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DISLOCATION ARRANGEMENTS IN ALUMINUM DEFORMED BY REPEATED TENSILE STRESSES*

by

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ABSTRACT

Transmission electron microscopy is used to study the dislocation arrangements formed in aluminum at 78^{o} and $300^{o}K$ by the action of repeated tensile stresses. Although a pronounced macroscopic cyclic stress induced creep effect is observed, the dislocation pattern formed on the first cycle at $78^{o}K$ is not grossly altered by the subsequent cycling. The dislocation density and cell size undergo a slight increase and decrease, respectively. Contrary to published results on the fatigue of aluminum under reversed stresses, the dislocation loop density in aluminum deformed by a repeated tensile stress is not significantly larger than the loop density produced by unidirectional straining.

The present information and additional results on the effect of cyclic straining on the low temperature yield phenomenon in FCC metals support a mechanism based on point defect-dislocation interaction as a satisfactory explanation for cyclic stress induced creep.

ACKNOWLEDGEMENTS

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I. INTRODUCTION

Under conditions of cyclic loading, large accumulations of plastic strain have often been observed when a mean or directing stress is present (1-3). In a recent investigation of this cyclic stress induced creep effect (4), it was found that the application of repeated tensile stress of sufficient magnitude would result in ductile failure in aluminum and copper in a few thousand cycles whereas the application of a steady creep stress of the same magnitude produced little deformation and no failure over a period of several days. An example of this effect at 78°K for high purity aluminum used in the present investigation is shown in Fig. 1. Curve B in Fig. 1a is simply the usual static creep curve obtained by applying a steady stress of 12,000 psi and measuring the total deformation over a period of several hours. Curve A represents the accumulation of strain as a function of cycles and time* for the same stress applied cyclically in the manner indicated in Fig. 1b. In the latter case, large accumulations of strain are observed and a ductile or "necked-out" cyclic failure occurred in 3440 cycles while in the static creep test the strain increased (only) 0.013 in the same period of time and did not fail after 40 hours at load.

This marked difference in behavior under cyclic loading suggested that there might be a conspicuous difference between the dislocation arrangements of samples strained statically and cyclically as in Fig. 1. This being so, a study was undertaken to examine the dislocation distributions developed in high purity aluminum under these conditions. A temperature of 78°K was chosen because the macroscopic effect as shown in Fig. 1a is pronounced at this temperature and no results have been reported (to the author's knowledge) on the dislocation arrangements in metals subjected to cyclic loads at depressed temperatures. Additional experiments have been completed at room temperature.

II. EXPERIMENTAL PROCEDURE

Annealed polycrystalline aluminum sheet specimens 0.005 inches thick of nominal purity 99.999% were statically and cyclically loaded at 78°K, to obtain macroscopic strain-time and strain-cycle curves similar to those in Fig. 1a. After straining, the center sections of the specimens were electrolytically thinned in a perchloric acid bath (maintained at 0 to 5° C) by means of the "window"

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* Time in this case means time of test obtained from the equation $t = N/f$, where f is the frequency of loading.

method. (5) Thin foil fragments were examined in a Siemen Elmiskop I operating at 100 kv.

At initial strains as large as those shown in Fig. 1a, dislocations introduced during handling of the thinned foils were easily detected. These dislocations were long, generally straight and invariably associated with bend extinction contours. The reliability of the observations was improved by using foils greater than 2500 Å thick in accordance with the suggestions of Wilsdorf and Schmitz (6).

Dislocations densities were measured by employing the method outlined by Ham (7) in which the number of intersections N which dislocations make with a set of random grid lines of total length L, determines the mean length of dislocation line per unit volume as $R = 2N/Lt$, where t is the local foil thickness. This method is much faster than the technique of actually measuring the mean projected length of dislocation line (8). Nevertheless, both of these methods depend on the assumption that the dislocation segments are randomly oriented with respect to the plane of the foil and thus do not account for the fact that the distribution of dislocation orientations may be biased in favor of the direction normal to the foil by the shortening and realignment of dislocation segments during thinning. In thick aluminum foils (3000-5000 Å) having an oxide coating, this biasing of the orientation distribution is not too serious as density measurements in the present work made by the surface intersection counting technique (9) indicate a systematic, 12 percent higher variation with the other methods.

Additional factors which result in systematic errors such as the dislocation loss during thinning (10) and the problem of correlation between dislocation length and density (11) have not been considered in the present work.

Since the electropolishing of aluminum foils cannot be done at low temperatures, specimens strained at 78° K necessarily undergo some annealing just below room temperature and prior to examination in the electron microscope. Although the work-hardening introduced at low temperatures is thought to be unstable at room temperature, Cottrell and Stokes (12) have shown that the rate at which it disappears from thermal effects alone is rather slow.

In order to obtain some quantitative results, the dislocation arrangements were examined in a specimen deformed at low temperature which had undergone a minimum of annealing treatment (approximately 15 minutes at 0° C and 5 minutes at 20° C before the first picture). Comparison of micrographs from identical regions after different amounts of elapsed time at 20° C, showed no significant change over a period of four days.

Undoubtedly some relaxation and rearrangement of dislocations occurs when the temperature is raised from that of liquid nitrogen to 20° C but this effect cannot be determined from the above procedure. However, the X-ray micro-beam results of Kelly and Roberts (13) indicate that the basic size of the dislocation cell structure produced in aluminum during low temperature deformation is retained upon warming to room temperature but that migration of matrix dislocations to the cell walls during warming creates larger local differences in the dislocation density when examination is carried out at room temperature. Moreover, it is well known that prismatic dislocations loops in aluminum are retained up to temperatures as high as 170° C (14).

On the basis of the above observations, it is concluded that the dislocation patterns in an aluminum sample deformed at a low temperature and subsequently examined at room temperature are representative of the patterns formed at the lower temperature with the exception that the difference between the cell wall and matrix dislocation density is accentuated by recovery.

III. RESULTS

After the examination of a large number of micrographs taken from approximately 20 different specimens, the most important result to be reported is that there is no striking difference between the dislocation patterns in aluminum strained statically and cyclically under the conditions given in Fig. 1a.

Micrographs which are representative of the average dislocation patterns developed at different places along the cyclic creep curve are shown in Figs. 2-4. After one cycle (the equivalent of a 10% tensile strain) the dislocations are arranged in an irregular cell structure (Fig. 2a). The average dislocation density is 6.1×10^{9} lines-cm/cm^{3} and the cell size is approximately 1.75 microns. By comparison, Swann (15) has reported an average cell size of one micron for aluminum deformed ten percent by rolling at a temperature of 78° K. On the basis of the warming experiments of Kelly and Roberts (13) a more realistic picture of the over-all crystal structure (than that shown in Fig. 2a) would exhibit less clearly defined cell walls. This means that the dislocation density in the subgrains is probably higher than that shown. Accordingly the cell structure at this point can be considered to be poorly developed*.

*There is often a great variability in the dislocation pattern even within the same crystal. Even though a cellular structure is formed such as in Fig. 2a, the appearance of the dislocation arrangement may be quite different in a neighboring region as shown in Fig. 2b. In this micrograph several cell walls apparently lie nearly parallel to the plane of the foil and it is impossible to measure cell sizes. Accordingly, all cell size measurements have been made only from micrographs in which the cell structure is clearly present. Each cell size reported in this study is the average of a total of approximately sixty measurements derived from four widely separated regions from each of three different specimens subjected to the same test conditions.

After 500 cycles the structure has undergone little change (Fig. 3). The dislocation density is now 6.9 x 10^9 lines-cm/cm^3 and the cell size has decreased slightly to 1.7 microns. Further changes in dislocation density and cell size take place very slowly until ductile failure occurs. At this point the dislocation density is 7.3 x 10^9 lines-cm/cm^3 and the cell size is 1.7 microns (Fig. 4). These results substantiate the conclusion that the over-all dislocation pattern changes little during the development of a cyclic creep failure. The dislocation cell walls do become slightly 'tighter' as measurements of the number of dislocations in a cell wall and the mean free length of dislocation line are found to increase and decrease, respectively.

A simple calculation based on the formula $\epsilon = \rho b \ell$ where ϵ is the total strain, ρ is the dislocation density, b is the Burgers vector and ℓ is the average distance traveled by a dislocation, suggests that the large amount of plastic strain accumulated during a cyclic creep experiment may occur by the continued motion of the dislocations produced on the initial loading and not by the multiplication of large numbers of new dislocations as the observed dislocation density increases only about 20 percent. On this basis, the total distance traveled by a dislocation is expected to be approximately 15 microns and therefore any one dislocation will visit, on the average, nine different cells during its lifetime. There is also the possibility that large numbers of dislocations are mutually created and annihilated; a process that would also result in only a small change in the dislocation density.

In view of the fact that aluminum at 78° K exhibits logarithmic creep, little change is expected in the dislocation patterns over periods of time comparable to those given in Fig. 1. Examination of several static creep specimens at various positions along the curve of Fig. 1a and measurements of dislocation density, etc. from the corresponding micrographs substantiate this conclusion. No change could be determined within the error of measurement.

In a cyclic creep failure the total strain at failure is nearly the same as that produced by unidirectional straining. Fig. 5 shows the dislocation structure in a static tension sample which has been pulled to failure (approx. 30% strain). The dislocation density is approximately 2 x 10^{10} lines-cm/cm^3 which is considerably higher than that produced in a cyclic creep failure. In addition the average cell size has decreased from 1.75 microns at a strain of 10% and is now 1 micron at failure.

In contrast to the poorly developed and ragged cell structure formed at 78°K, a well defined substructure is produced by repeated tensile loading at room temperature. Figure 6 is an example of the dislocation distribution in an aluminum sample which has been strained 10% on the first cycle and subjected to a constant repeated tensile stress thereafter until failure at 14,800 cycles. The interiors of the cells are relatively free of dislocations but are bounded by dislocation walls of a high density. Nevertheless these cell walls have not reached the more advanced stage of polygonization which has been observed in aluminum subjected to reversed strain cycling at room temperature (16, 17).

At lower stresses and correspondingly longer lives a similar well defined cell structure is observed (Fig. 7). Although many of the subboundaries are arranged irregularly there is some tendency for them to lie parallel to low index planes. Subboundaries which lie nearly parallel to the foil surface often appear in the form of twist boundaries such as at A in Fig. 8. At first glance the boundary at A appears to have a zigzag configuration similar to that reported by Thomas (18) but this is due to the fact that one set of screw dislocations making up the twist boundary is nearly out of contrast and appear as 'ghost' dislocations.

Although systematic measurements have not been carried out at room temperature, it appears that the cell size formed on the first cycle does not change appreciably with the repeated application of a tensile stress. The approximate cell sizes of 5 and 10 microns in Figs. 6 and 7, respectively, are in good agreement with the results of Kelly (19) on aluminum for comparable amounts of tensile deformation.

The presence of large numbers of dislocation loops in fatigued FCC metals has been reported by several investigators (16, 17, 20). In all cases these observations have been made on samples subjected to <u>reversed</u> stresses. On the contrary, the dislocation loop density in aluminum deformed by repeated tensile stresses at 78 and 300°K is not significantly larger than the loop density produced by unidirectional straining (Figs. 8 and 9). It appears that the stress must be reversed in order to produce a high dislocation loop density. One possible explanation is that reversing the stress results in a greater number of jog producing intersections. The accumulation of the intersection jogs in the manner described by Washburn (21) would eventually produce such a deep jog that the formation of dislocation dipoles would be favored over nonconservative motion and point defect production.

IV. DISCUSSION

In the following discussion, remarks are centered around the cyclic creep effect as it occurs at temperatures of less than one fourth of the melting temperature for it is in this region that the cycle-dependent effects are dominant and most time dependent effects are virtually neglible.

The observation that the dislocation structure in aluminum does not change appreciably during cyclic creep at $78^{o}K$ is particularly significant. However, in order to achieve the large observed creep rates under cyclic loads some change must be taking place. This suggests that the change is 'dynamic' and thus periodic examination of the dislocation pattern when viewed in the 'static' condition shows no gross difference. This behavior is somewhat analogous to the motion of gas molecules in a container. If at any-one time the motion of all molecules is stopped and a picture taken of the molecular distribution, this picture would not be significantly different from a picture taken at some later time. Although the distribution appears to be approximately the same at different times, individual gas molecules will have changed position many times between the observation. It is suggested that a similar sequence of events takes place in the course of a cyclic creep experiment. Thus, it is possible to cause a strain increment by two means without a correspondingly large increase in dislocation density; the production of many new dislocations which move short distance and are then annihilated or the motion over relatively long distances of the already existing dislocations. The delineation between these two processes is not necessary for the cyclic creep model to be discussed

It is apparent by the examination of results in which the low temperature static and cyclic creep curves are compared for the same maximum stress (such as in Fig. 1) that dislocation models which depend on the circumvention of barriers with the aid of thermal fluctuations are not adequate. This generalization is justified on the basis that dislocations acted upon by a steady stress (as opposed to those acted upon by a cyclic stress) spend a much greater amount of time in a position of readiness to accept any local thermal fluctuations which might assist them over their barriers; thus greater creep rates would be expected under a steady stress. Inasmuch as the exact opposite behavior is observed at low temperatures ($T/T_m < 0.25$), it concluded that this class of dislocation model is incorrect. It is therefore necessary to envisage a low temperature mechanism whereby the dislocations are deflected out of their glide planes on the unloading portion of a cycle such that on reloading, the obstacle is surmounted.

Under the assumption that low temperature cyclic stress induced creep is caused by the operation of one basic mechanism it is of interest to examine the possible role of cross-slip. Since cross-slip is considered to be thermally activated (22), it cannot be the controlling mechanism on the loading portion of a cycle for the reasons discussed in the previous paragraph. The possibility remains that cross-slip occurs during unloading and that upon reloading, barriers are circumvented. Unfortunately there does not appear to be any plausible mechanism by which dislocations can cross-slip out of their natural glide planes by the reduction of the applied stress at low temperatures.

Having considered cross-slip, we then turn to the remaining possibility by which dislocations can move out of their glide planes, that is by the interaction with point defects. Evidence for the production and migration of large numbers of point defects during low temperature plastic deformation is formidable. Adequate summaries of this convincing evidence are available (23, 24) and only information pertinent to the present problem will be considered.

There are a variety of ways in which point defects might be produced during cyclic creep straining. One of the more prolific sources is the nonconservative motion of elementary jogs in dislocations with a strong screw component. The jog may be dragged along by the action of the dislocation cusp or it may remain stationary and pull out a long dislocation dipole which is pinched off. The operation of the latter mechanism would result in the formation of large numbers of elongated dislocation loops, a condition which has not been observed in the present work on aluminum. On the other hand, dragging of the jog by the dislocation cusp will result in the periodic emission of strings of point defects, which would not be observed if they are annihilated at nearby sinks.

A variety of defects can be formed including vacancies, interstitials, divacancies and various vacancy and interstitial clusters or aggregates. In view of the number of annealing stages present in cold worked metals, especially copper (23), it seems likely that all of the above defects or defect clusters are present to some extent. Recently, Hirsch (25) has suggested that jogs in FCC metals are extended at equilibrium. After an extensive examination of the details of jog geometry under an applied stress, he has concluded that interstitial-producing jogs are generally constricted by the stress and thus become glissile while vacancy-producing jogs are extended by the stress and are sessile. Therefore, the tendency would always be to create a net concentration of vacancies or vacancy clusters and there would be little vacancy-interstitial annihilation by the reverse motion of a vacancy (or interstitial jog under an oscillating stress. In further support of this, Hirsch has shown that certain jog configurations will emit vacancies in both the forward and reverse direction as a result of the peculiarities of jog constriction. However, these results must be regarded with some caution as they are based on uncertain estimates of the constriction energy and other assumptions of details of the jog geometry.

The present evidence is not sufficient to ascribe the cyclic creep effect to a particular defect. However, it has been found that the incipient low temperature yield point in FCC metals is greatly enhanced by the action of a repeated tensile stress (26). In this work the increase in flow stress or the yield drop as shown in Fig. 10 increases with increasing number of cycles. On the basis of strain-ageing experiments in aluminum (27) and copper (28-30) which have employed this technique of measurement, the cyclic enhanced yield effect is interpreted as being due at least in part to the interaction between dislocations and excess point defects. Consequently, FCC metals subjected to a repeated tensile stress at low temperatures exhibit both a hardening (enhanced yield effect) and a simultaneous sofening (cyclic creep).

Initial hardening and subsequent resoftening is a typical consequence of point defect strain-ageing. That this is so, is effectively shown by the change in yield stress of quenched FCC metals with increased ageing time. In quenched copper (31) and gold (32), the yield stress initially rises to a maximum as the dislocations are pinned by the excess point defects. After prolonged ageing the yield stress decreases as a result of the pipe diffusion and eventual destruction of point defects (at jogs) which leave the dislocations unpinned. An additional by-product of this point defect precipitation is the climb of dislocation segments at the place of defect annibilation. A similar hardening and resoftening occurs at low temperatures as a result of the eventual annihilation of deformation produced point defects (28).

During the course of a cyclic stress induced creep experiment, hardening and softening occur simultaneously and complete overageing is never observed. This effect arises as a result of cyclic stress acting as a point defect 'pump'. This idea gains considerable support from the literature (33-35). Accordingly, point defects are continually being generated, cycle by cycle, by the oscillatory motion of longer segments of dislocations attached to the complex tangles and cell walls formed at low temperatures. As these defects are emitted in clouds or strings they will either form clusters or will disperse. In light of the relatively high motion energy of monovacancies, there appears to be little chance of wholesale dispersal of this type of defect at low temperatures. Rather it appears that either more mobile small vacancy clusters such as divacancies or individual interstitials are formed and a net flux of defects are emitted into the core of nearby dislocations, thereby pinning the dislocation, or else oscillating dislocation segments or gliding dislocations pass through the freshly generated cloud of point defects and sweep up a sufficient number to become pinned. It is this pinning effect which gives rise to the yield point or increased flow stress as indicated in Fig. 10. In order to continue the deformation unidirectionally, a slightly higher stress must be applied to overcome the binding energy between the dislocations and point defects or defect clusters.

However the pinning effect is not permanent. Individual point defects attached to the dislocation core do not lose their identity even though their self energy is reduced by an amount equal to the binding energy between the defect and the dislocation. The point defects will move along the dislocation core, some occasionally nucleating new jogs (36), but the majority of them eventually being destroyed at intersection jogs. As a result of this annihilation, segments of the dislocation climb and the pinning atmosphere is temporarily lost. The dislocation will either have climbed far enough to escape its permanent obstacle or else it will continually repeat the generation-pinning-annihilation-climb process until it has traveled the necessary distance to move by gliding.

It is the detailed balance between pinned dislocations and climbing dislocations which determines the steady state cyclic creep rate. The yield point measurements are a reflection of the fraction of dislocations which are pinned at any particular number of cycles and these measurements also indicate that a steady state is reached by the leveling off of the yield drop to a constant value as shown in Fig. 10. Since most of the point defects are annihilated almost as quickly as they are generated, few collapsed vacancy (interstitial) loops will be observed. In addition, most of the strain is accumulated by either a process of mutual dislocation creation and annihilation or the migration of dislocations over large distances. Although some increase in dislocation density is expected, it will not be as large as if the deformation is continued by enforced straining such as in unidirectional tension.

The general class of dislocation model discussed appears to have all the necessary features to account for the observed low temperature cyclic creep behavior, yet many of the details are open to question. A number of quantitative calculations need to be made to attempt to assign limits to the rates at which the various processes may take place. A systematic investigation of these effects is still in progress.

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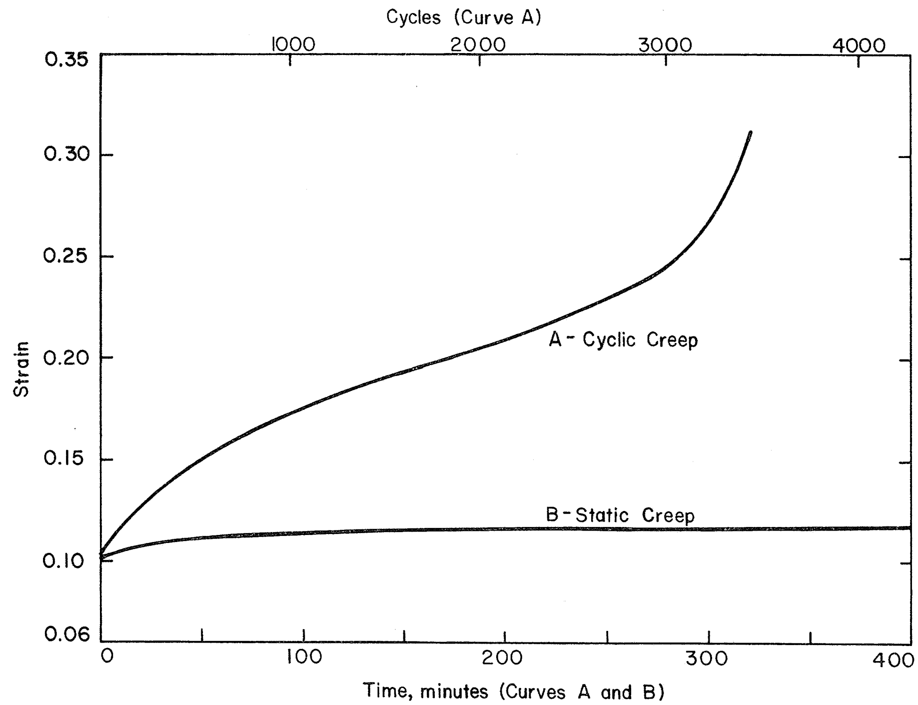
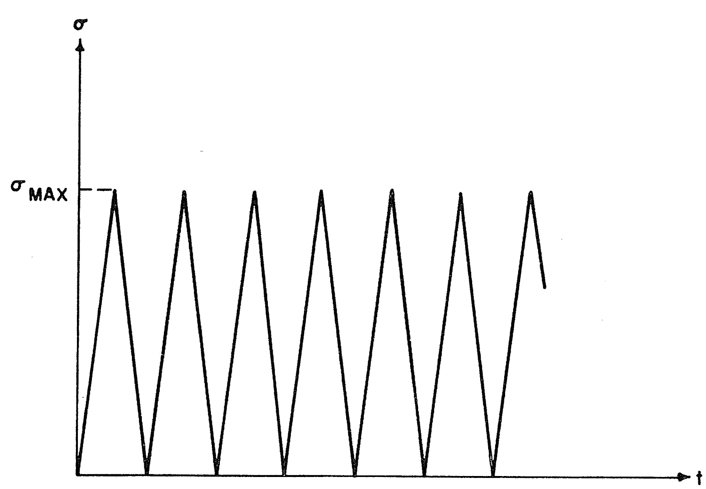
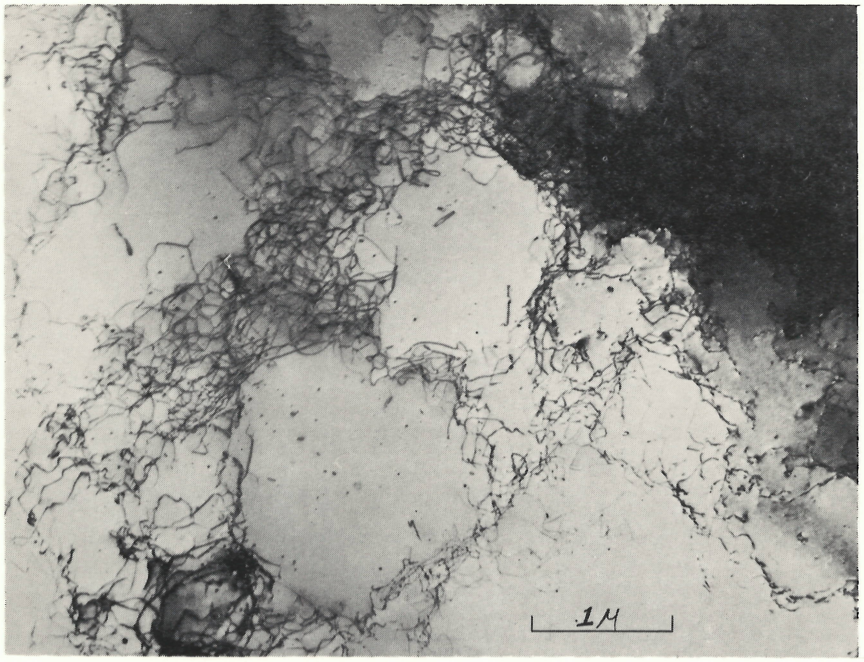


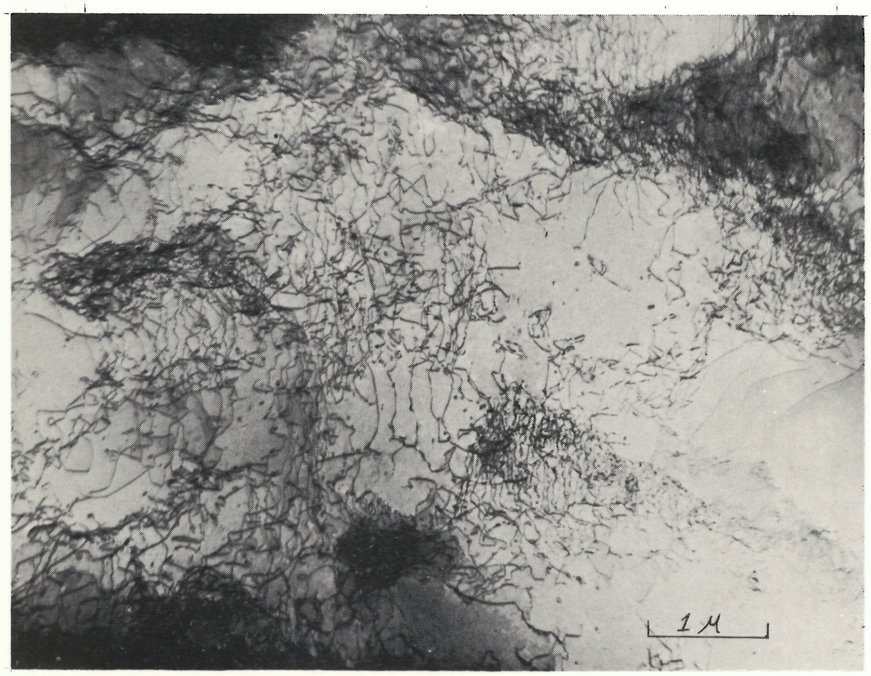
1. (a) Static and Cyclic Creep Curves for 99.999% Aluminum at 78^{O}K.



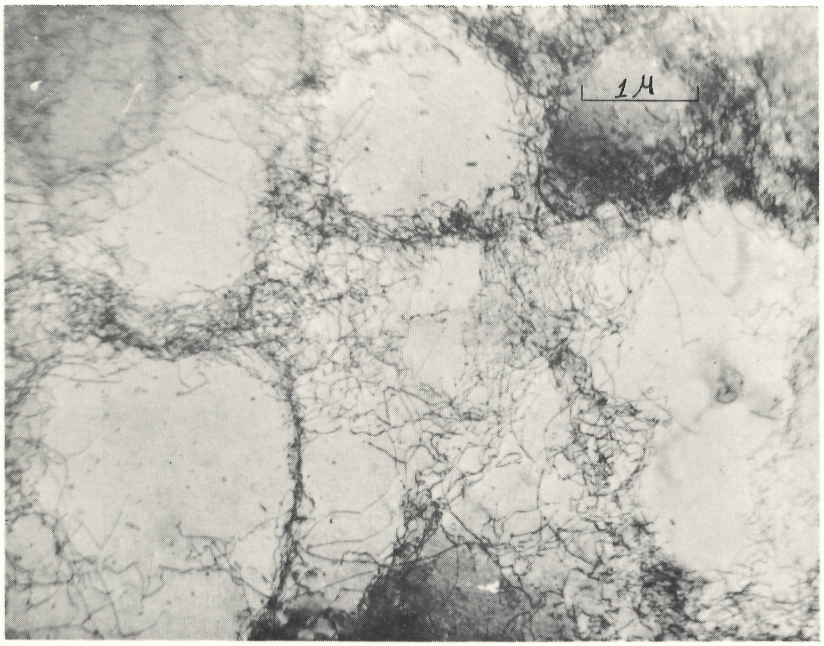
1. (b) Stress-Time Diagram for the Cyclic Creep Curve (curve A) in Fig. 1. (a).



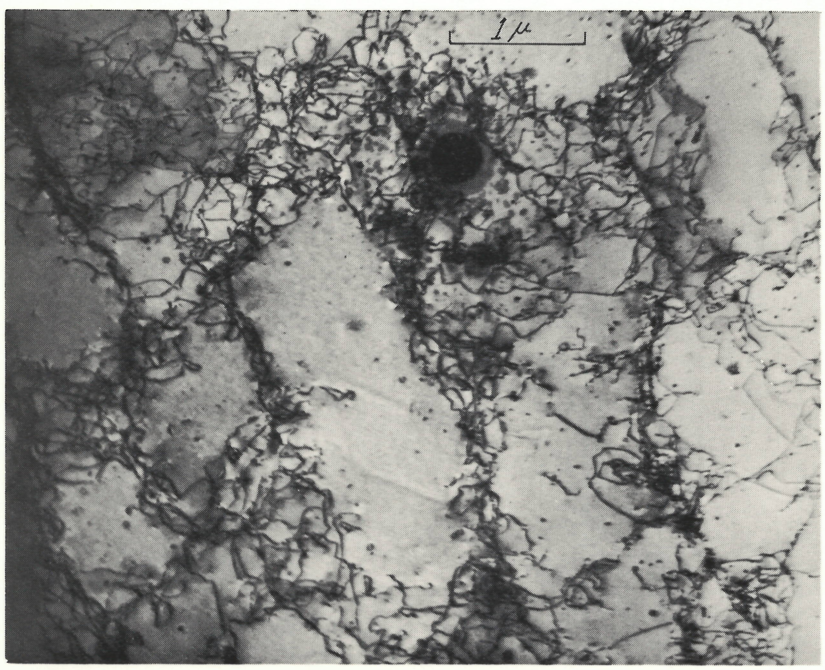
2. (a) Dislocation Arrangements after One Cycle of Load to a Peak Stress at 12,000 psi at 78°K (Equivalent to 10% tensile strain).



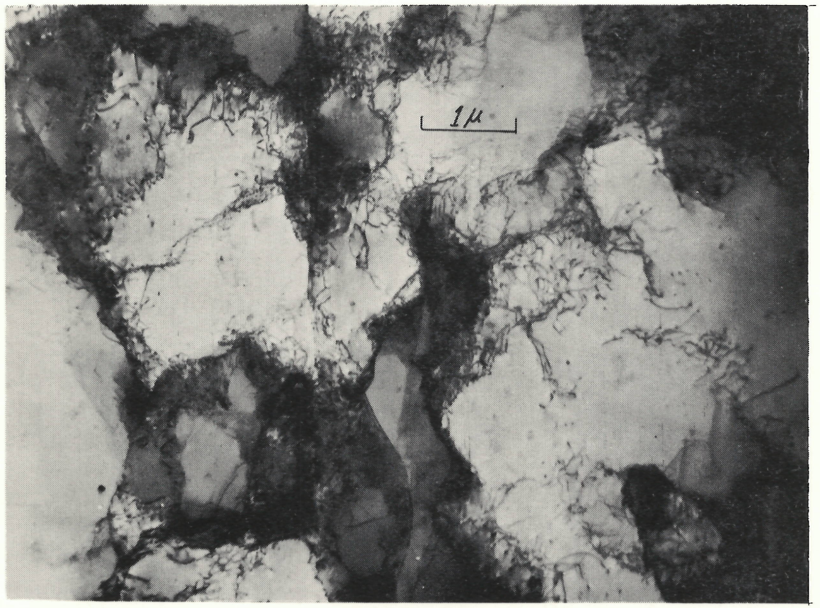
2. (b) Same specimens in (a) showing the variability sometimes observed in the dislocation pattern.



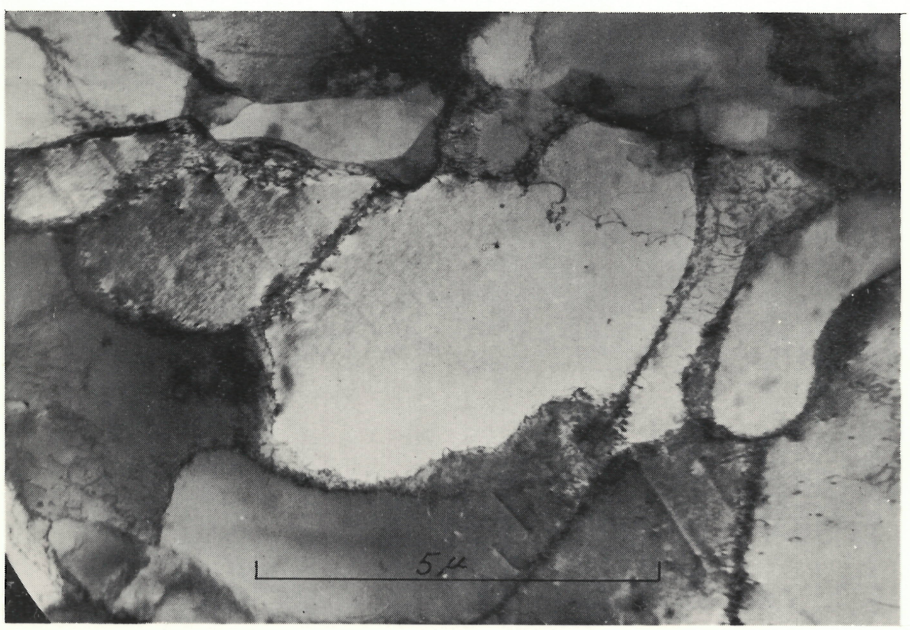
3. Dislocation Arrangement after 500 Cycles of Repeated Tension (12,000 psi) at 78°K.



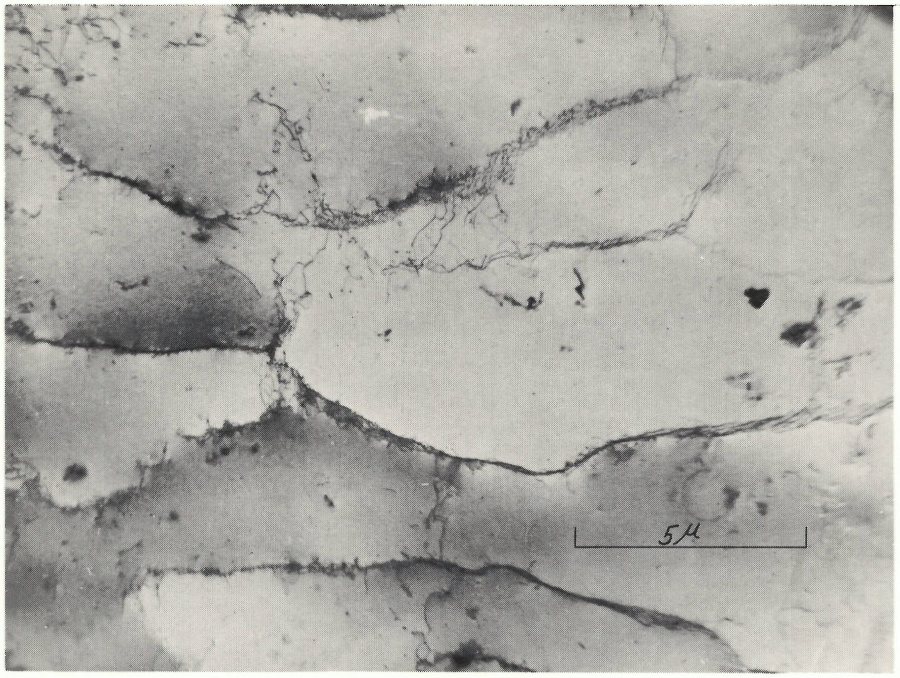
4. Dislocation Structure in Specimen which Failed after 3440 Cycles (12,000 psi) at 78°K. Note the small difference between the dislocation pattern in this Figure and that in Fig. 2 after only one cycle.



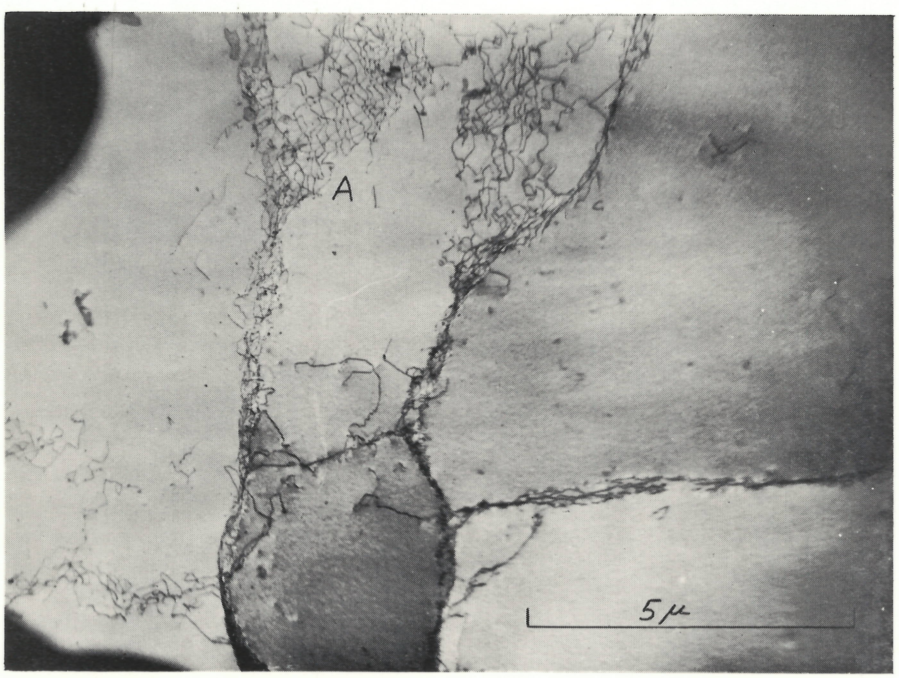
5. Dislocation Arrangement in Specimen Pulled to Failure in Static Tension at 78^{o} K. Compare with Fig. 4.



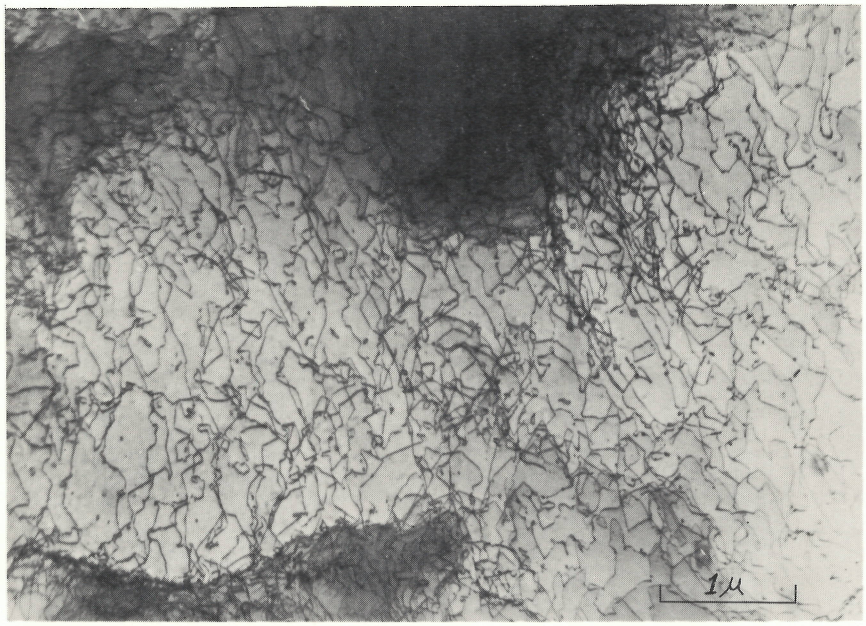
6. Well Defined Cell Structure in Aluminum at 300^{o} K after 14, 800 Cycles of Repeated Tensile Stress.



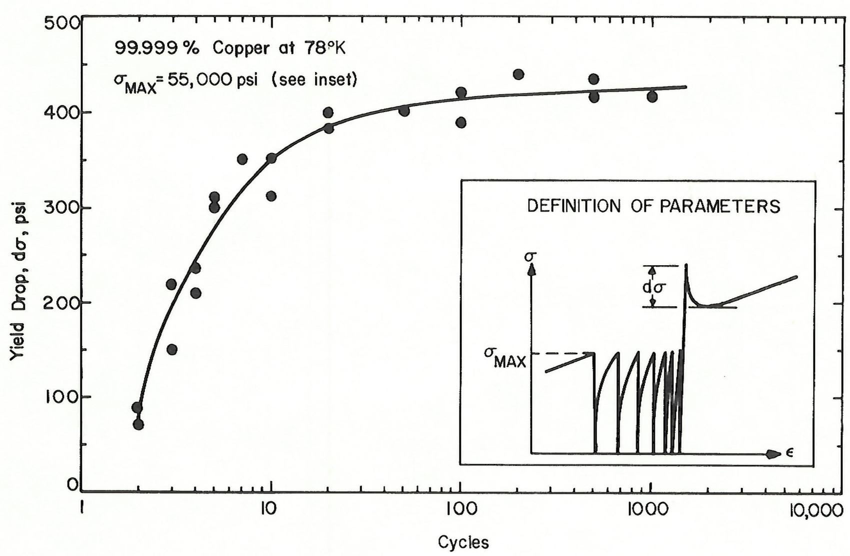
7. Cell Structure after 100, 000 Cycles (approx. 20% of life) of Repeated Tension (20, 000 psi) at 300° K.



8. Different Area of Same Specimen as in Fig. 7. Apparent Twist Boundary at A Lies Nearly Parallel to Plane of the Foil.



9. Cell Wall Lying in Plane of Foil (12, 000 psi for 500 cycles at 78°K). Note the Low Density of Dislocation Loops in this Micrograph and also in Fig. 8.



10. Increase in the Hardening of Copper Subjected to Repeated Tensile Stress. Inset shows Test Conditions.