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EFFECT OF INITIAL DISLOCATION DENSITY
ON SINGLE SLIP STRAIN HARDENING OF COPPER

Jack Washburn and Gollapudi Murty

August 1967

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Paper to be presented at the International Conference on the Strength of Metals and Alloys, Tokyo, Japan, Sept. 4-8, 1967. To be published in the Proceedings.

UCRL-17774

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AEC Contract No. W-7405-eng-48

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August 1967

ABSTRACT

Copper crystals in which different dislocation densities were introduced by multiple slip were subsequently deformed in tension in single slip. In all cases three stages were found on the stress strain curves. Detailed observations of surface slip markings were carried out on all specimens. It was shown that Stage I in prestrained specimens was due to progressive deformation starting at points of stress concentration and propagating through the gauge length. Stress strain curves and shear strain distribution were found to change in a regular way with initial dislocation density; the results from as-grown specimens are seen as part of a homologous series which includes all of the results from prestrained specimens. Therefore it is suggested that Stage I in pure copper is at least partly due to unavoidable prestrains during cooling, handling, and pre-yield loading.

Linking of slip clusters to form groups that grow entirely across the specimen was found during all stages of deformation, even for as-grown crystals. Surface slip markings that were formed at a given stress level, whether in Stage I of a prestrained crystal or in Stages II or III of an as-grown crystal, were found to be similar. It was suggested that the type

of instability of the dislocation substructure that is responsible for clustering of slip into definite bands and the existence of Stage I in prestrained specimens also develops gradually in a specimen deformed from the start in single slip and that the decrease in hardening rate in Stage III is associated with the instability of dense three-dimensional dislocation tangles within which the net Burgers vector is nearly zero.

1. INTRODUCTION

It is now well known that when a single crystal is deformed in tension along an axis that results in slip on a single glide plane, the stress strain curve typically has three stages: an initial region of small hardening rate, followed by a stage of approximately linear maximum hardening rate, and finally a region in which the rate of strain hardening continuously decreases. This characteristic S shape first became well verified for FCC crystals,[1] but is now found to be a more general result. Similar curves are obtained for BCC,[2] Diamond Cubic[3] and NaCl structure crystals.[4] The stress strain curves for alloy crystals and for irradiation or quench hardened specimens also have the same general shape. In these cases the region of low hardening rate that follows yielding is clearly associated with non-uniform distribution of the instantaneous strain along the length of the specimen. Slip starts at a point or points of stress concentration such as at the grip sections of a tensile specimen and spreads gradually along the crystal at nearly a constant stress level or at least with only a slow increase in stress as an increasing fraction of the volume is strained. Only when the entire volume has been strained by this first wave of slip does the hardening rate increase. In all of these cases a type of hardening exists initially which is unstable in the presence of the primary dislocations that begin to multiply at points of

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(1) J. Diehl: Z. Metallk., 47 (1956), 331.
(2) R. A. Foxall, M. S. Duesbery and P. B. Hirsch: Canadian J. Phys., 45 (1967), 607; and A. S. Keh and Y. Nakada, 1101.
(3) H. Alexander and S. Mader: Acta Met., 10 (1962), 887.
(4) D. Hoover and J. Washburn: J. Appl. Phys., 33 (1962), 11.

stress concentration. For irradiated and for quenched and aged FCC metals the initial hardening is due to a high density of small vacancy clusters either in the form of voids or small dislocation loops. It is well established that prismatic loops are swept away by moving dislocations.[5] Therefore, when a dislocation moves along one glide layer, it becomes easier for others to move on close-by parallel glide layers. The result is growth of clearly defined slip bands. The shear strain within a band continues to increase until the more stable kind of hardening within the band is of the order of the hardening due to the loops in the surrounding material. Under these conditions deformation should obviously be progressive and the stress required for continuing elongation should not rise much as long as new bands of slip can form in previously undeformed volumes.

Another type of "unstable" hardening exists initially in whisker crystals and in many diamond cubic and NaCl structure specimens. There are few, if any, mobile dislocations initially present. Deformation must spread through the specimen by cross slip starting from a few points of severe stress concentration.

It is the purpose of the present paper to suggest that small initial hardening rates and the clustering of slip in crystals oriented for single slip may always be due to the existence of a type of hardening that is unstable in the above sense when large numbers of moving dislocations on the primary system are generated at favorable sites. Under single slip conditions the replacement of this hardening by a relatively more stable arrangement of defects generally requires large plastic strains. Therefore, an

(5) J. Strudel and J. Washburn: Phil. Mag., 9 (1964), 491.

easily observable region of low hardening rate is found as the initial part of the stress strain curve.

It is possible that even for "as-grown" pure FCC metals, Stage I should not be considered as a special case. Most single crystal specimens from which stress strain curves have been obtained were grown from the melt or by recrystallization and subsequently cooled at a fairly rapid rate from the growth temperature. For pure FCC crystals, dislocations move and multiply at such low stresses[6] that during cooling, handling, mounting in the testing machine, and loading within the pre-yield range, large numbers of dislocations move and multiply. Also, the stresses developed during these operations do not often produce slip only, or even primarily, on the slip system that eventually becomes the primary one. Therefore, it seems likely that few, if any, single slip stress strain curves for pure FCC metals have been recorded for which the specimen had not been subjected to some pre-strain in multiple slip. In a previous report[7] the result of intentionally prestraining by various amounts in multiple slip prior to testing in single slip has been described. Some of the results of these experiments are important for the purposes of the present discussion and are included in this paper. These experiments show that prestraining in multiple slip causes changes in the stress strain curve and in the slip markings that are strikingly similar to those caused by irradiation hardening. They suggest that the initial stage of low hardening rate, even for pure copper, may be due, at least in part, to unintentional multiple slip prestrains.

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(6) F. W. Young: J. Appl. Phys., 32 (1961), 1917.
(7) J. Washburn and G. Murty: Canadian J. Phys., 45 (1967), 523.

2. EXPERIMENTAL PROCEDURE

Details of the experiments on which this discussion is based have been described previously[7] and will not be repeated in this paper.

Large seeded copper crystals oriented as shown in Fig. 1a were grown from the melt and prestrained by various amounts in tension along a [111] axis or by explosive shock loading. Smaller tensile specimens were then cut from them which were oriented for single slip. The active slip plane for the new specimens was (111). This was the inactive plane during the tensile prestrain.

The crystals were extended in an Instron testing machine at a strain rate of 6.6×10^{-5}/sec. Shear stress shear strain curves were computed from the load elongation data by assuming that lattice rotation resulted from glide on the primary system only.

Surface observations were made at all stages of deformation at low, high, and intermediate magnifications. In order to follow the growth of slip markings, several consecutive observations of the same area were made following small strain increments without any intermediate polishing.

For the electron microscope replica observations a two stage formvar carbon replica technique was used.[8] This permitted consecutive observations of the same area because removal of the replica from the surface did not destroy the markings. A Hitachi HU 11A electron microscope was used for observation of the replicas. In some cases shear displacements for individual slip bands were measured using the latex ball method.

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(8) J. T. Fourie: J. Appl. Phys., 29 (1958), 608.

3. RESULTS AND DISCUSSION

3.1. Stress Strain Curves

Tensile stress strain curves[7] for an as-grown and for four prestrained specimens are shown in Fig. 2. All of the curves could be described as having the typical three stages characteristic of FCC tensile specimens oriented for single slip. However, the first stage for the specimen prestrained in multiple slip to 2.54 kg/mm^2 occurred at a stress level which would have been in Stage III in the as-grown specimen. Clearly the three stages for all these curves cannot be explained by the usual arguments which attribute the beginning of Stage II to the onset of dislocation multiplication on secondary systems and the beginning of Stage III to the start of extensive thermally activated cross slip.

Increasing the initial dislocation density by prestraining in multiple slip or by shock loading caused regular changes in the stress strain curve resulting in a homologous series of curves. The stress level of the initial small hardening stage was related to the maximum resolved shear stress reached during the prestrains and was always about 30% higher. The extent of the first stage increased and the hardening rate decreased with increasing initial dislocation density. A second "linear hardening" stage always followed Stage I. Its length also increased and its slope decreased with increasing prestrain. The stress level at the beginning of Stage III increased with increasing prestrain.

All of the curves eventually approached each other at very large strains, but it was clear that the initial dislocation density had a marked effect on the shape of the entire stress strain curve.

3.2. Distribution of Plastic Strain

There has been a regretable tendency in the literature to use the terms: "fine slip" and "coarse slip", "uniform slip" and "Luders band deformation". The present observations suggest that these labels are more confusing than illuminating because they imply sharp distinctions where no sharp distinctions exist. Slip in pure copper is always fine slip in the sense that large shear displacements on a single atomic glide layer are never found; it is always non-uniformly distributed in that slip markings appear on an initially polished surface when viewed at an appropriate magnification. The uniformity with which fine slip is distributed among all the possible glide layers changes in a systematic way as the dislocation density and flow stress level increase. However, the changes cannot be adequately described by the terms: "fine slip", "coarse slip", or "uniform slip". Also, the apparent uniformity or fineness or coarseness of slip markings depends critically on experimental variables such as: the magnification at which the surface is observed, the lighting or replica technique used to make surface contour visible, the amount of plastic strain that has occurred since the last polishing of the surface.

These considerations make it very difficult to define what is meant by the term "slip band". The measurement of slip band lengths or slip band spacing or slip band shear displacement is certainly a highly subjective process. Also, as pointed out later in this paper, slip clusters usually are correlated or linked together. Therefore, at least for pure copper, these measurements are of questionable value.

3.3. Stage I Deformation

In Figs. 3, 4 and 5 surface markings on an as-grown crystal are shown after 1, 3, and 5% strain in Stage I. The electron microscope replicas (Fig. 3) show only about a 6μ length of the crystal. Within this length the shear strain is fairly uniform. "Fine slip" events appear to have taken place approximately at random spacings. In Fig. 4 a 220μ length of the crystal is visible. At this scale it is obvious that there was clustering of the fine slip. The entire area shown in Fig. 3 represents an area of about 2 mm × 3 mm on Fig. 4. The most prominent markings of Fig. 3 might be called "slip bands" in that they clearly consist of regions of fine slip concentration. However, the much larger regions of concentrated shear displacement observed in Fig. 4 could alternatively be considered as slip bands. In Fig. 5, where a one-centimeter length of the specimen is shown and on which the entire area of Fig. 4 would cover a region about 1 mm by 1.5 mm, it can be seen that the short regions of concentrated slip seen on Fig. 4 were themselves arranged in a correlated way so that on the scale of Fig. 5 there seem to be bands of concentrated shear displacement that extend entirely across the specimen. Finally, when the specimen, as seen in Fig. 5a, was viewed without any magnification, there appeared to be a further larger-scale concentration of slip near the left end and another near the center of the gauge length. This concentration of slip markings at two regions along the specimen length can be clearly seen in Fig. 5a.

On the basis of the high magnification electron microscope observations of slip markings in Stage I the shear strain distribution could be

characterized as "uniform fine slip". However, at an intermediate magnification, clusters of fine slip began to appear right after the yield and as Stage I continued their number increased so that gradually the entire field of view was covered with prominent markings. At the lowest magnification slip markings appeared first in only certain regions of the specimen and as deformation continued the entire length gradually became covered.

Figs. 6 and 7 show slip markings on a crystal that was prestrained by an explosive shock load which resulted in raising the stress level of "Stage I" to about 2 kg/mm^2. As is seen in the electron microscope replicas of Fig. 6, the slip is still fine slip but the tendency to cluster is now more evident even at high magnification. At the intermediate magnification of Fig. 7 it is clear that the clusters of fine slip were correlated with other clusters to form groups of slip clusters that started from one edge of the crystal and grew across it by the addition of new fine slip clusters in the regions of stress concentration at the ends of the groups. Propagation of slip across the crystal was like the growth of a mechanical twin or the growth of a crack. In addition to this progressive growth of individual groups of slip clusters, it was obvious even at intermediate magnification that these groups started to form first near the macroscopic stress concentrations at the ends of the specimen and gradually filled the gauge length. At very low magnification the front between undeformed and deformed parts of the crystal was much sharper than was the case for the as-grown lower dislocation density crystal.

Figs. 8 and 9 show electron microscope replicas and intermediate magnification optical micrographs of slip markings on a crystal which was

prestrained in multiple slip to achieve a "Stage I" stress level of about 3 kg/mm^2. In this case the fine slip was sharply clustered even at the highest magnification and the correlation between fine slip clusters to produce slip groups that grew across the specimen was again observed.

For the heavily prestrained crystals "Stage I" clearly had a small slope, because the deformation was progressive. Undeformed regions of the specimen were gradually filled by slip cluster groups. This progressive nature of the deformation in Stage I became less obvious for smaller prestrains. As the initial dislocation density was decreased, the fronts between regions containing slip markings and undeformed parts of the specimen became more spread out or diffuse. In order to see clustering of the fine slip, it was necessary to observe at lower magnification. However, it was not clear that the "as-grown" crystal should be considered as a special case. Because of the ease with which dislocations move and multiply in pure copper and the difficulty of achieving perfect alignment in a tensile test, it is doubtful that many stress strain curves have been recorded for which there were not unintentional prestrains. This is especially true if the initial dislocation density is less than 10^5 cm/cm^3.

If the initial stage of low hardening rate is attributed to progressive filling of the specimen with slip cluster groups, even for as-grown crystals, then the strain hardening rate during this stage must be determined by the uniformity of the pre-existing dislocation and point defect substructure. The smaller the prestrain, the less is the chance that it will be uniform along the entire length of a single crystal specimen. Therefore, the slope of Stage I should be expected to be greater for the most nearly perfect crystals. A frequent cause of macroscopic non-uniformity

in highly perfect specimens is pre-yield operation of a few sources on secondary systems. Because of the low dislocation density, loops emanating from active sources can spread over large areas of a secondary slip plane. In this way barriers to primary dislocation motion are likely to be created in some, but not all, parts of the specimen.

The systematic lengthening of Stage I and the increasing tendency for clustering of the fine slip events as the amount of prestrain is increased suggests that the dislocation arrangement introduced by the prestrain is unstable in the presence of primary dislocations in a way similar to the loop substructures that form after quenching and aging or after irradiation. When the applied stress becomes high enough to start multiplying primary dislocations in a region of stress concentration, the chance that other primary loops can be formed on nearby parallel layers must be increased. As previously suggested by the authors[9] three dimensional arrangements of dislocations for which the net Burgers vector is nearly zero should generally be stable only under the conditions that existed when the array was formed. The introduction of a few foreign dislocations should frequently cause instability and set off a sequence of rearrangements with consequent annihilation of a length of dislocation line that in some cases may even be greater than the length of foreign dislocation that was introduced. Detailed examples of annihilation that could be set off by the arrival of a primary dislocation at an existing metastable tangle of dislocations have been described previously.[9] These examples are necessarily highly idealized. The actual sequences involving formation of

(9) J. Washburn and G. Murty: J. Appl. Phys., 37 (1966), 2511.

new attractive junctions and displacement of many surrounding dislocation segments and nodes would generally be too complex to be easily visualized. However, the net effect should often be the bringing together of dislocation segments of opposite sign and the introduction of new mobile segments of primary dislocation on different parallel glide layers.

As dislocation density increases, this kind of instability would be expected to increase, because attractive forces between neighboring segments of dislocation of opposite sign increase as 1/r where r is the average distance between them. For example, below a critical separation a pair of screw dislocations of opposite sign in a FCC crystal cannot continue to exist even if both are split on the primary plane and they lie on different glide layers, because their attraction stress becomes great enough to cause cross slip. There should be a maximum dislocation density above which three-dimensional arrangements of dislocations spontaneously break down into two dimensional arrays in which only the net excess dislocations of one sign remain. This maximum dislocation density should increase with decreasing temperature and stacking fault energy.

3.4. Mechanism of Slip Cluster Formation

During the linear hardening stage, it is usually assumed that the shear strain is "uniformly" distributed. This is only true on the macroscopic scale. On a microscopic scale the slip line observations show that slip still propagates from one point to another in a highly correlated way. Fig. 10 shows growth of slip cluster groups on a surface that was polished after the start of linear hardening in the heavily prestrained specimen. Figs. 11, 12, 13 show surface markings produced by the final 2% strain at three different total strains within the linear hardening ranges of two

prestrained specimens and the as-grown crystal respectively. Correlation between the slip markings that form within a small strain increment is most obvious in the prestrained specimens, but can still be recognized in the as-grown crystal. If surface observations are made after larger strain increments, information as to the order in which individual slip clusters are formed is lost, because the number of slip clusters becomes so great that it is no longer possible to identify individual slip cluster groups. For the "as-grown" crystal, even the 2% strain increment was too great for the observations made at 15% and 23% total strain.

The results suggest that new slip clusters always start only at regions of stress concentration. During the earliest stages of plastic deformation, these are at external surfaces near changes in section and along specimen edges. As deformation proceeds, increasingly numerous regions of favorable internal stress concentration are developed where, within a previously formed slip group, there had been a particularly large step in the direction perpendicular to the primary plane. At some of these sites relaxation by operation of secondary systems or by cross slip is probably not complete.

It is proposed that the formation of slip clusters takes place in the following way: when a loop of dislocation expands on a given glide layer of the primary system, it is pulled into the cross-slip plane at many points where it forms an attractive junction.[10] Strain multiplication and the formation of a slip cluster becomes possible only when these segments that lie in the cross-slip plane can be made to bow out on new glide layers of the primary plane near the original one. The stress to cause

(10) J. Washburn: Appl. Phys. Letters, 7 (1965), 183.

bowing of these segments should be determined by the spacing of the forest dislocations. When the forest contains approximately equal numbers of dislocations of opposite sign, the first dislocation that moves across the original glide layer promotes annihilation of some segments of the forest. This increases locally the average spacing of forest dislocations for a distance, above and below the original layer, that should itself be proportional to the average distance between dislocations. Therefore in Stage I of an as-grown crystal the slip clusters are very diffuse and can only be recognized at low magnification. According to this model the original loop not only introduces many segments into the cross-slip plane, but it also makes it easier for them to bow out on new layers near the first. A slip cluster is formed by the simultaneous bowing of many separate segments on close-by parallel glide layers. Wherever opposite screw dislocations meet, they annihilate by cross slip which also is initiated at intersections. The edge segments meet to form dipoles or more complex bundles of edge dislocations.

During all stages single slip is stabilized by this process. However, when the tensile axis is near one of the multiple slip orientations, there is also multiplication on secondary slip planes which causes forest density to rise rapidly with increasing strain. The increase in forest density with strain is due to combined action of the applied stress and internal stresses associated with groups of primary dislocation of one sign.

3.5. Stage III

The observations on slip markings in all stages, even for the "as-grown" crystal, indicate that slip is clustered and that slip clusters link

together to form groups or avalanches of slip that grow progressively across the specimen. As the flow stress increases, due to strain hardening, the average strain within clusters increases because they become narrower. As a result the linking of slip clusters into groups becomes more apparent at high magnification. However, since linking of slip clusters occurs at all stages, the decrease in hardening rate during Stage III cannot be caused by it.

The general appearance of slip markings produced in Stage III of an as-grown crystal and those formed at the same stress level during Stage I on a specimen that has been prestrained in multiple slip is strikingly similar. If clustering of slip in the latter is due to inherent instability of a three-dimensional dislocation tangle that has a net Burgers vector near zero, it is reasonable to suggest that the same type of instability exists within the dislocation arrangement in a crystal that has been deformed from the start in single slip. Electron microscope observations[11] show clearly that as the stress level rises during Stage II, profuse multiplication of the length of dislocation lines of all Burgers vectors occurs. Only those of the primary system move far enough to make an important contribution to the total plastic strain. If all dislocations of secondary systems move only short distances during a great increase in the total length of line present, then it is obvious that in each of the small volumes within which the multiplication takes place the net Burgers vector must be near zero. This kind of dislocation substructure certainly becomes increasingly unstable as the dislocation density rises. Therefore,

(11) U. Essman: Phys. Stat. Sol., 17 (1965), 725.

the increased tendency for slip to cluster and the decrease in rate of strain hardening during Stage III reflect an approach to a maximum dislocation density above which three-dimensional tangles will spontaneously break down into two-dimensional subgrain boundaries. Cross slip is an essential process for the rearrangements within a three-dimentional tangle that permit annihilation of segments of opposite sign. Therefore, this model is consistent with the many experimental observations which show that the stress level and therefore the dislocation density at which Stage III begins increases with decreasing temperature and stacking fault energy.

4. SUMMARY

Stress strain curves of many different single crystal tensile specimens deformed so as to produce slip on a single system, have an S shape: an initial region of small hardening rate followed by a region of maximum hardening rate and finally a region where the hardening rate continuously decreases. In the vast majority of cases, the initial region of low hardening rate is associated with progressive deformation which gradually spreads through the volume of the specimen. This type of deformation reflects the presence of an unstable type of hardening such as a lack of slip sources or an initial defect arrangement that can be swept away by moving dislocations. When unstable hardening exists, operation of a source on one glide layer tends to promote slip on nearby parallel glide layers so that slip is strongly clustered and appears as definite slip bands even at higher magnification.

Single slip tensile tests on prestrained copper single crystals show that the arrangements of dislocations produced by multiple slip are unstable in the above sense. The initial stage of the new stress strain curve clearly has a small hardening rate, because the deformation is progressive. Slip starts only at a few points of stress concentration and gradually spreads through the specimen. As the amount of multiple slip prestrain is reduced the progressive character of slip in Stage I is increasingly difficult to observe at high magnification. However, what appears to be uniformly distributed fine slip at high magnification is still definitely non-uniform at very low magnifications. The observations suggest that Stage I in "as-grown" pure copper may at least be made more

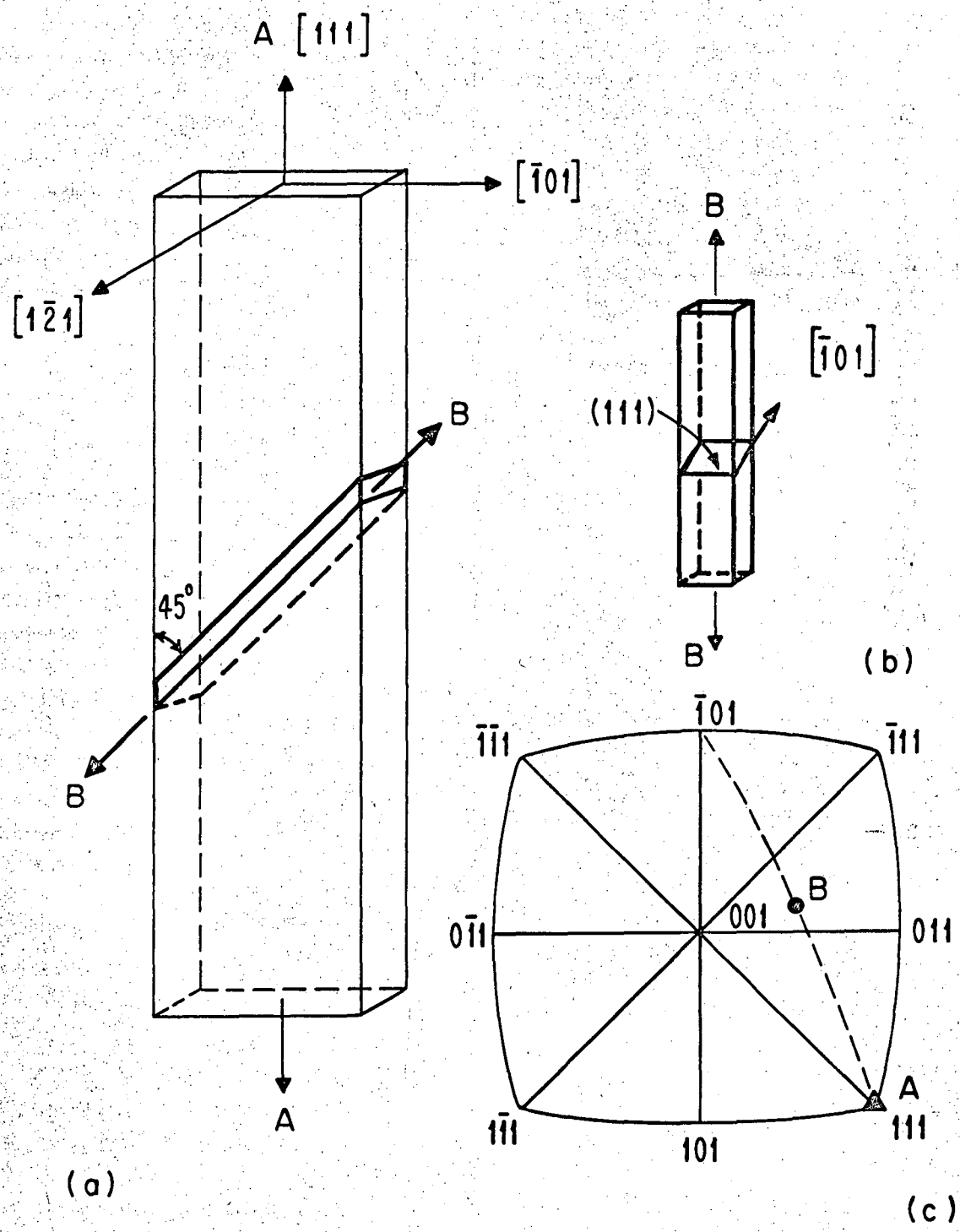
prominent by unintentional multiple slip prestrains during cooling, handling, and initial pre-yield loading. If this prestrain could be completely avoided the yield stress would be lower and Stage I would be shorter or even completely absent.

For prestrained specimens the deformation in Stage II is still non-uniformly distributed on the microscopic scale. If the slip markings formed by the last small increment of strain are observed, it is clear that slip clusters are linked together and tend to grow entirely across the specimen by the progressive formation of new slip clusters near the front of an advancing group. This same linking of slip clusters was less obvious but still observable in the as-grown crystal.

Since linking of slip clusters occurs at all stages, it cannot be used as an explanation for the beginning of Stage III. The striking similarity between the slip markings at any given stress level in Stage II or III in the "as-grown" crystal to those formed at the same stress level in Stage I of a specimen prestrained the necessary amount suggests that deformation from the start on a single slip system also finally produces an arrangement of dislocations that is unstable. As the maximum density at which this arrangement would spontaneously break down into two-dimensional arrays is approached, annihilation of dislocation segments, within the most dense regions of substructure near a growing slip cluster, should gradually become equal to the new lengths created in other less dense regions. When these two lengths do become equal near the end of Stage III, then dislocation density and flow stress stop increasing with further strain.

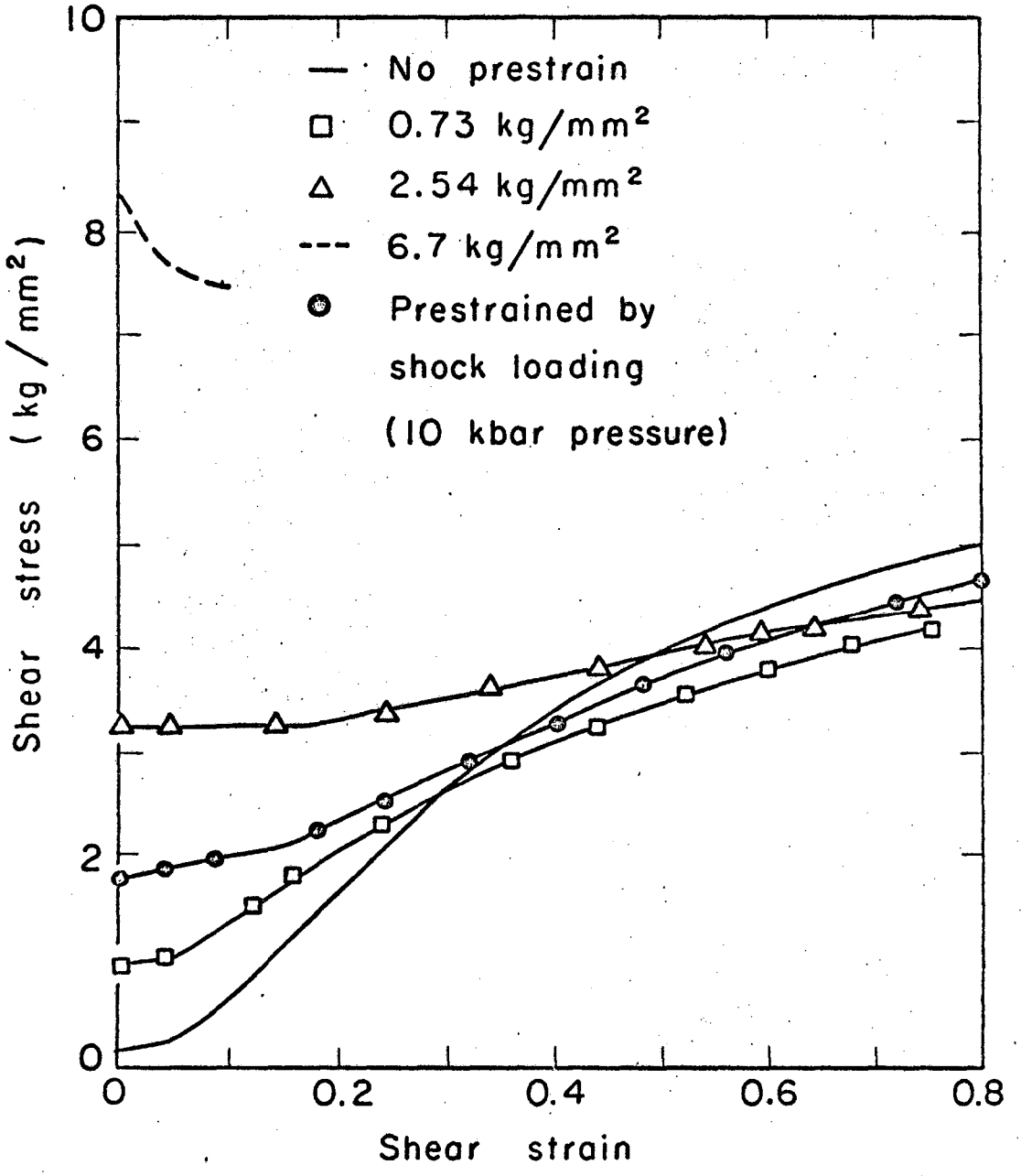
ACKNOWLEDGMENTS

The support of this work by the United States Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Radiation Laboratory is gratefully acknowledged.



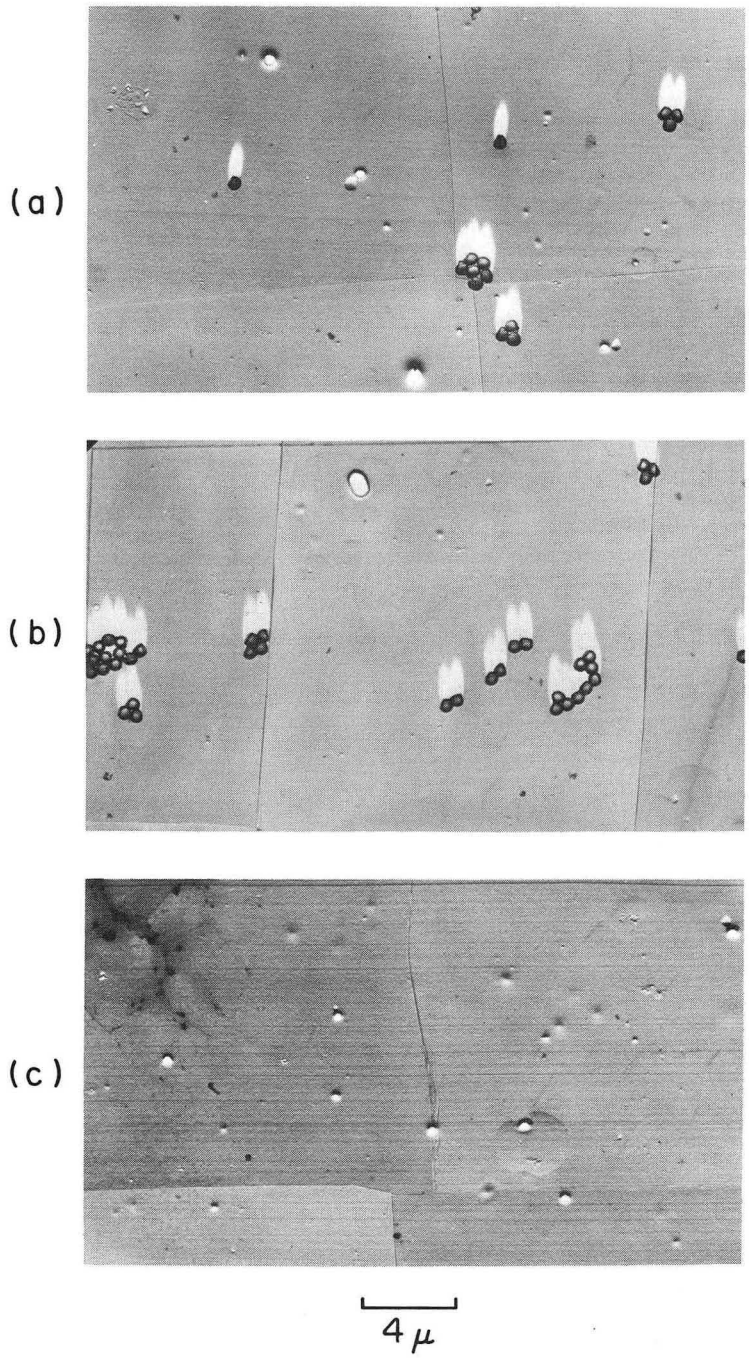
MUB-11515

Fig. 1 Specimen orientations (a) prestrain; (b,c) test specimens.



MUB-11514

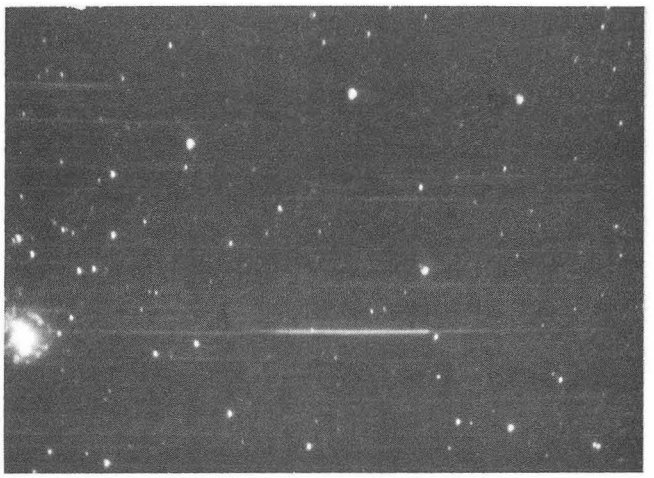
Fig. 2 Single slip stress strain curves showing effect of increasing initial dislocation density by prestrain in multiple slip.



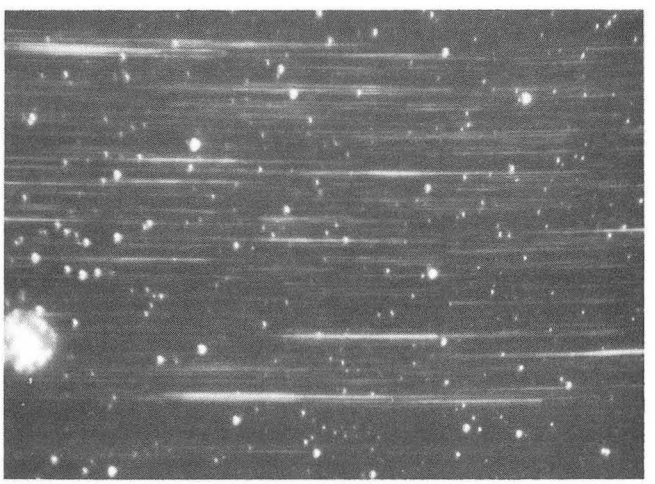
ZN-5970

Fig. 3 Electron microscope replicas showing surface slip markings in Stage I for an as-grown crystal (a) 1%; (b) 3%; (c) 5%. Same area is shown at all strains.

(a)



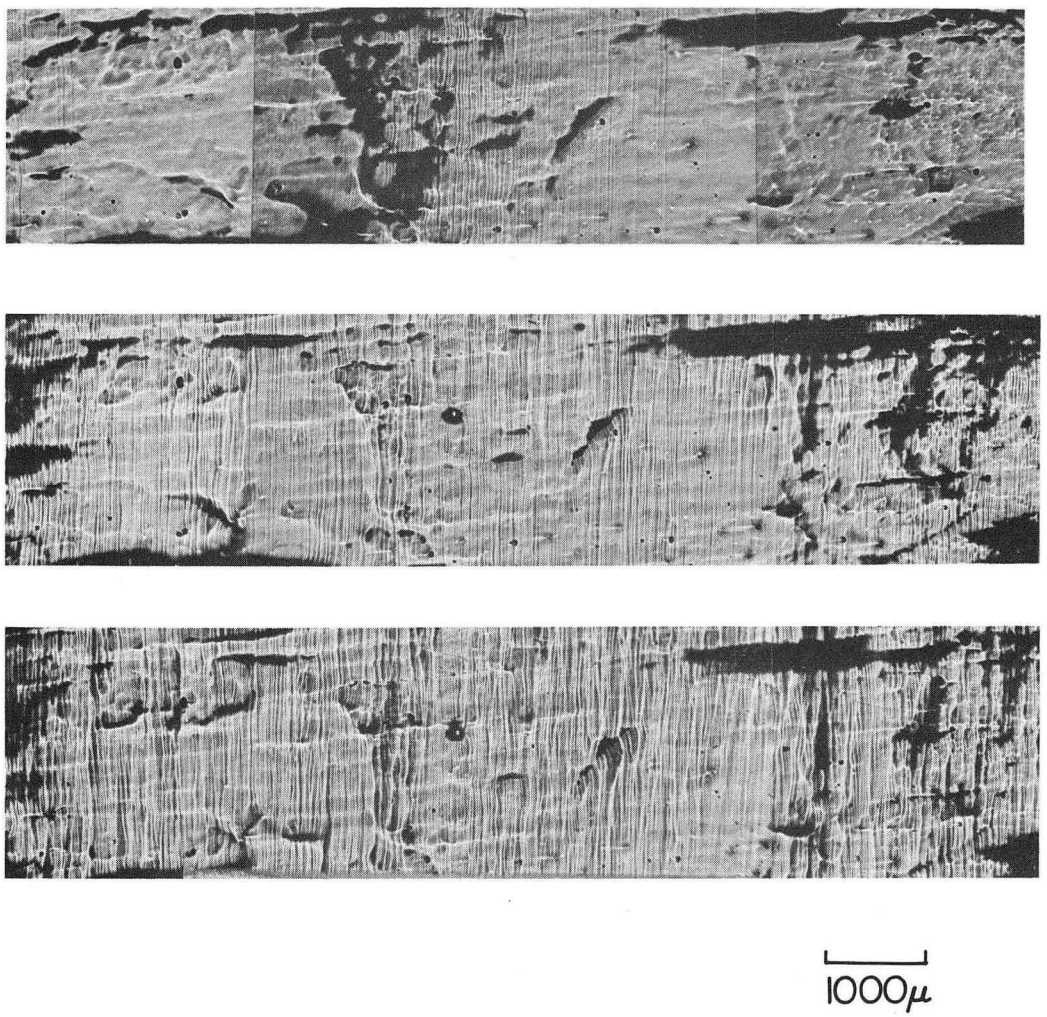
(b)



100μ

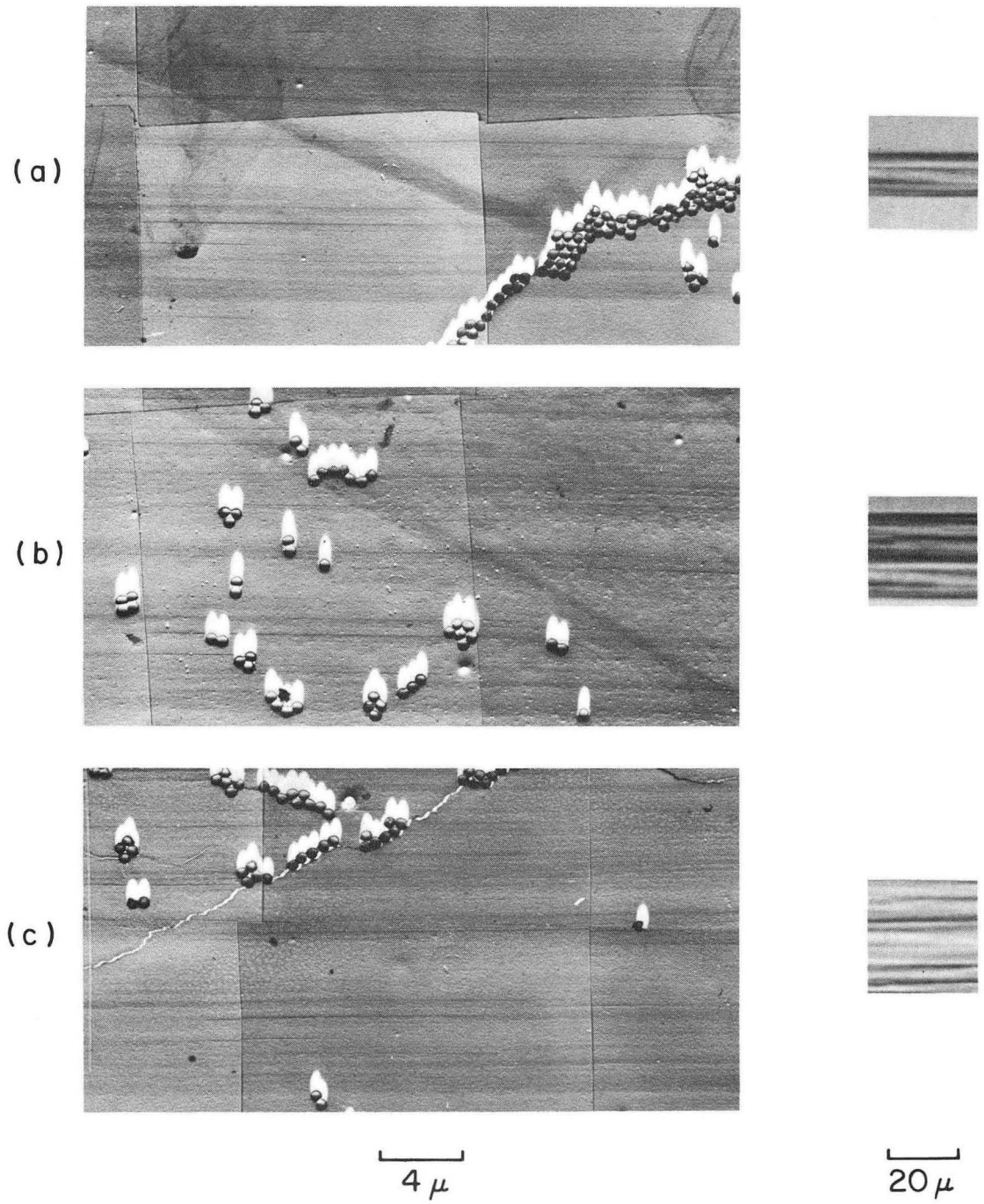
ZN-5678

Fig. 4 Dark field optical micrographs of the same surfaces shown in (a) and (c) of Fig. 3



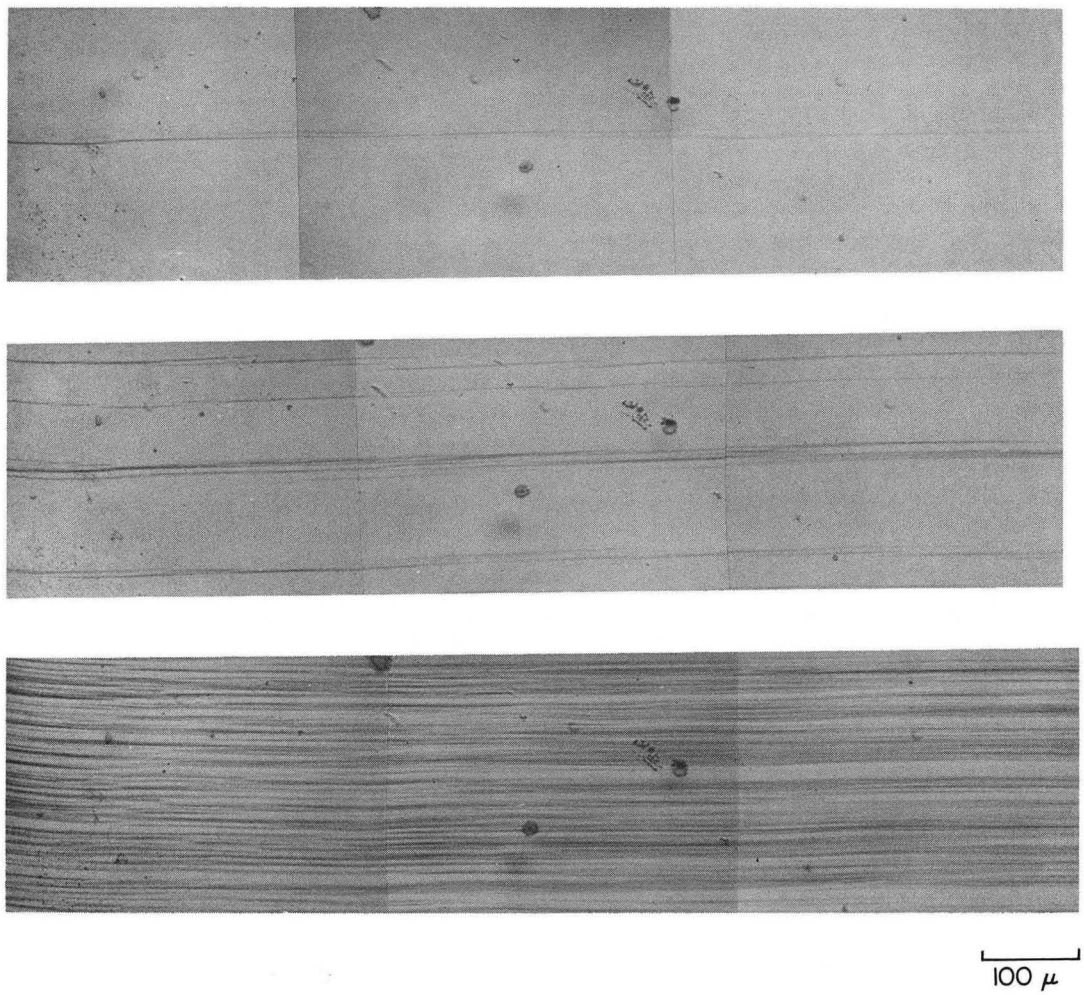
ZN-5958

Fig. 5 Low magnification macro-photograph of a crystal for the same strains as Fig. 3.



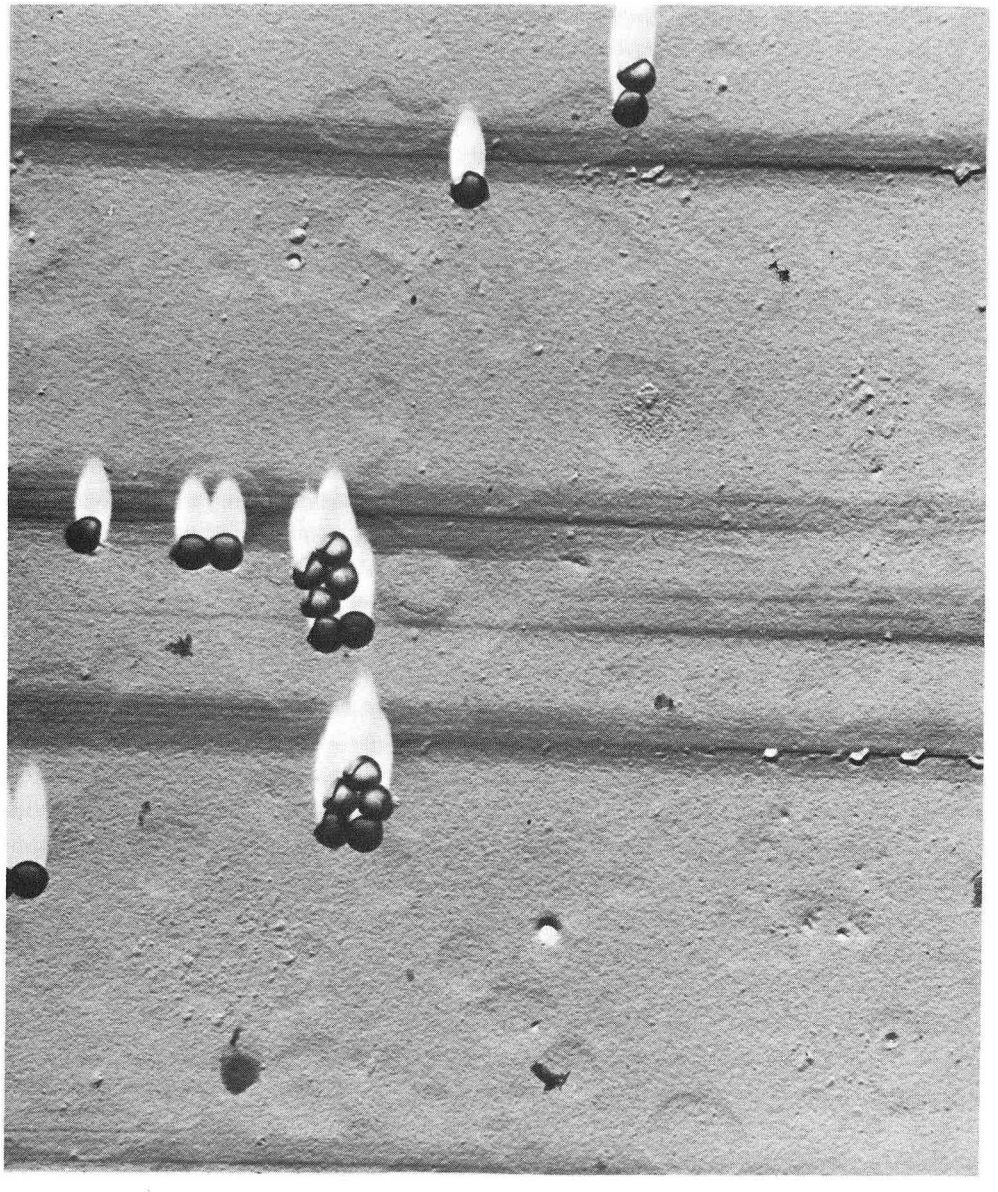
ZN-5963

Fig. 6 Electron microscope replica photographs showing slip marking in "Stage I" for a crystal prestrained by explosive shock loading. Same area of crystal surface is shown at (a) 4.2%; (b) 6.2%; and (c) 12% shear strains.



ZN-5957

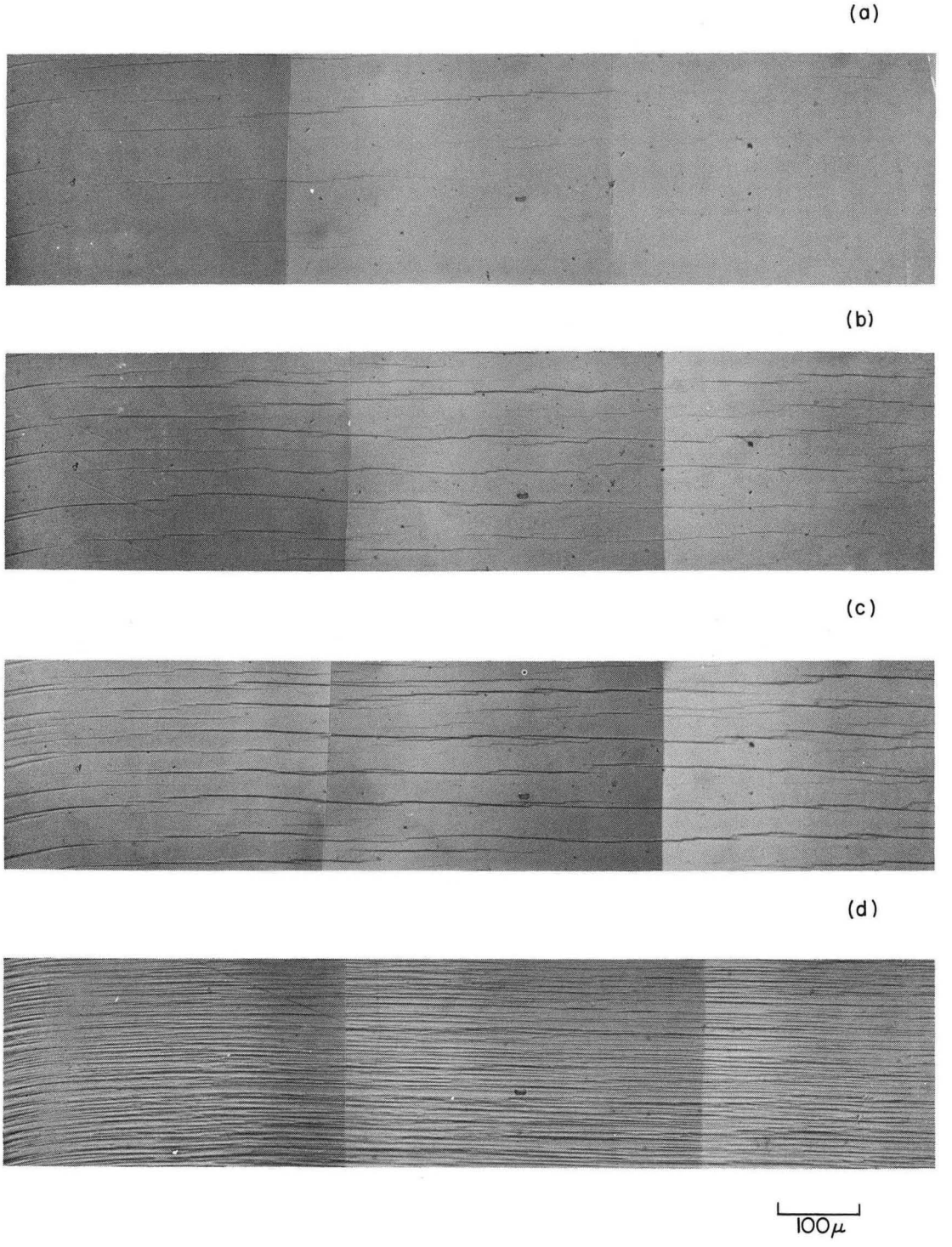
Fig. 7 Growth of fine slip cluster groups starting at left edge and growing across the crystal during "Stage I" of shock loaded crystal. Same area is shown at (a) 0.5%; (b) 0.85%; and (c) 2% shear strains.



2 μ

ZN-5690

Fig. 8 Electron microscope replica showing slip markings formed during "Stage I" on a crystal prestrained to 2.54 kg/mm^2.

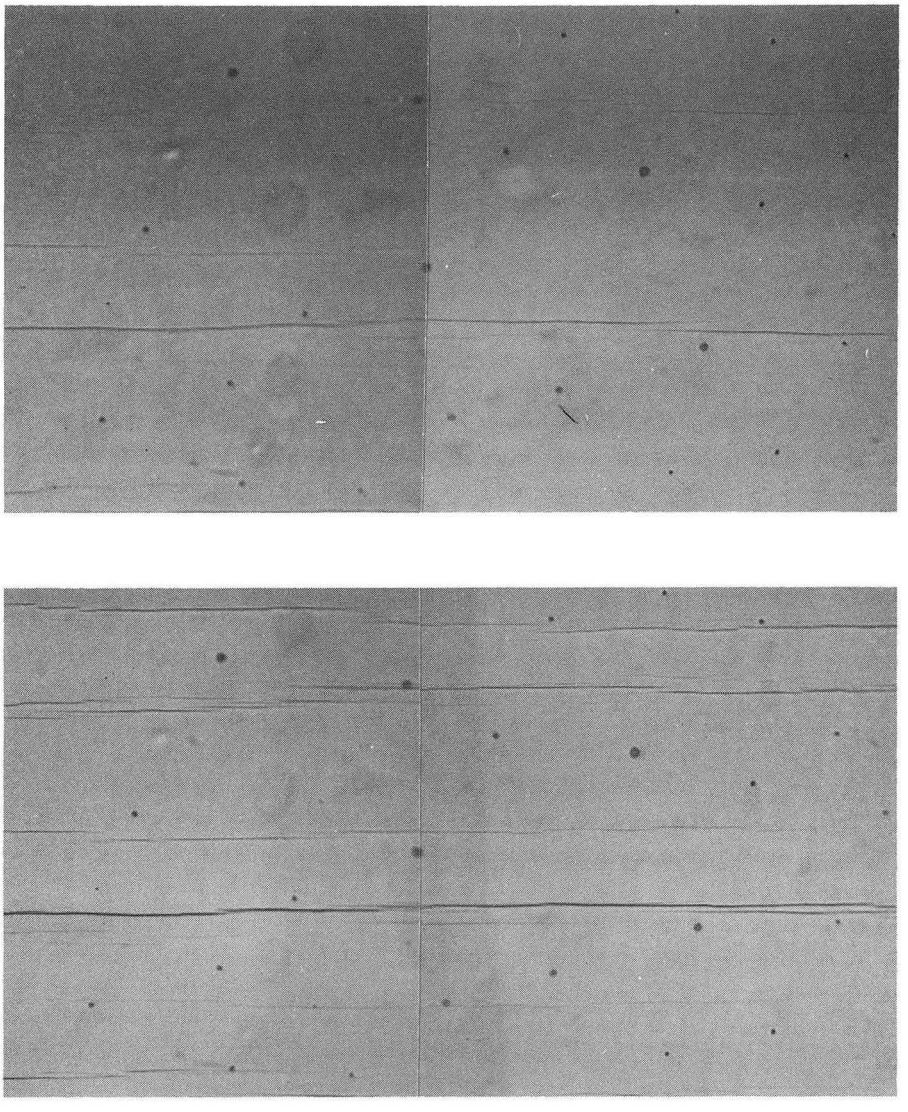


ZN-5959

Fig. 9 Growth of slip cluster groups during "Stage I" of crystal prestrained to 2.54 kg/mm^2, (a) 4.8%; (b) 5.7%; (c) 6.0%; and (d) 9.5% shear strains.

(a)

(b)



ZN-5694

Fig. 10 Successive pictures of the same area of the surface with an intermediate increment of strain of 0.1% during "Stage II" linear hardening of a crystal prestrained to 2.54 kg/mm^2.

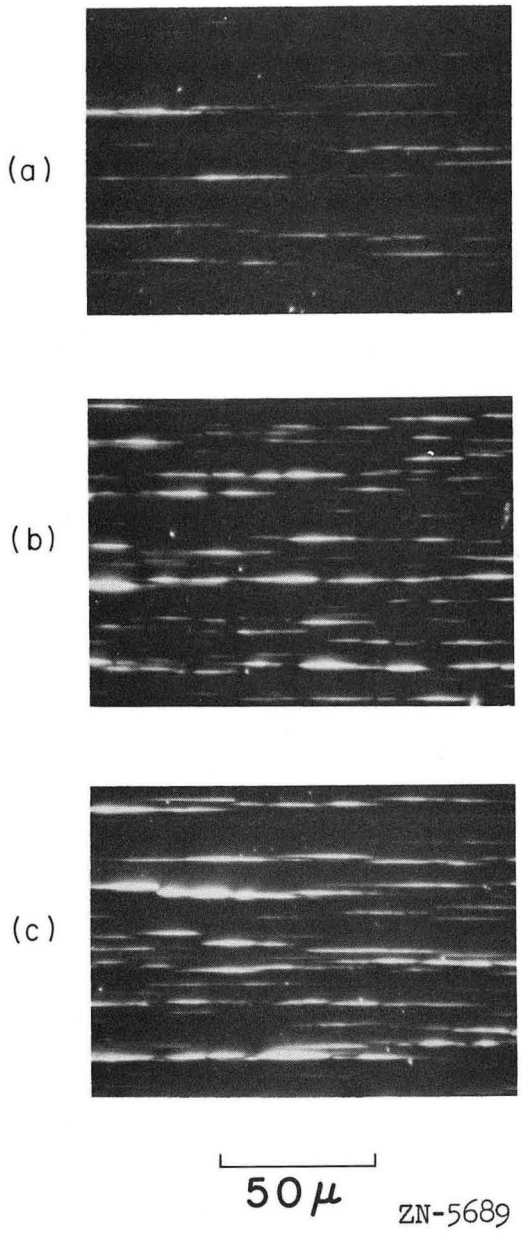


Fig. 11 Dark field photographs of slip markings formed by the final 2% strain at total strains in Stage II of (a) 23%; (b) 33%; (c) 43% on a crystal prestrained to 2.54 kg/mm^2.

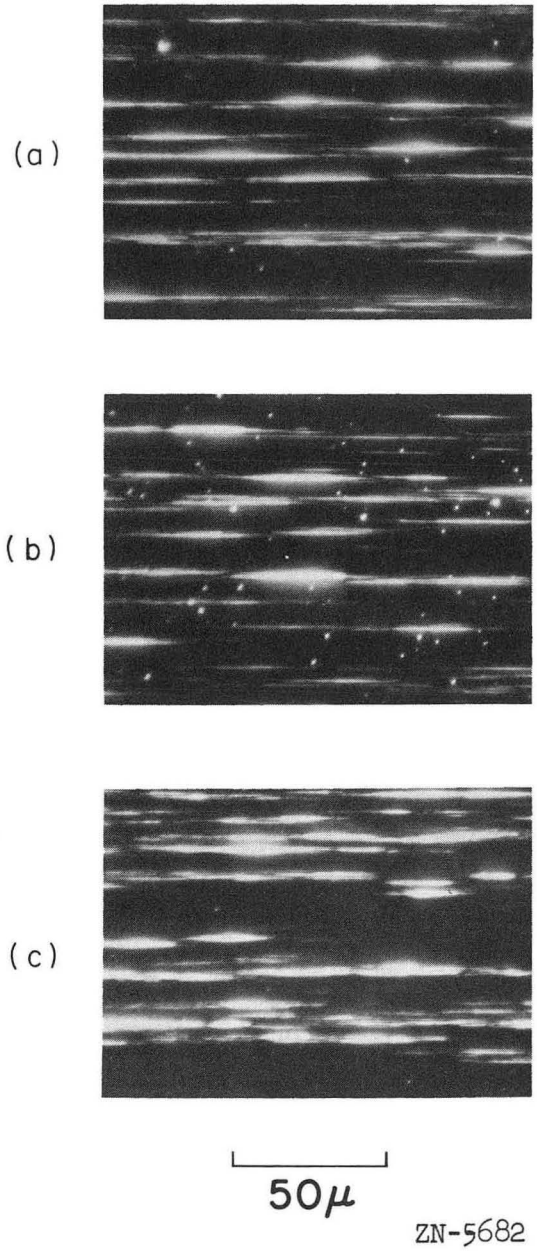


Fig. 12 Same as Fig. 11 for crystal prestrained by shock loading; (a) 16%; (b) 26%; and (c) 36% total strains.

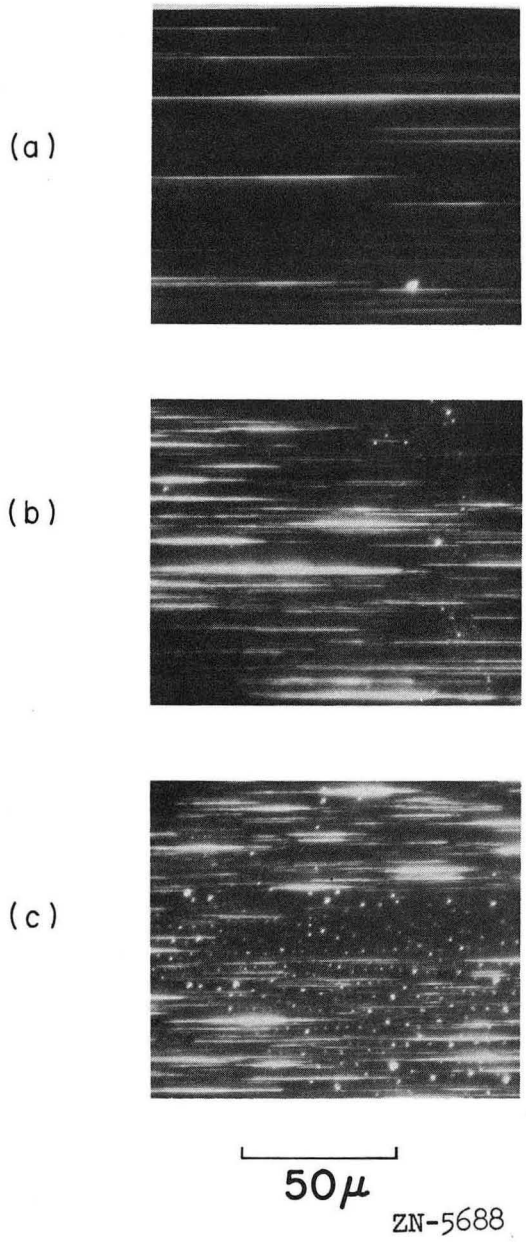


Fig. 13 Same as Fig. 11 for as-grown crystal; (a) 7%; (b) 15%; (c) 23%.