The influence of the gaseous environment on the crack initiation mechanisms has been studied on copper specimens loaded in cyclic creep conditions in air and in vacuum. Detailed metallographic examination of specimens surfaces by scanning electron microscopy have revealed an environmental influence on the crack initiation process: in vacuum a creep type damage mechanism induces wedge crack initiation at grain boundaries, while in air the rupture is produced by transgranular cracks growing from microcracks initiated in persistent slip bands.

INTRODUCTION

The influence of gaseous environments on fatigue crack initiation remains a controversial subject [see D.J. Duquette (1) for a general review]. If some authors [Thompson et al (2), Broom and Nicholson (3)] thought that gaseous atmospheres strongly affect the nucleation processes, some others [Wadsworth and Hutchings (4), Laird and Smith (5)] related the increase of fatigue lives in inert atmospheres to the crack propagation stage and considerably reduced the effect of gaseous atmospheres on crack nucleation as negligible.

To specify the stage from which environment plays a role on the fatigue damage process and the mechanisms of cyclic deformation which could be modified, we undertook a study of the cyclic damage features of a polycrystalline copper cycled in vacuum and in air (oxygen and water vapor in air being damaging constituents for copper (4)).

This paper illustrates environment induced differences on damage mechanisms in cyclic creep deformation conditions.

EXPERIMENTAL CONDITIONS

Bars of OFHC copper (12 mm in diameter) were used to obtain cylindrical specimens with a gauge length of 20 mm and a diameter of 4 mm. Specimens were electropolished before receiving an annealing treatment for 3 h at 630 °C in vacuum. The subsequent

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mean grain diameter obtained is 0.015 mm. All specimens were electropolished once more just before the tests.

Tests were carried out on a servohydraulic testing machine, at room temperature, in the laboratory air and in an environmental chamber providing a vacuum lower than $10^{-6}$ Pa.

A cyclic load was applied with a load ratio $R = \frac{P_{\text{min}}}{P_{\text{max}}} = 0.1$

The loading amplitude was slowly increased during several cycles at a low frequency until the desired stress level was obtained; then, the loading amplitude was held constant for the remainder of the test and the frequency was increased to 70 Hz.

The cyclic load was applied either up to the specimen failure or to different fractions of the expected life. The data recorded during the test were the maximum load and the total extension strain.

**EXPERIMENTAL RESULTS**

**Cyclic creep deformation**

A cyclic creep deformation process occurs under the loading conditions. The plastic strain is accumulated in a monotonic manner during the test and can be expressed as a function of time or of the number of cycles.

Fig. 1 illustrates the relationship between the cyclic creep and the number of cycles in air and in vacuum for a maximum engineering stress of 165 MPa.

The cyclic creep curves reveal the following features

1) the initial parts of the curves are the same in both environments,

2) the curve in vacuum shows a steady-state stage of plastic deformation while this stage is not observed in air.

It can be noticed that in these experimental conditions and for all the tests, the maximum true stress increases with the reduction in diameter of the specimen due to monotonic deformation. In the example illustrated by Fig. 1, $P_{\text{max}}$ reaches a value of 180 MPa at the end of the loading sequence, 196 MPa at the rupture of the specimen in air and 224 MPa at the rupture in vacuum.

**Number of cycles to failure in air and in vacuum**

Fig. 2 shows the variation of the fatigue lives in air and in vacuum with the maximum applied engineering stress.

In general only one experiment was conducted at a particular stress level with an exception for tests conducted at 165 MPa. For this stress level the scatter bands are representative of 7 tests in air (mean life: $4 \times 10^6$ cycles) and 4 tests in vacuum (mean life: $24 \times 10^6$ cycles).

Dark points in Fig. 2 correspond to quasi-static fractures.
following necking of the specimens. No evidence of crack propagation by fatigue is found in this case.

Considering the scatter in the results, significant differences in fatigue lives in air and in vacuum are observed only for stresses less than 170 MPa. In this case, specimens fail by the propagation of a crack initiated on the surface and fatigue lives can be 6 times higher in vacuum than in air.

**Damage features in specimens cycled to failure**

The surface damage of the failed specimens in air and in vacuum was characterized by Scanning Electron Microscopy. The common general features of the plastic deformation in both environments are:

a) an important elongation of the grains associated with grain boundary depressions at the specimen surface.

b) in most grains, fine slip perforations, typically associated with extrusions and intrusions. In this case, only one slip system is activated.

c) in some other grains, persistent slip bands (PSB's), associated with extrusions and intrusions. In this case, only one slip system is activated.

However, important differences of crack initiation and propagation features, are observed with the environment. Examination of the fracture surfaces shows that, at the rupture in vacuum, the fatigue cracks are still superfluous, while in air the fatigue crack grows across about half the specimen section. This results from the higher values of the maximum true stress induced by a greater elongation of the specimens tested in vacuum and in this case a very little propagation of the crack into the bulk is sufficient for producing the rupture.

On the surface of specimens tested up to failure in air, a few secondary cracks can be observed extending over about ten grain diameters (Fig. 5). Some microcracks which did not extend beyond grain boundaries are also visible in persistent slip bands. Observations of secondary cracks features indicate that in air, stage II fatigue cracks start from microcracks initiated in slip bands and grow transgranularly under the action of normal stresses.

On the other hand, examination of the surfaces of specimens tested in vacuum (Fig. 6) reveals a great number of cracks 3 or 4 grain diameters long distributed all along the gauge length. These microcracks are localized at grain boundaries, with a mean length in the same range as the grain size, about 100-200 microns.

Observations made inside the specimens after cutting across its length, confirm the observations made on the surface for specimens tested in air, with regard to the size and the localization of the cracks. However, examination of longitudinal cross sections of specimens tested in vacuum reveals that, firstly, the numerous microcracks observed on the surface at the grain boundaries have not traversed further than the first grain and secondly, internal microcracks are present at grain boundaries inside the specimen.
Evolution of the damage with the number of cycles in air and in vacuum.

The differences in the nature of damage reported above concern specimens tested up to failure in air and in vacuum and therefore subjected to different numbers of cycles.

At a maximum engineering stress of $\sigma_{\text{max}} = 165$ MPa, for which significant differences in fatigue lives are observed in air and in vacuum (see Fig. 1), some specimens have been cycled up to $0.5 \times 10^6$, $10^6$, $2 \times 10^6$ and $4.3 \times 10^5$ cycles in both environments and up to $6.3 \times 10^6$, $9 \times 10^6$ and $11.10^6$ in vacuum.

S.E.M. examinations of these specimens were carried out either immediately after cycling or after having subjected the specimen to a tension test to open any pre-existing microcracks. Observations on the same area before and after this tension test showed that this loading action modified the aspect of the macrocracks without creating any further ones.

These tests have shown that in a first time persistent slip bands are formed in air as well as in vacuum and it was not possible to detect any environment induced differences in their number or distribution.

On the surface of the specimens loaded up to $0.5 \times 10^6$ cycles, intrusions are observed in both environments but it is difficult to assert that any microcracks are present. However, during the tension test, for the specimens tested in air, microcracks tend to open in PSB’s while in vacuum a few microcracks are opened in grain boundaries.

These observations show that environment induced differences in the nature of crack initiation process, appear at very early stages of the fatigue life.

With an increase in the number of cycles, the general aspect of the surface does not evolve much in air. A few deep microcracks are formed in the grains in persistent slip bands (Fig. 3) and some of them propagates in stage II localizing the deformation in a few zones (Fig. 5).

In vacuum, the number of microcracks at the grain boundaries increases with the number of cycles. The density of wedge cracks at isolated triple points increases without any propagation (Fig. 4). At about $10^7$ cycles these microcracks link together over a range of 2 or 3 grain diameters (Fig. 6).

**DISCUSSION**

At 70 Hz and at a maximum engineering stress above 180 MPa, cyclic creep leads to quasi-static fracture of the specimens. At a lower stress level, fracture is produced by a fatigue process with the propagation of a crack from the specimen surface.

Cracks initiate always at the specimen surface. This results from the action of two factors:
i) the conditions of stressing induced by the existence of a free surface modifying the mechanical behaviour of the surface grains.

ii) the reactivity of the gaseous atmosphere on the metal surface and specially on the new surfaces created by cyclic deformation. It is evident that this is the factor governing the nature of the damage and leading to the differences found in the two environments.

The important question is : why should under some conditions of cyclic loading microcracks initiate in PSB's in air, while the initiation takes place in grain boundaries in vacuum?

One possible reason is that in air, preferential oxidation at grain boundaries can lead them to harden during the initial stages of cycling. However, some complementary experiments shown that a recycling in air do not avoid the formation of grain boundary cracks when the test is continued in vacuum.

Another hypothesis is that the stress-strain levels at the grain boundaries are higher in vacuum due to the different nature of damage occurring in the slip bands.

S.E.M. observations carried out on specimens just cycled in air and in vacuum do not show any environment induced differences in PSB's features. However some physicochemical mechanisms occurring in these bands must be different since PSB's play a very different role in air or in vacuum.

Indeed, if a specimen cycled under vacuum conditions is then loaded in air or stocked in the air atmosphere, we have observed the formation of a thick oxide layer on the extrusions-intrusions sites. This has not been observed on the specimens tested in air; these observations show a higher reactivity of the PSB's created in vacuum and therefore that they differ at a microscopic scale from those created in air.

The following explanations of the fatigue damage features in air and in vacuum, are proposed:

In a first time, PSB's are formed in both cases in a few grains at the surface which allow the accommodation to cyclic deformation. But, in the vicinity of the surface, the stress and strain conditions and the eventual presence of oxygen give rise to new mechanisms.

In vacuum, none oxidation process limits the slip reversibility. But the accumulation of cyclic deformation in the PSB's can produce high stress level in the vicinity of grain boundaries. Moreover, the extension of the specimen entails additional shear stresses which lead to stress concentrations at the triple junctions, and finally microcracks are initiated at the grains corner. The number of these microcracks increases with the number of cycles up to they link together.

In air, the oxygen action on the steps formed by the slip bands at the surface, prevents the slip reversibility and increases the stress concentration in the PSB's leading to the initia-
tion of microcracks. Once formed, the stresses at the crack tip are sufficient to generate fatigue-crack propagation from some of them. The absence of microcracks in the grain boundaries, can be explained by the localized plastic strain which takes place in the PSB's leading to a reduction of the stress concentration at the joints.

CONCLUSION

The analysis of the results obtained by tension-tension tests in air and in vacuum with polycrystalline copper specimens leads to consider two different mechanisms of crack initiation

1) cyclic loading entails the formation of PSB's which become in air preferential sites for crack initiation; the propagation of these microcracks leads rapidly to fracture.

2) cyclic creep deformation associated with a continuous elongation of the specimen induces shear stresses at the joints and produce microcracks initiation at the triple points. This second mechanism prevails in the vacuum environment.

REFERENCES


Figure 1: Cyclic creep curves in air and in vacuum. $\sigma_{\text{max}} = 165$ MPa

Figure 2: Relation between the maximum engineering stress and the number of cycles to rupture in air and in vacuum.
Figure 3 Microcrack in PSB air, $2 \times 10^6$ cycles.

Figure 4 Grain boundary microcracks-vacuum, $4.3 \times 10^6$ cycles.

Figure 5 Stage II fatigue-crack air, $2 \times 10^6$ cycles.

Figure 6 Coalescence of G.B. microcracks-vacuum, $9 \times 10^6$ cycles.