack tip. These interactions result in the relative embrittlement of the zone referred to, enhancing its sensitivity to the propagation of the crack. If the zone is sufficiently large, the sensitivity to cracks spreads in a number of directions, and the crack accordingly splits up into several branches. It is stressed that this branching involves processes of mechanical relaxation of the stress field developing at the crack tip. The blunting of the crack tip originates in the growth kinetics of the oxides which fill the crack space. It is also emphasized that the blunting appears to be taking place mainly after the crack has propagated and new areas have been exposed to oxidation.

On the strength of the reactions so far described, a characteristic failure mechanism under oxidizing conditions is suggested. According to this mechanism the incubation stage of the cracking process is that in

which internal oxidation takes place and the uniphase begins to develop. Oxygen diffuses through the matrix and combines with the reactive elements of γ' . The preferred zone for this occurrence is the area in which the interdendritic microsegregation reaches the surface. At a later stage an oxide spike develops in this area, supported by the diffusion of the reactive elements. The spike, which forms in the interdendritic area also contains oxides of the elements making up the γ' , such as Al_2O_3 , HfO_2 , NbO_2 and TiO_2 , as well as oxides of the constituents of the matrix, e.g. Cr_2O_3 , NiCr_2O_4 and NiWO_4 . The cracking of the oxide spike during cyclic loading forms the initiation stage of the crack in the material. This happens preferentially along an interdendritic path due to the removal of hafnium from the area (by the oxygen), as a result of which the interdendritic segregation zone is subjected to characteristic creep deformation processes. The oxide spike, which cracked during the cyclic loading, constitutes the critical nucleus for the intercrystalline crack propagation.

During the propagation of the crack, all the processes described above take place locally in the plastic zone adjacent to the crack tip. The events at the crack tip in oxidizing conditions and at high temperature serve as chemical and mechanical relaxation processes for the stress field developing in this area in the conditions described. Chemical relaxation sets in following the interaction between oxygen derived from the atmosphere and the elements making up the alloy. That interaction is mainly manifested by the creation of oxides and by internal oxidation. The mechanical relaxation is chiefly reflected in the development of the crack, the presence of coarsening processes in the uniphase layer, and the appearance of branching at the crack tip. Most of the processes described arise mainly during tension in creep conditions, whereas the compression step in conditions of plasticity does not lead to a restoration of the state of things before these processes. At the same time, as the crack continues to grow and together with the branching, sufficient time is left for the formation of oxides and their condensation in the crack space. This condensation of oxides manifests itself in the appearance of blunting which, together with branching, restrains the growth rate of the crack towards failure. The sequence of events in the process of cracking under oxidizing conditions is summarized schematically by the illustration given in Fig. 12.

The general finding resulting from the present research, that the presence of 20% oxygen in the surrounding atmosphere involves an increase in the effective service life of the alloy compared with that in an inert atmosphere, is due to a retardation of the effective growth rate of the crack towards failure. That retardation in turn is due to the branching of the crack and the simultaneous occurrence of blunting in the space of the crack at its tip.

5. Conclusions

The main conclusion to be drawn from the present research is that the surrounding atmosphere exerts

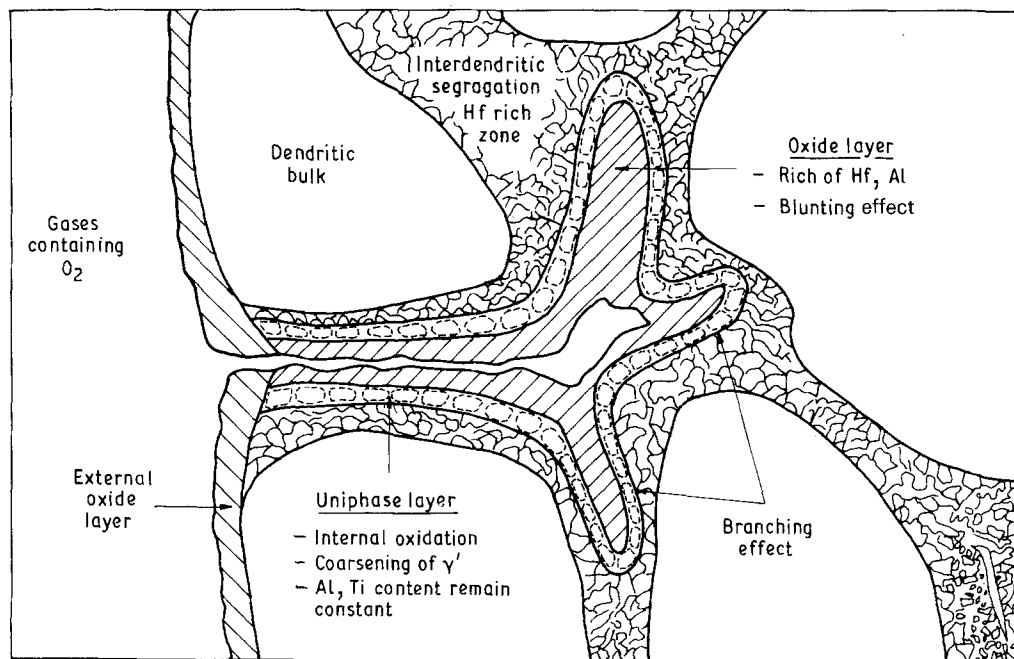


Figure 12 Schematic illustration of the sequence of events accompanying crack propagation in oxygen-containing environments.

a decisive influence on the mechanism and nature of the failure in low-cycle fatigue (CP mode) of the unidirectionally solidified MAR-M200 + Hf alloy. The addition of 20% oxygen to the inert argon atmosphere leads to an increase in the service life of the alloy. From the results and discussion it seems to be clear that the totality of the phenomena and changes at the edge of the crack and in the area adjacent to its tip, originate in chemical and mechanical relaxation processes of the material system arising in reaction to the stress concentration and the presence of the atmosphere in these regions. The chemical relaxation is the result of an interaction of the alloy's constituent elements with those of the atmosphere surrounding it. That interaction may be manifested in the accumulation of oxides in the space of the crack and by internal oxidation, which lead to the formation of a uniphase layer at the crack edge. The term "internal oxidation" refers to a process in which the oxygen penetrates through the surface and underneath it preferentially creates oxide tape ligands with the reactive elements of the alloy in a matrix having metal bonds. The mechanical relaxation may be reflected in the development and growth of cracks, plastic deformation, and a coarsening of the main precipitating phase, γ' , in the zone of the uniphase.

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