CANADA

MECHANICAL AND STRUCTURAL CHANGES DURING THE DEFORMATION OF COPPER BY FATIGUE

DEPARTMENT OF MINES AND TECHNICAL SURVEYS, OTTAWA

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PHYSICAL METALLURGY DIVISION

MINES BRANCH

RESEARCH REPORT

R 68

Price 25 cents.

JULY, 1960

Mines Branch Research Report R 68

MECHANICAL AND STRUCTURAL CHANGES DURING THE DEFORMATION OF COPPER BY FATIGUE

by

J. T. McGrath* and R. C. A. Thurston**

ABSTRACT

The structural changes which take place in polycrystalline copper under the action of alternating cycles of torsion stress were studied, using the optical microscope and the X-ray diffraction camera, and an attempt was made to correlate them with changes in the shape of the cyclic stress-strain curve. It was found that the period of work-hardening was less than one per cent of the fatigue life, and that persistent surface markings, which are assumed to be incipient or actual microcracks, occur upon completion of work-hardening. X-ray diffraction patterns revealed considerable grain distortion at the completion of work-hardening in specimens cycled at a high strain amplitude. This distortion became progressively less as the strain amplitude decreased.

Mechanisms for the completion of work-hardening and the formation of microcracks are discussed.

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Direction des mines

Rapport de recherches R 68

CHANGEMENTS MÉCANIQUES ET STRUCTURAUX LORS DE LA DÉFORMATION DU CUIVRE SOUS L'ACTION DE LA FATIGUE

par

J. T. McGrath* et R. C. A. Thurston**

RÉSUMÉ

Les auteurs ont étudié les changements structuraux qui se produisent au sein du cuivre polycristallin sous l'action de contraintes alternées de torsion, utilisant à cette fin le microscope optique et l'appareil de radiocristallographie par diffraction. Ils ont également tenté d'établir une relation entre ces transformations et les changements qui se produisent dans la forme de la courbe cyclique effort-déformation. Il a été établi que la période d'écrouissage était inférieure à un pour cent de la durée de résistance à la fatigue et que, cette période d'écrouissage terminée, il se produit des rayures superficielles persistantes qui sont, semble-t-il, des fissures microscopique naissantes ou définitives. Les diagrammes de diffraction des rayons X révèlent une distortion poussée des grains à la fin de l'écrouissage dans le cas d'échantillons soumis à de violents efforts cycliques. Cette distortion décroît progressivement à mesure que diminue l'amplitude des efforts.

La présente étude traite des phénomènes qui se produisent pendant l'écrouissage et du processus de formation des fissures microscopiques.

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INTRODUCTION

Investigations[1, 2, 3, 4] with both polycrystalline and single crystal specimens under cyclic loading have revealed that the work-hardening period of the stress-strain curve is very short. In general, work-hardening ends within the first few thousand cycles of stress, and a form of work-softening or non-hardening slip continues.

In studying surface changes during fatigue, Wadsworth and Thompson,[5] by electropolishing away thin surface layers, have been able to detect a point at which slip bands become "persistent". Persistent bands are thought to be incipient or actual microcracks which eventually cross the grain boundaries and join up to form a major crack.

X-ray diffraction patterns have also been used in fatigue studies. It has been found[6] that X-ray patterns taken of annealed copper specimens subjected to high alternating stresses develop a continuous, diffuse diffraction ring, while patterns from similar specimens fatigued at low stresses show little change from the annealed material, i.e., the reflection spots remain relatively sharp. This indicates that grain fragmentation is much more severe at high stresses.

The present work was undertaken as an attempt to correlate changes in the shape of the stress-strain curve with structural changes in the specimen surface, determined by the optical microscope and by X-ray diffraction patterns, and thus to throw further light on the mechanism of the fatigue process.

EXPERIMENTAL PROCEDURE

Samples of polycrystalline, high purity, OFHC copper were machined into test pieces, as shown in Figure 1, suitable for testing in a Chevenard micro-torsion fatigue machine. Prior to cyclic stressing, the specimens were annealed in vacuo at 700°C for 2 hours, producing a grain size of approximately 45 microns. Subsequent electro-polishing by the probe method[7] with orthophosphoric acid ensured a smooth surface suitable for microscopic examination.

The Chevenard fatigue machine applies alternating torsional stress to a sample at a particular strain amplitude and at a test frequency of 1500 cycles per minute. The machine is equipped to make photographic records of couple-twist diagrams at a low frequency of about 1 cycle per minute. These diagrams or mechanical hysteresis loops are subsequently analyzed, and yield values of surface shear strain and surface shear stress acting on the specimen. A typical hysteresis loop showing the quantities measured can be seen in Figure 2. In practice, the amplitude of total strain, ϵ_t, was fixed at a particular value and the stress amplitude, σ_o, required to maintain this strain amplitude was measured after various numbers

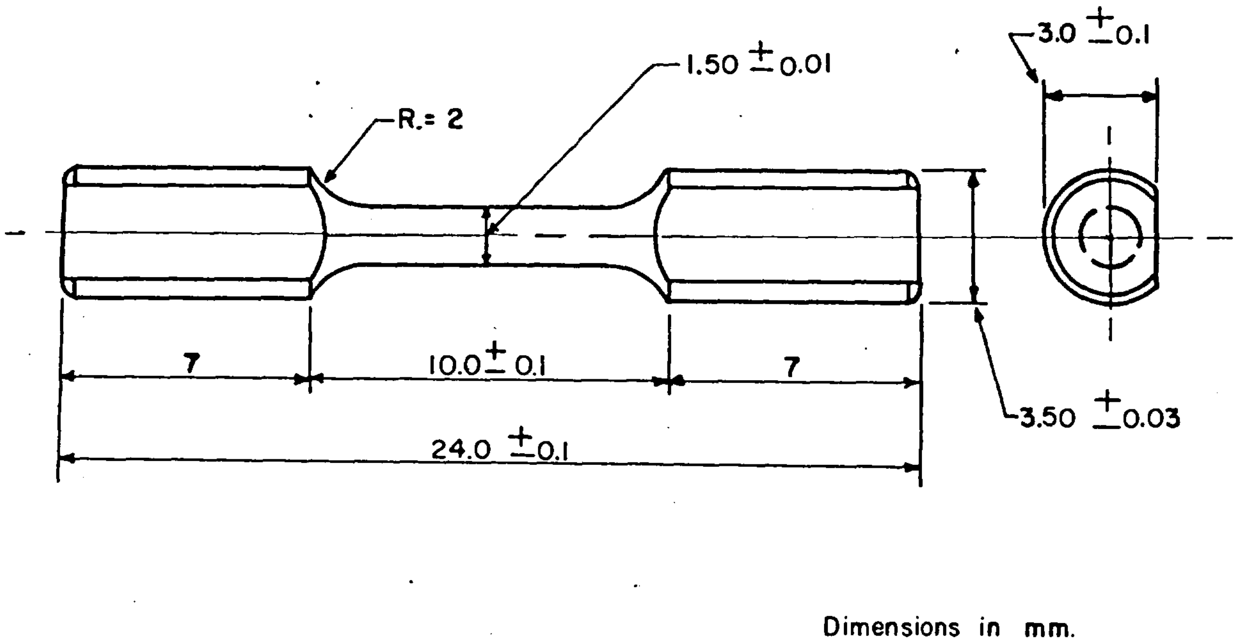


Figure 1 - Micro-torsion test specimen.

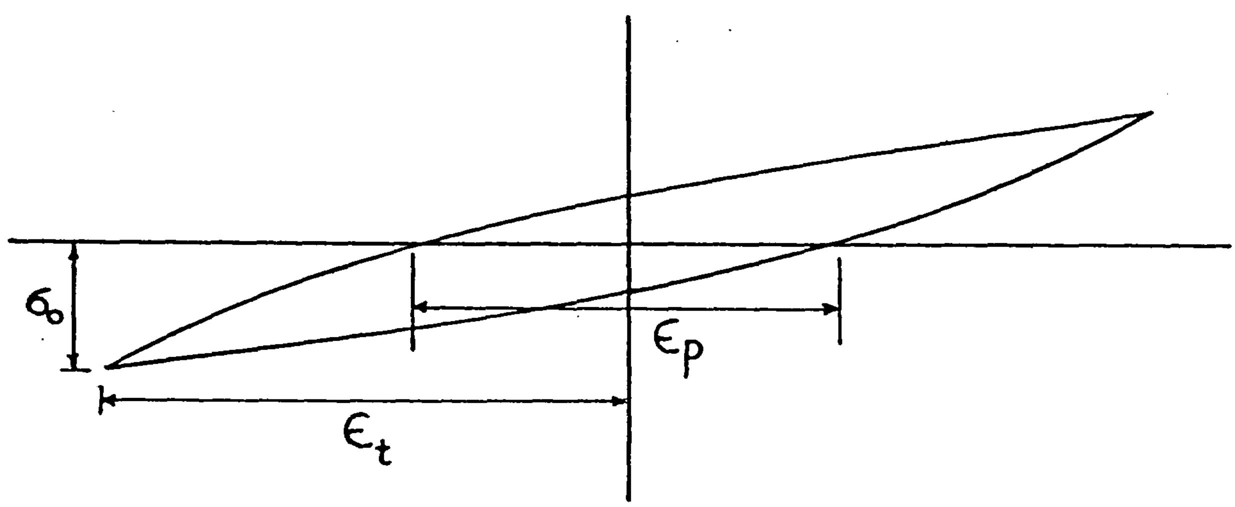


Figure 2 - Stress-strain hysteresis loop.

of cycles. Although the amplitude of total strain remains constant during a test, the elastic and plastic components of the strain will vary. The plastic strain increment, ϵ_p, for one half-cycle is simply the width of the loop measured along the strain axis. Cumulative plastic strain was obtained by summing the products of loop width and the incremental number of cycles. The total strain amplitude ranged from 0.0013 to 0.011 radian. As mentioned earlier, there were two frequencies at which the machine could operate. The low frequency, approximately 1 cycle per minute, was used in recording hysteresis loops and for the initial stages of cyclic stressing. Beyond 200 cycles the higher frequency of 1500 cycles per minute was employed.

A stress-strain curve was obtained in each case by plotting stress amplitude against cumulative plastic strain.

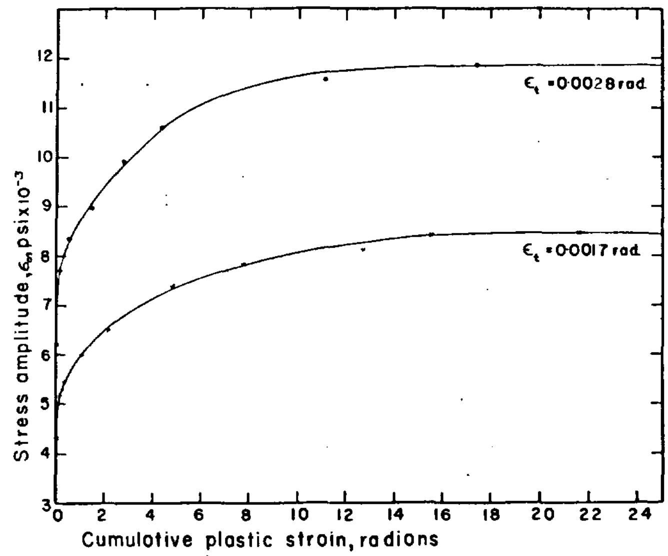
Photomicrographs and Laue X-ray diffraction patterns were taken of the specimen surface periodically, in conjunction with the stress-strain measurements. The optical microscope was used to follow the development of slip lines into persistent slip bands. In establishing the number of cycles at which bands begin to appear, a specimen was probe-polished at intervals throughout the test, and photomicrographs were taken of the surface. By polishing away approximately 15 microns of the surface at each interval, most of the fine slip was removed and only the persistent slip bands, if any, remained.

The X-ray diffraction patterns were taken using chromium radiation with a vanadium filter. The elongation of the reflection spots into a diffuse ring gave an indication of grain distortion in the surface layers.

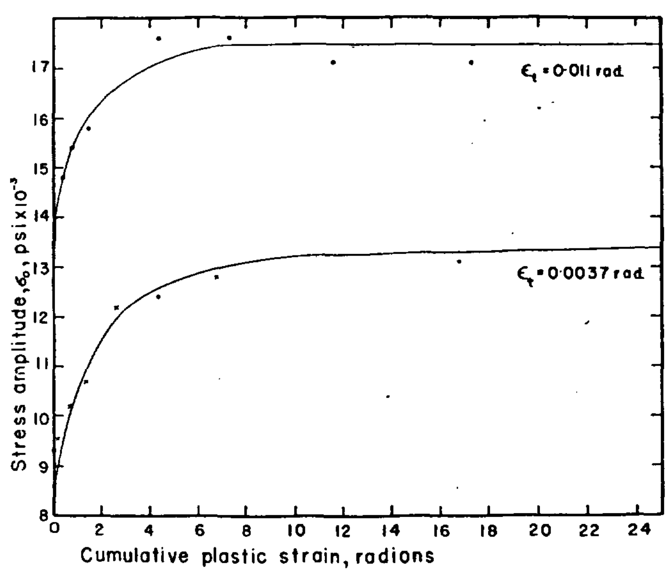
RESULTS

1. Cyclic Stress-Strain Curves

Typical stress vs cumulative plastic strain curves for polycrystalline copper under cyclic torsion are shown in Figure 3. The curves all indicate that work-hardening reaches saturation early in the fatigue process, that is, after less than one per cent of the fatigue life. Following work-hardening, the stress-strain curves tend to remain flat until just before fracture, when the stress amplitude decreases rapidly. The shape of the curves differs, however, depending on the amplitude of total strain. The slope of the work-hardening portion of the curve is much steeper at a higher strain amplitude. This finding agrees with the observations of Wood and Segall(1) and Dugdale.(4) At the lower strain amplitudes, the work-hardening region is progressively longer and requires more plastic strain to reach the maximum stress amplitude. The durations of the work-hardening period for each strain amplitude are recorded in the table of results (Table 1) on page 14, together with the cumulative plastic strain values.



(a)



(b)

Figure 3 - Cyclic stress-strain relationships for annealed copper.

The variations in the width (ϵ_p) of the hysteresis loop with the number of cycles followed a similar pattern. As cycling continued, the loop width decreased until it reached a stable value at the completion of work-hardening; no further change occurred until just before fracture. The majority of the cycling, therefore, was carried out under constant plastic strain amplitude. In view of the work of Coffin[8] and Manson[9] on the low-cycle fatigue behaviour of ductile metals, and the suggestion that their findings might be extrapolated to longer endurances ($>10^5$), the data obtained in the present study were substituted in their equation, $N^k.\epsilon p$ = constant, where N is the number of cycles to failure and k is a material constant. Published data to date have indicated that for most of the metals examined the index k is approximately 0.5.

When cyclic plastic strains were plotted against life, on a log-log scale, the values for the three shorter tests fell very close to a straight line, whereas the fourth point was well removed and appeared to correspond to a premature failure. Using the method of least squares, the best-fitting straight line was determined for the shorter test results, and gave a value for k of 0.5, which is in close agreement with existing data. This finding indicates that the Coffin-Manson equation may be applicable up to 10^6 cycles for copper, though further work is obviously necessary to substantiate the suggestion and to determine the significance of the reduced life at the lowest strain amplitude.

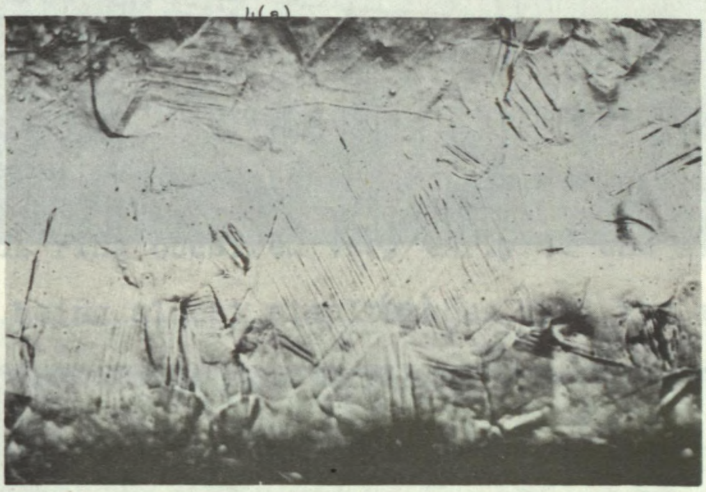
2. Persistent Markings

Observations of slip markings revealed that the work-hardening portion of the stress-strain curve was associated with the appearance of fine slip in the form of well-defined clusters within the grains. Coarse slip bands then develop from the fine slip. As summarized in Table 1, persistent markings were found to occur almost simultaneously with the end of work-hardening at the higher strain amplitudes. At lower strain amplitudes, persistent markings occurred shortly after the completion of work-hardening.

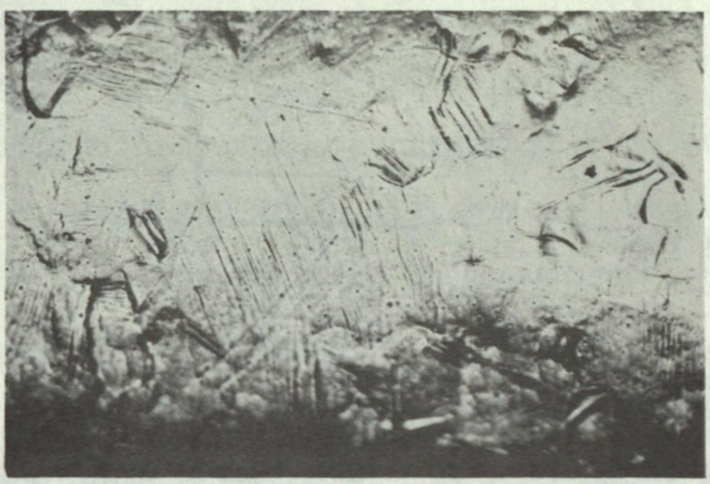
Persistent grain boundary markings were observed at the higher strain amplitudes. Figure 4, taken after electropolishing, shows the development of both slip band and grain boundary persistent markings in a specimen fatigued at a high strain amplitude; general rumpling of the surface is also visible. At low strain amplitudes the persistent markings were almost entirely in slip bands as shown in Figure 5. These observations support the findings of the authors and others[10] that crack paths in copper tend to follow grain boundaries at high stresses and slip bands at low stresses.

One specimen was tested at a strain amplitude of 0.0013 radian, which was insufficient to produce failure after ten million cycles. After removing 15 microns of

4(a)



4(b)



4(c)

Figure 4 - Copper at different stages of a fatigue test performed at a total strain amplitude of 0.011 radian. (Mag. X400). Reduced 50 per cent for reproduction.

(a) After 300 cycles and removal of 15 microns of the surface by electro-polishing.

(b) After 600 cycles and removal of 15 microns.

(c) After 1000 cycles and removal of 15 microns.

(a)

(b)

Figure 5 - Copper fatigued at a total strain amplitude of 0.0017 radian. (Mag. X400)

(a) After 2,836,400 cycles.

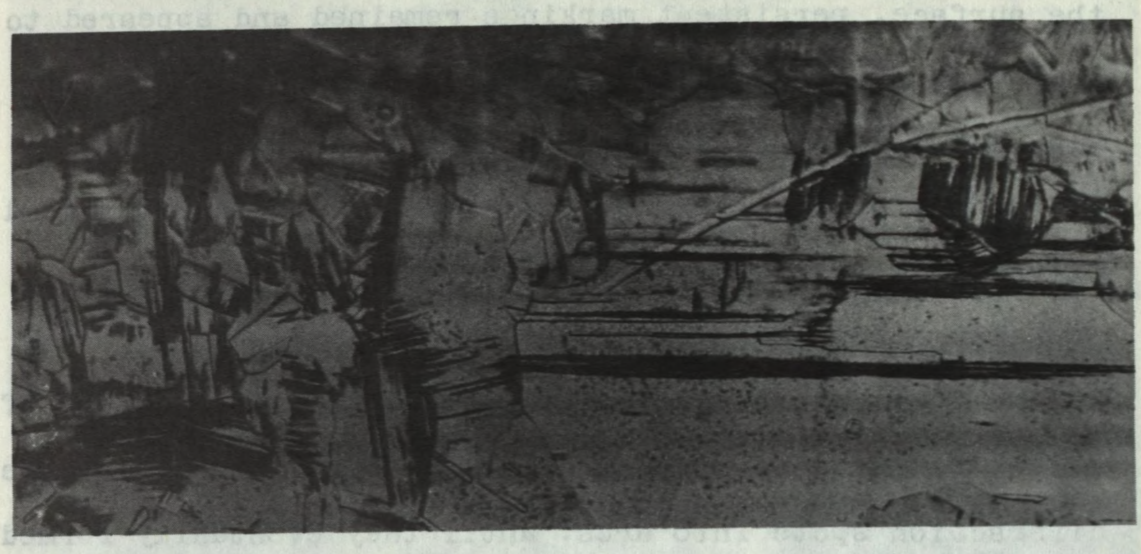
(b) Same as (a) after removal of 15 microns.

the surface, persistent markings remained and appeared to be composed of short irregular segments as shown in Figure 6. This observation is similar to the finding of pitted surface markings by Smith[11] in aluminum fatigued at very low stresses.

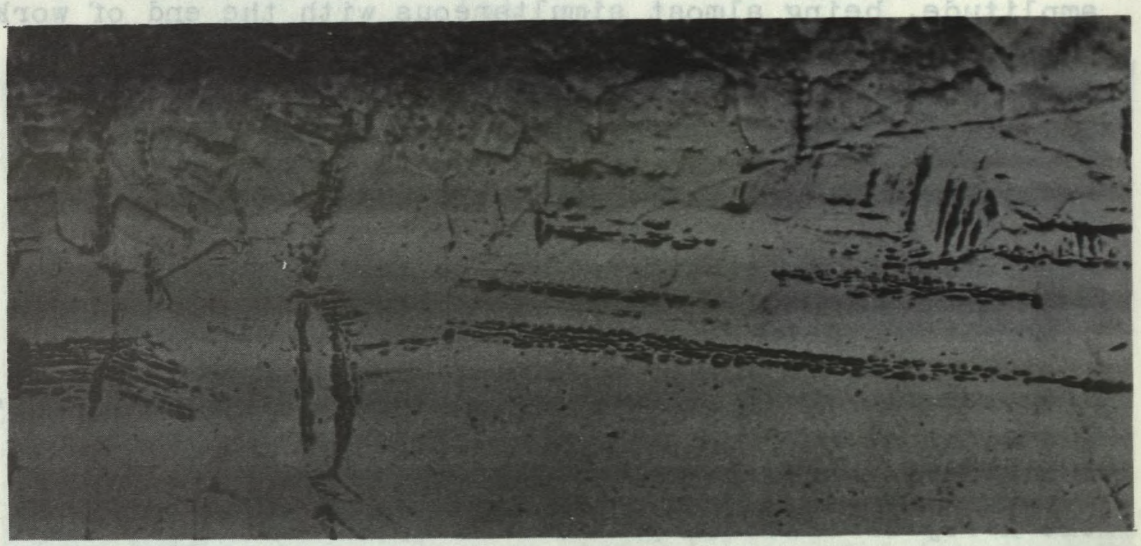
3. X-Ray Diffraction Patterns

The distortion of grains with increasing number of cycles was studied by observing the spreading of discrete diffraction spots into arcs, until they eventually formed a continuous Debye-Scherrer ring. As summarized in Table 1, a continuous ring occurred very early at the highest strain amplitude, being almost simultaneous with the end of work-hardening. The formation of a complete diffraction ring was progressively delayed as the strain amplitude was decreased. In general it was found that following the formation of the ring there was no further change in the diffraction pattern. At low strain amplitudes the diffraction ring never becomes continuous, even at fracture, indicating only slight grain distortion. Figure 7 shows the X-ray diffraction patterns of specimens fractured at a high and a low strain amplitude.

The X-ray diffraction results seem to be in agreement with the observations of persistent markings. The large number of persistent grain boundary markings at high strain amplitudes suggests that heavy deformation within grains has resulted in contact being disrupted along



(a)

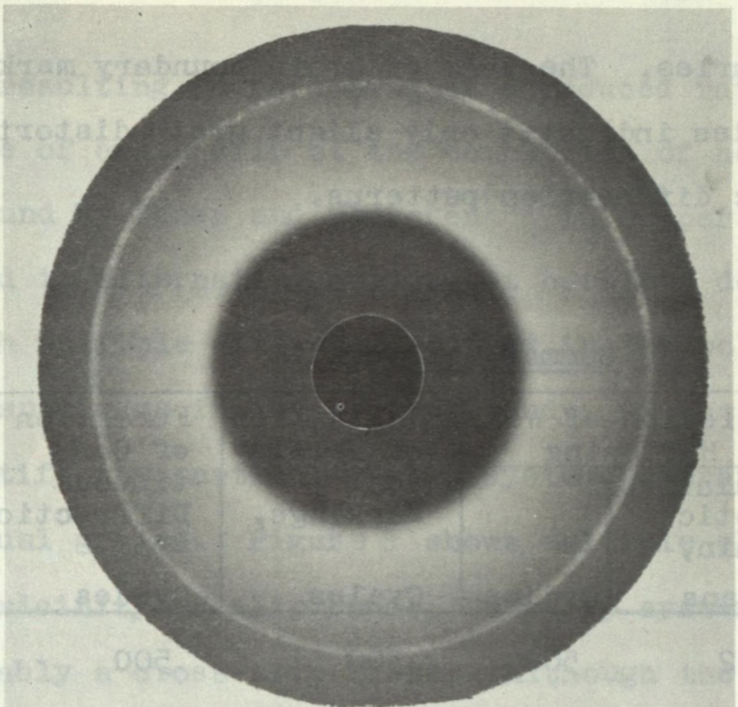


(b)

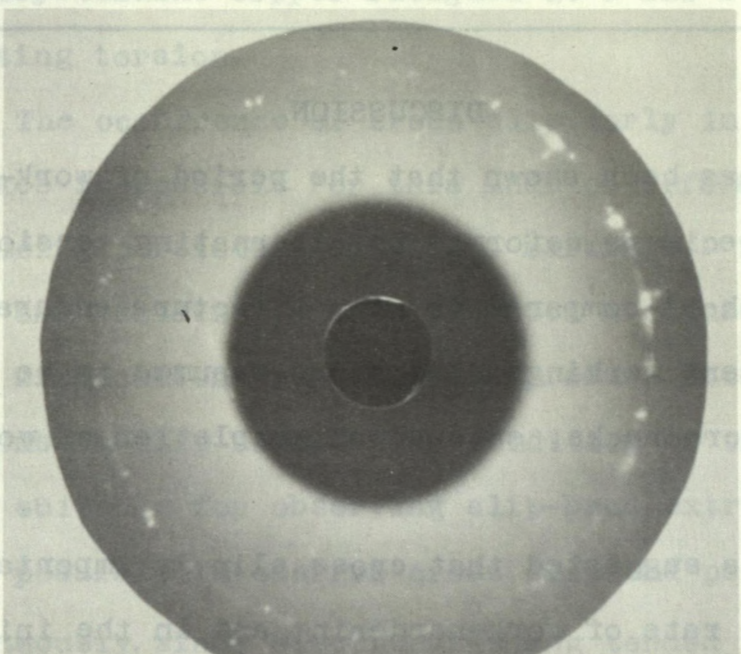
Figure 6 - Copper fatigued at a total strain amplitude of 0.0013 radian. (Mag. X400)

(a) After 10,180,000 cycles.

(b) Same as (a) after removal of 15 microns.



(a)



(b)

Figure 7 - X-ray back-reflection patterns of copper.

(a) Fatigued to fracture at a total strain amplitude of 0.011 radian.

(b) Fatigued to fracture at a total strain amplitude of 0.0017 radian.

grain boundaries. The lack of grain boundary markings at low amplitudes indicates only slight grain distortion, as shown by the diffraction patterns.

TABLE 1

Summary of Results

Total Strain Amplitude (ϵ_t), Radians	Completion of Work Hardening: Cumulative Plastic Strain, Radians	Completion of Work Hardening: Cycles	Initiation of Persistent Markings, Cycles	Formation of Continuous Diffraction Ring, Cycles	Fatigue Life, Cycles
0.011	7.2	500	300	500	42,000
0.0037	9.9	1600	2000	5,000-10,000	410,000
0.0028	14.3	4000	>3000	50,000	900,000
0.0017	17.2	18500	25000	(Not formed)	3,853,300

DISCUSSION

It has been shown that the period of work-hardening in copper specimens deformed in alternating torsion is relatively short compared to the fracture endurance, and that persistent markings, which are assumed to be incipient or actual microcracks, occur upon completion of work-hardening.

It is suggested that cross slip is important in reducing the rate of work-hardening and in the initiation of microcracks. Seeger(12) has proposed that screw dislocations, which have piled up at Lomer-Cottrell barriers during hardening, can escape by moving in the cross-slip plane, resulting in hardening at a reduced rate. Direct evidence of cross slip at the completion of hardening has been found by Ebner and Backofen(3) in copper single crystals deformed in alternating bending at constant deflection. Although multiple slip was observed in the polycrystalline copper specimens in the present study, it was impossible to identify the particular slip systems operating in individual grains. Figure 8 shows multiple slip occurring in the vicinity of slip bands, one slip system of which is probably a cross-slip plane. Although the photomicrograph was taken of a copper specimen stressed in alternating bending, the slip pattern is typical of that observed in polycrystalline copper fatigued at a low stress in alternating torsion.

The occurrence of cross slip early in the fatigue life makes it possible for this mode of deformation to aid in producing persistent slip-band markings. Mott(13) and McEvily and Machlin(14) have proposed mechanisms which require dislocation cross slip to produce intrusions and extrusions in slip bands. The condition of the surface was not suitable for observing slip-band extrusions. It was not possible to observe cross slip and persistent slip simultaneously, since electropolishing tended to remove cross-slip lines. The surface shown in Figure 8 has not been electropolished, and the occurrence of what is considered to be cross slip near the crack path would lend support to the idea that cross slip is important to crack initiation.

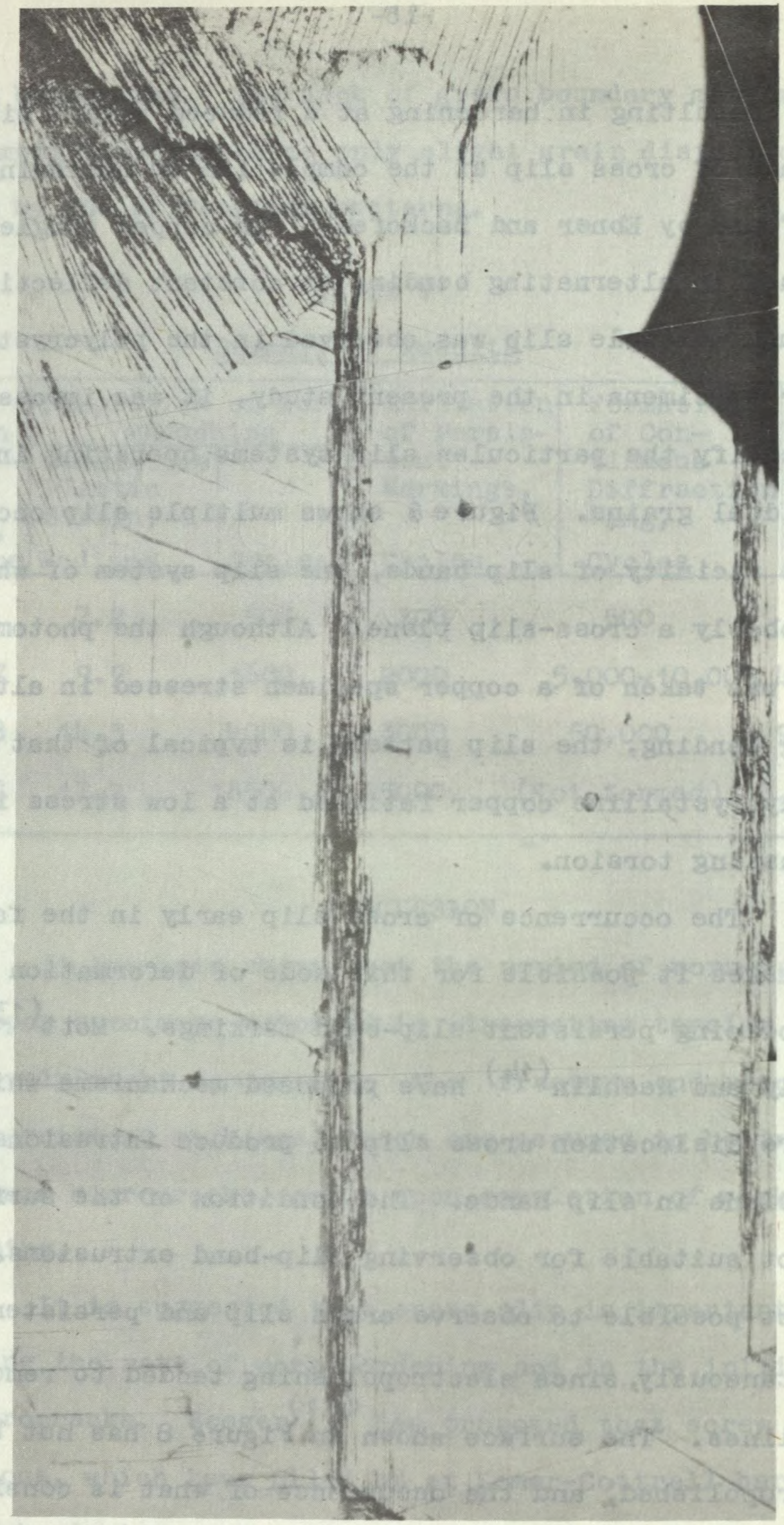


Figure 8 - Copper deformed in reversed alternating bending at $\pm 10,000$ psi for 1,881,000 cycles. (Mag. X400). Reduced 25 per cent for reproduction.

The theories for the mechanism of fatigue all postulate that cracks initiate in slip bands. However, particularly from the observations of persistent markings at high strain amplitudes, it is evident that cracks can initiate in grain boundaries in copper. It is doubtful that any of the above-mentioned theories would be applicable. Thompson[15] has suggested that cracks which occur in twin boundaries may be initiated by slip at the boundaries, but this is a special case. What happens at ordinary grain boundaries is not known. Perhaps an investigation using bicrystals with various types of grain boundaries would be useful in solving this problem.

CONCLUSIONS

From the present study of the behaviour of vacuum-annealed, polycrystalline copper under alternating torsion, the following conclusions can be drawn:

(1) Work-hardening commences immediately and is completed before one per cent of the fracture endurance is reached.

(2) The rate of work-hardening and the saturation level depend upon the applied cyclic strain amplitude, both decreasing with decreasing strain. At low cyclic strains the work-hardening period is longer and requires more cumulative plastic strain.

(3) Persistent surface markings occur at the conclusion of the work-hardening period. At high cyclic strain amplitudes, the persistent markings are found both in the grain boundaries and within the grains. At low amplitudes, they are mainly confined to the slip bands.

(4) At very low cyclic strain amplitudes (endurance $> 10^7$ cycles), the persistent markings take the form of short, irregular segments.

(5) Grain distortion, as shown by X-ray diffraction patterns, increases with the number of cycles. At high strain amplitudes, the Debye-Scherrer ring becomes continuous at or near the end of the work-hardening period. At low amplitudes, the diffraction ring never becomes continuous, indicating only slight grain distortion.

The experimental observations from the hysteresis loops, X-ray diffraction patterns and microscopic examination are consistent, and the evidence presented supports the suggestion that cross slip is a factor in reducing the work-hardening rate and in initiating microcracks.

ACKNOWLEDGMENT

The technical assistance of Mr. J. A. Ellis of the Engineering Physics Section in all phases of the experimental work was greatly appreciated.

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