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DISLOCATION SUBSTRUCTURE IN STRAIN-CYCLED COPPER AS INFLUENCED BY TEMPERATURE

By

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ABSTRACT

Polycrystalline copper was cyclically deformed at various strain ranges and temperatures. The resulting dislocation substructure was examined by means of transmission electron microscopy, and a quantitative relationship between saturation flow stress and mean substructure size was found. A comparison of these results was made with other dislocation substructure and density measurements for copper and aluminum.

ACKNOWLEDGEMENT

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TABLE OF CONTENTS

LIST OF TABLES AND FIGURES

I. INTRODUCTION

In the past few years many investigations have been made of the relationship between flow stress and the associated microstructure in metals subjected to fatigue or tension tests. Since the development of X-ray and electron microscopy techniques, various relations between flow stress and dislocation density, or flow stress and "cell size" have been suggested.

Recently, a quantitative relationship between cyclic strain range, saturation stress, and cell size in polycrystalline copper deformed at room temperature has been reported [1]. For each strain range, a peak flow, or saturation stress is reached depending on the material, temperature, and cyclic frequency [2]. Altering the strain range allows the metal to harden or soften, depending on the initial state of the material, until another saturation stress is reached. A return to the original strain range causes the saturation stress to return to its original value. This behavior of the material occurs only if fracture does not interrupt the hardening or softening process.

If the strain range applied is great enough, the dislocation arrangements formed by plastic deformation of copper during cyclic straining consists of stable cells or subgrain boundaries. Similar subgrain formation has been reported for aluminum[3, 4], brass[5], silver[4], iron[6], and nickel[7]. At lower strain levels, dislocation loops and tangles appear to be the prevalent means of dislocation interaction[8, 9]. In either event, an increased tangling or more complex arrangement of dislocations is seen as the strain level increases.

At large cyclic strain ranges where cell formation occurs, the mean diameter of the cells can be determined. While the saturation stress increases with increasing strain range, the cell size decreases. Thus, the mean size of cells varies inversely with saturation stress when copper is cyclically deformed at room temperature.

Not only is the cell size inversely proportional to the saturation stress, but the formation of cells is a reversible process. If the saturation stress level of a cyclically strain hardened material is lowered by reducing the applied strain range, the cells will grow until a size associated with the lower stress level is reached. Therefore, cell size appears to be independent of the previous cyclic history and only related to the current stress level for pure polycrystalline copper. Similar conclusions have been drawn by Weissmann[10] from studies of zinc and silver single crystals fatigued in alternating bending at a constant strain amplitude[11]. By means of X-ray techniques, the cell size was found to grow as the strain was reduced.

Other quantitative investigations relating copper substructure to saturation stress have been performed in tension. Livingston[12] found a direct proportionality between (resolved shear stress)^2 and dislocation density for single crystal copper deformed in tension. Warrington[13] was able to find a relationship between saturation stress and (cell size)$^{-1/2}$ for polycrystalline copper deformed 20 per cent in tension at nine different temperatures from 78°K to 923°K. Johari and Thomas[14] were unable to show any quantitative relationship although some qualitative effects were noted, such as the cell size decrease with a decrease in temperature. Though all their specimens were deformed 20 per cent in tension, a variety of single and polycrystalline copper specimens were chosen. These were subjected to two different strain rates at three temperature levels.

Specimens, fatigued at lower temperatures, harden until a saturation stress level is reached that is higher than that found at room temperature for the same applied strain range[15]. This temperature dependence of the flow stress suggests that if the previous cell size observations are correct, cell sizes smaller than those at room temperature should be found for samples strain-cycled at the same strain ranges, but at lower temperatures. Low temperature electron microscopy investigations by Warrington and Johari and Thomas, as mentioned above, indicate this decrease in cell size with decreasing temperature.

II. OBJECT AND SCOPE

It was the purpose of this investigation to study the relationship between cell size and saturation stress for copper cyclically deformed at 78°K in order to supplement similar room temperature data[1]. Polycrystalline copper was subjected to controlled cyclic shear strain with the cyclic frequency held constant. Various strain ranges were selected to produce different saturation stress levels. The cell structure was investigated by transmission electron microscopy and mean cell sizes were then determined by statistical analysis.

III. EXPERIMENTAL PROCEDURE

Annealed OFHC copper (99.997 per cent copper) tubing was chosen with an average O.D. of 0.3739 in., wall thickness of 0.0303 in. and a gage length of 10 in. The tubes were subjected to cyclic torsion by imposing a constant shear strain range and allowing the stress to adjust to the conditions imposed. Specified shear strain ranges were 0.010 and 0.020 in./in. One sample was run at 0.020 in./in. and then two were subjected to an axial load of 11,000 psi as well as a strain range of 0.010 or 0.020 in./in. Samples were run at a constant cyclic frequency of 15 cpm which produced a negligible change in strain rate for the imposed strain ranges. Cycling was performed in a tube testing machine built in the Department of Theoretical and Applied Mechanics, University of Illinois[16]. The test temperature of 78°K was obtained by surrounding the specimen and specimen grips with a liquid nitrogen bath.

After cycling to failure, each specimen was halved lengthwise and sections of the tube away from the fracture zone and grips were selected for study. Foils for transmission electron microscopy were then prepared by a preliminary chemical polish and then by electropolishing in a solution of 33% nitric acid in methyl alcohol at -30°C. Foils were examined in a Siemens Elmiskop I electron microscope operated at 100 KV.

Cell size measurements were made from approximately 20 randomly selected electron micrographs (or about 1000 cells in all) for each specimen. From this data, the mean cell size and standard deviation was determined by statistical methods. The 95 per cent "confidence" limits on the mean were also calculated by standard procedures.

IV. DISCUSSION AND RESULTS

A. Saturation Stress - Cell Size Relationship

Cell size determination for the specimens at 78°K revealed a continued decrease in cell size with increasing saturation stress. A summary of these results is given in Table I along with the original room temperature data, Samples 1-5. Determination of the standard error of the mean diameters for the 95 per cent "confidence" limit indicated overlap for the limits of Samples 4 and 6. Since, by definition, the population mean (the mean of all the cell sizes for a particular saturation stress)lies within the "confidence" limits of the sample mean (calculated value); there is, in the case of Samples 4 and 6, some question of the significant difference in cell size.

Taking a look at the inverse relationship between cell size and saturation stress,

$$\tau = K/\ell \tag{1}$$

where τ = saturation stress, ℓ = cell size, K = a constant, Fig. 1 depicts saturation stress versus (cell size)$^{-1}$ for the low temperature as well as room temperature data. It can be seen that the low temperature data lies on a line parallel to, but above the original curve. Because the low temperature data lies on a line parallel to the original curve, it seems reasonable to expect that some temperature dependent factor should be used in order to compare the 78°K and 295°K results.

Thus, the temperature dependence of the rigidity modulus, G, was considered. From the data of Barrett and Sherby [17], the temperature dependence of the dynamic modulus of elasticity, E, of polycrystalline copper was taken. Then, using the general relationship between the three elastic constants

$$G = \frac{E}{2(1+\nu)} \tag{2}$$

and assuming $\nu = 0.33$, the 78°K value of the rigidity modulus was found to be 7.3×10^{6} psi, and the 295°K value was taken as 6.8×10^{6} psi. After dividing all the stress values by the appropriate G factor, the dimensionless, and now temperature independent factors, τ/G, were ready for comparison. In order to have a completely dimensionless plot, the Burgers vector, b, for copper, with the value of 1.80×10^{-8} cm., was divided by the various cell sizes.

Figure 2 represents the resulting curve of τ/G vs. b/ℓ. Now the low temperature data falls directly on the room temperature curve which shows that the inverse relationship between cell size and saturation stress does hold, within experimental error, for polycrystalline copper at 78°K as well as 295°K.

Since Fig. 2 is a plot of dimensionless ratios, similar data for other materials could fall on this curve if the saturation stress - cell size relationship is a general one. A search of available literature disclosed no extensive quantitative cell size data for fatigued specimens. Qualitatively, many investigators have indicated that some kind of relationship between flow stress and microstructure could exist in fatigue, and more definite statements have been made about metals in tension.

B. Dislocation Density Model

As was mentioned earlier, Livingston found that the dislocation density, N (number of dislocations/cm^2) was directly proportional to the (resolved shear stress)^2 for single crystal copper. It can be shown that this relationship, $\tau \alpha N^{1/2}$, is consistant with the inverse relationship, $\tau \alpha 1/\ell$ if careful definitions of terms are made.

Consider the two-dimensional model of a unit area containing a uniform distribution of square cells a length ℓ on a side. The number of cells per square centimeter would then be $1/\ell^2$, and the perimeter of each cell would be 2ℓ since each cell shares four sides with its neighbors. Thus, the total perimeter of the cells per square centimeter will be $2\ell \times 1/\ell^2 = 2/\ell$.

Now, looking at the dislocations perpendicular to this surface area, the number along a side of a cell will be n, or the number per unit length will be n/ℓ. Thus, the area density of dislocations, ρ, will be the number of dislocations per unit length x the total length of cell wall, or $n/\ell \times 2/\ell$, so

$$\rho = \frac{2n}{\ell^2} . \tag{3}$$

This same argument can be carried out for the three dimensional case with the model now a unit cube containing a uniform distribution of cubic cells a length ℓ on a side. Then, the total surface area per cell is $3\ell^2$ and the total surface area per cubic centimeter is $3/\ell$. Knowing that the number of dislocations per surface area is $2n/\ell^2$ from the previous model, the length of dislocation lines per surface area is $2n/\ell$. Then the total length of dislocation lines per cubic centimeter will be $2n/\ell \times 3/\ell$ or the volume density of dislocations, ρ', is

$$\rho' = \frac{6n}{\ell^2} = 3\rho . \tag{4}$$

The volume density is three times as large as the area density, which is a logical result of the given model.

In each of the above cases, the density is proportional to (cell size)$^{-2}$ or $\rho^{1/2} \alpha 1/\ell$. One of the expressions for flow stress[18] found in the literature is in terms of (density)$^{1/2}$,

$$\tau = \alpha G b N^{1/2} \tag{5}$$

where N = density/unit area and α = a constant dependent upon the dislocation arrangement. Another way of expressing flow stress[19, 20] is in terms of the average length of free dislocations, or the length of a Frank-Read source, $\bar{\ell}$;

$$\tau = \frac{G\,b}{h\,\bar{\ell}} \tag{6}$$

where h is a constant. It has been assumed that the length of free dislocation, $\bar{\ell}$, can also represent the mean diameter of a cell, since a dislocation must end at a node. From the conclusion of the previous model that $\rho^{1/2} \alpha\ 1/\ell$, it is apparent that these two ways of expressing the flow stress are equivalent.

If d is the spacing of dislocations in the walls of the cell, the number of dislocations, n, per boundary wall is ℓ/d and thus the density of dislocations in the boundaries becomes

$$\rho = \frac{2 \times 2\,\ell/d}{\ell^2} = \frac{4}{\ell d} \tag{7}$$

$$\rho' = \frac{6 \times 2\,\ell/d}{\ell^2} = \frac{12}{\ell d} \tag{8}$$

Since each cell shares its walls with its neighbors, the factor of two is again used. Considering a cell size of 10^{-4} cm. and a spacing of 10^{-5} cm, $\rho = 4 \times 10^9$ cm^{-2} and $\rho' = 1.2 \times 10^{10}$ cm^{-2}, which are reasonable values for dislocation densities in a work hardened material. Gay, et al.[21] have used this argument for determining dislocation densities from X-ray data. Now, dislocation density is proportional to (cell size)$^{-1}$, so that with this type of expression flow stress could be expressed in terms of (density)$^{1/2}$ or (cell size)$^{-1/2}$.

Three different ways of expressing flow stress have been stated from the dislocation models; one in terms of (density)$^{1/2}$, another in terms of (cell size)$^{-1/2}$, and another in terms of (cell size)$^{-1}$. All three of these statements can be found in the literature. The purpose of deriving these various expressions for flow stress has been to show that the particular form used depends upon the definition of dislocation density, that is, depends upon the model of dislocation arrangements that has been chosen.

C. Comparison of Results

Taking the relation $\rho = 4/\ell d$ and converting Livingston's density data into equivalent cell size form, it was found that the calculated values of ℓ were reasonable, but their associated flow stress values were far from consistent with the cyclic results of this investigator. Considering that Livingston used single crystals deformed in tension, and density measurements were made during rapid work hardening, it is probable that an equilibrium, low energy arrangement of dislocations had not yet formed so that these results could not be expected to follow the curve in Fig. 3 precisely.

Other work has been done by Hordon[22] measuring the dislocation density by an etch pit technique in single crystal copper during easy glide deformation in tension. Once again, the conversion of density to cell size appeared reasonable but the flow stress was so low that a comparison could not be made with the present work.

Warrington's data for polycrystalline copper covered a wide range of temperatures. Taking cell size measurements from 78°K to 695°K, the resulting points followed the original curve in Fig. 3 although the lower temperature points appear to be in closer agreement than the higher temperature ones. In a general way then this data follows the inverse relationship even though Warrington interprets his data as obeying the inverse square root relationship between cell size and flow stress.

Finally, Kelly studied polycrystalline aluminum annealed at two different temperatures and deformed in tension. By X-ray microbeam techniques, he was able to find a relationship between plastic strain and particle size at room temperature. The points for the two different annealing times are shown in Fig. 3. Once again, they fall along the original curve. This agreement in trend indicates that aluminum may possibly follow this inverse relationship between saturation stress and cell size.

In order to make a more precise comparison between different materials, it will be necessary to analyze statistically all cell size calculations so that the significance of the calculations can be known. Otherwise, it is difficult to predict how closely data from different sources should be in agreement. Also, uniform methods of determining cell size or density counts, either by transmission electron microscopy, X-ray techniques, or both, and uniform testing procedures would be useful for getting more precise results from various materials.

D. Dislocation Arrangements

Examples of the cell structures and cell wall dislocation arrangements studied in the electron microscope are given in Figs. 4-9. Figures 4 and 5 represent typical areas of samples deformed at 78°K. Although the cells in Fig. 4 appear to be elongated in a preferred direction, Fig. 5 shows a more random arrangement of cells. Figure 6 represents a typical region from a sample run at 295°K, and it has the same magnification as Figs. 4 and 5. The difference in cell size between the two temperatures appears very clearly in these micrographs. At higher magnifications, as in Figs. 7 and 8, the cell walls are more easily distinguished for the 78°K samples. A close comparison between these cell walls and those for a 295°K sample (Fig. 9), shows that the dislocation arrangements in the walls are similar. More tangling of dislocations and more segments traversing the cell can be seen at 78°K, but the basic dislocation arrangement in the walls does not appear to vary greatly from that seen at 295°K.

V. SUMMARY AND CONCLUSIONS

Polycrystalline copper was subjected to controlled cyclic shear strain at 78°K and a constant cyclic frequency in order to supplement similar results at 295°K. The relationship between cell size and saturation stress has been studied by means of transmission electron microscopy. Other cell size and density measurements for copper and aluminum were compared with the present results. It has been found that:

1. The inverse relationship found at 295°K between saturation stress and cell size holds for polycrystalline copper strain-cycled at 78°K, if the temperature dependence of the rigidity modulus is taken into account.
2. Other dislocation density and cell size measurements made in tension for aluminum and copper follow, in a general manner, the τ/G vs. b/ℓ curve found for polycrystalline copper in this report.
3. Three different expressions, depending upon the dislocation model chosen, can be derived for the flow stress in terms of dislocation density or cell size.
4. Dislocation arrangements in the cell walls appear to be similar at 78°K and 295°K even though the size of the cell changes.

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TABLE I

Compilation of Data

Sample	Saturation Stress psi	Strain Range in./in.	Cell Size microns
1	6400	0.005	1.37 ± .12
2	8000	0.010	1.14 ± .08
3	9200	0.015	1.07 ± .06
4	9900	0.020	0.88 ± .05
5	9900-8000	0.020 - 0.010	1.15 ± .06
6*	13500	0.020	0.81 ± .09
7*+	16000	0.010	0.65 ± .03
8*+	19000	0.020	0.55 ± .03

* Run at $78^{o}K$

\+ Applied axial load 11,000 psi

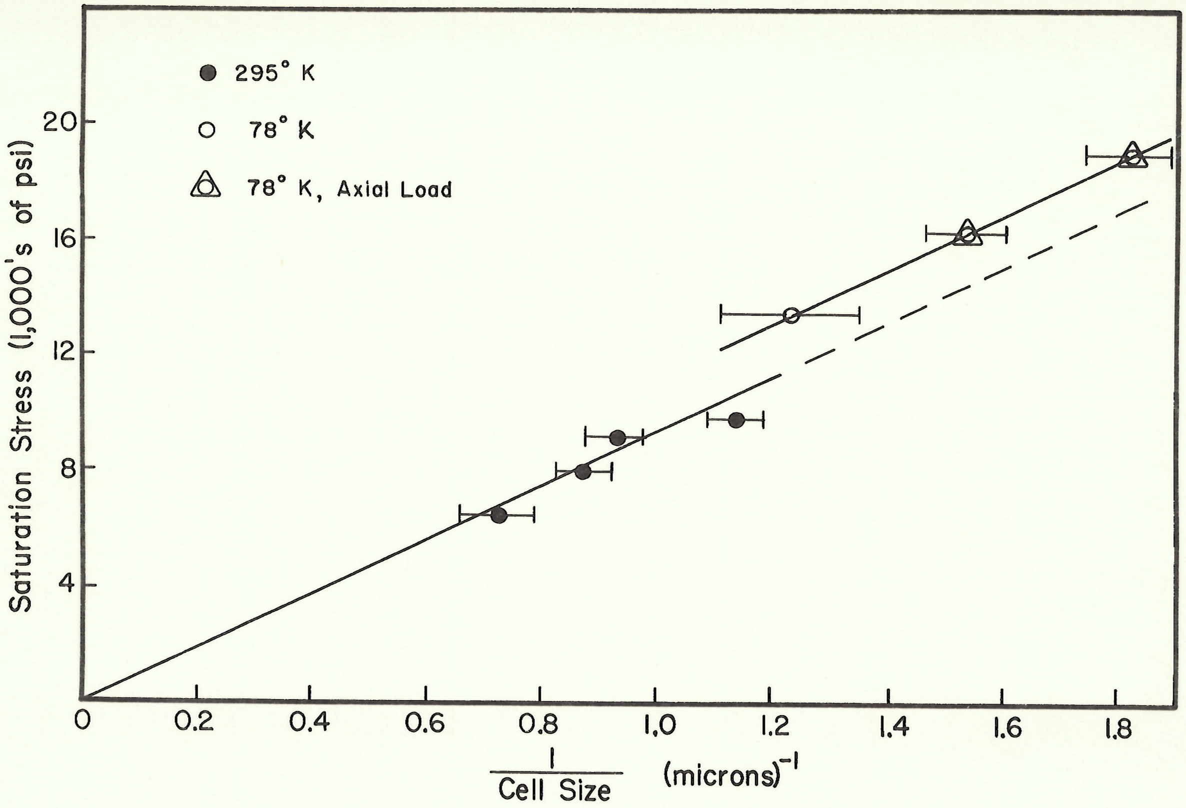

Fig. 1 Saturation Stress vs (Cell Size)$^{-1}$

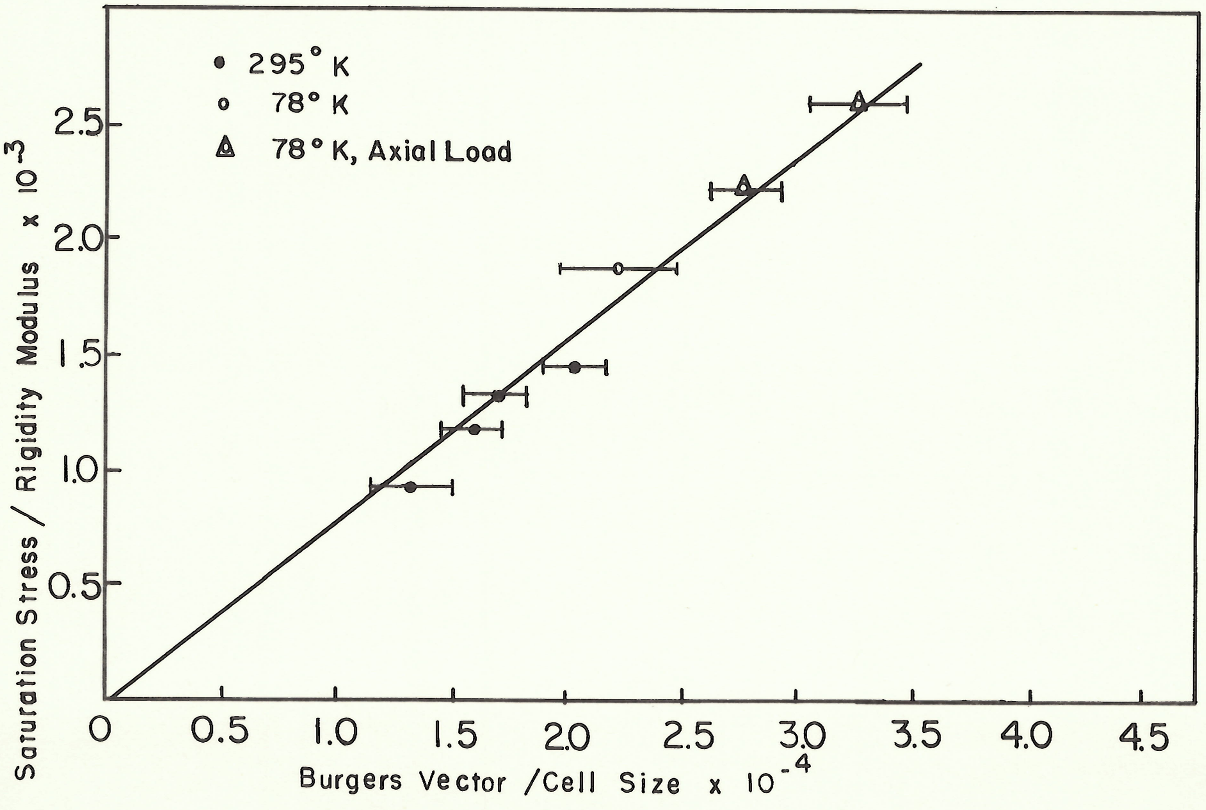

Fig. 2 Saturation Stress/Rigidity Modulus vs Burgers Vector/Cell Size.

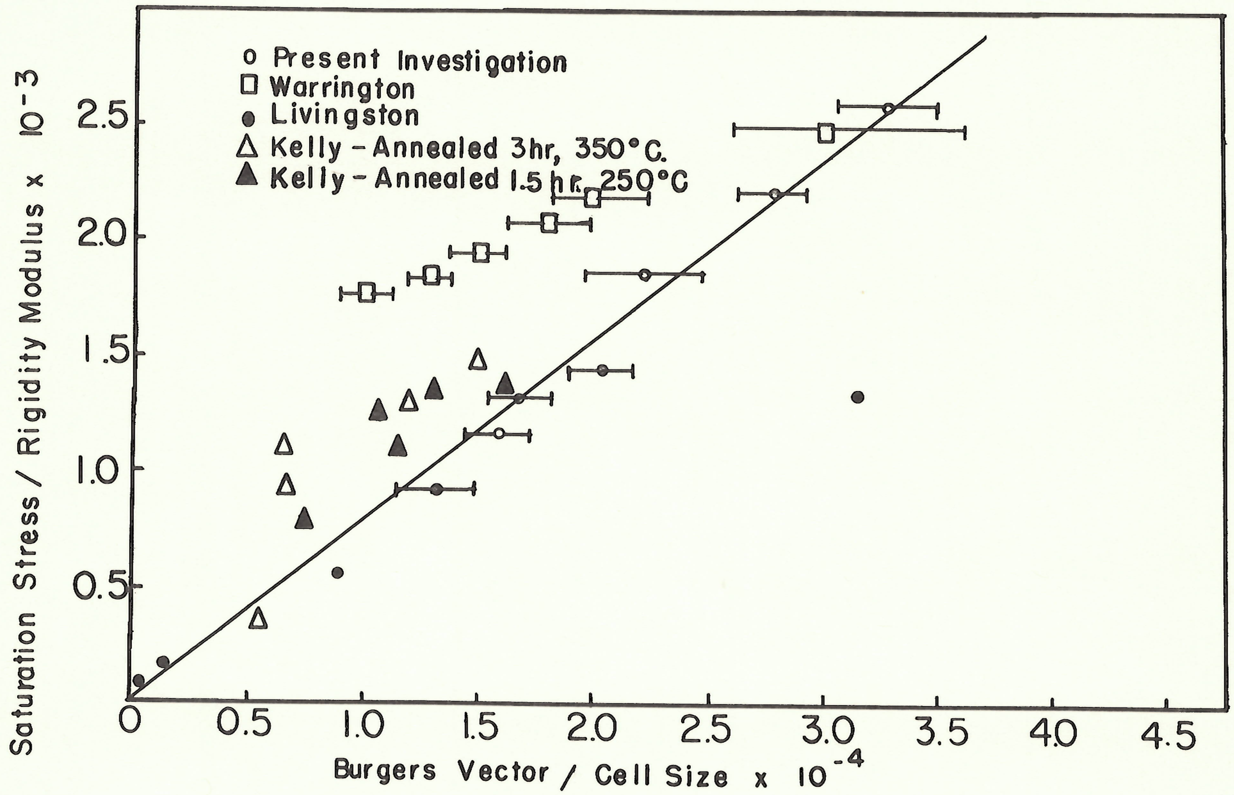

Fig.3 Variation of Cell Size With Saturation Stress for Copper and Aluminum.

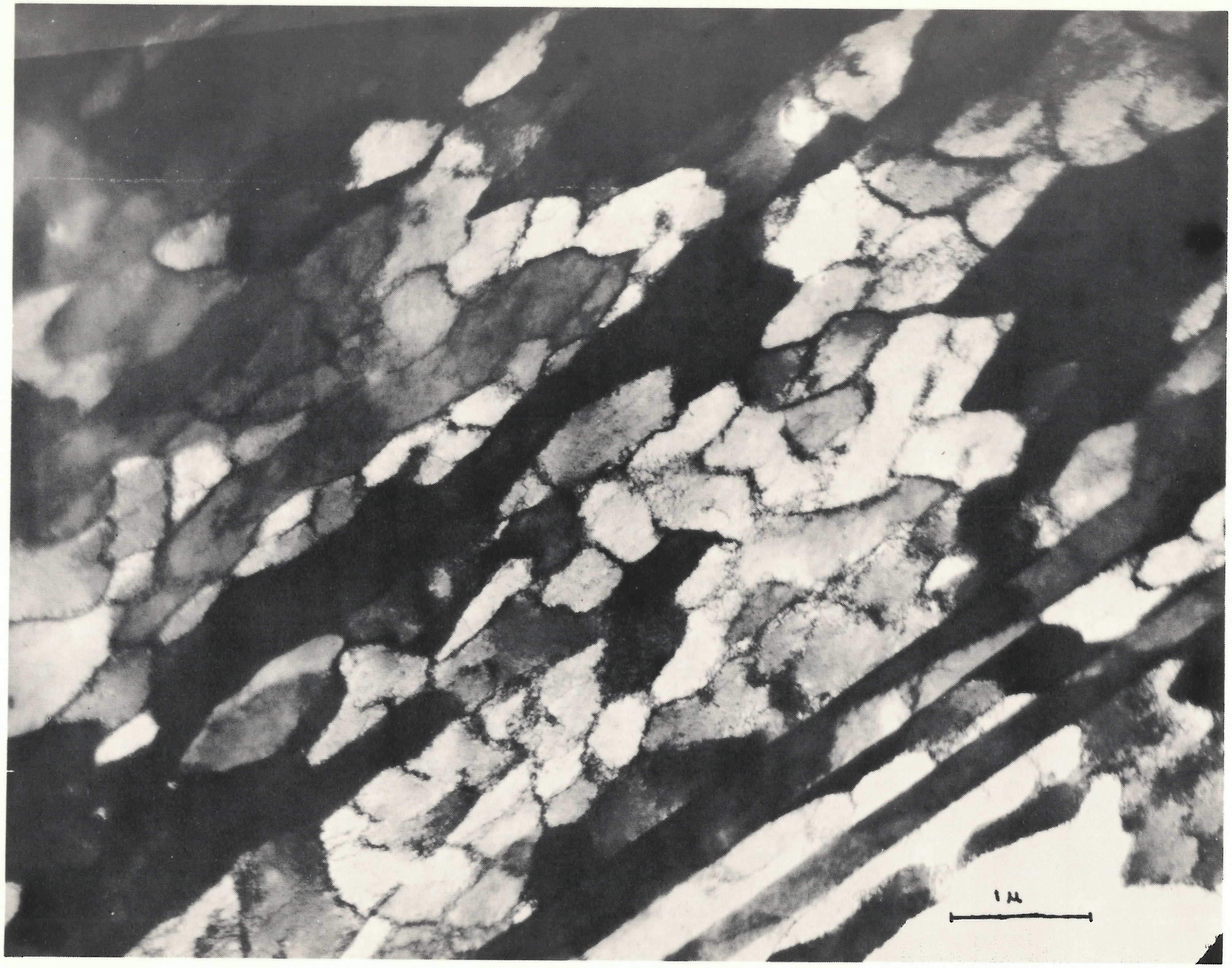

Fig. 4 Dislocation Cell Structure at a Saturation
Stress of 19,000 psi at 78°K. 21000x

Fig. 5 Dislocation Cell Structure at a Saturation
Stress of 13,500 psi at 78K. 21000x

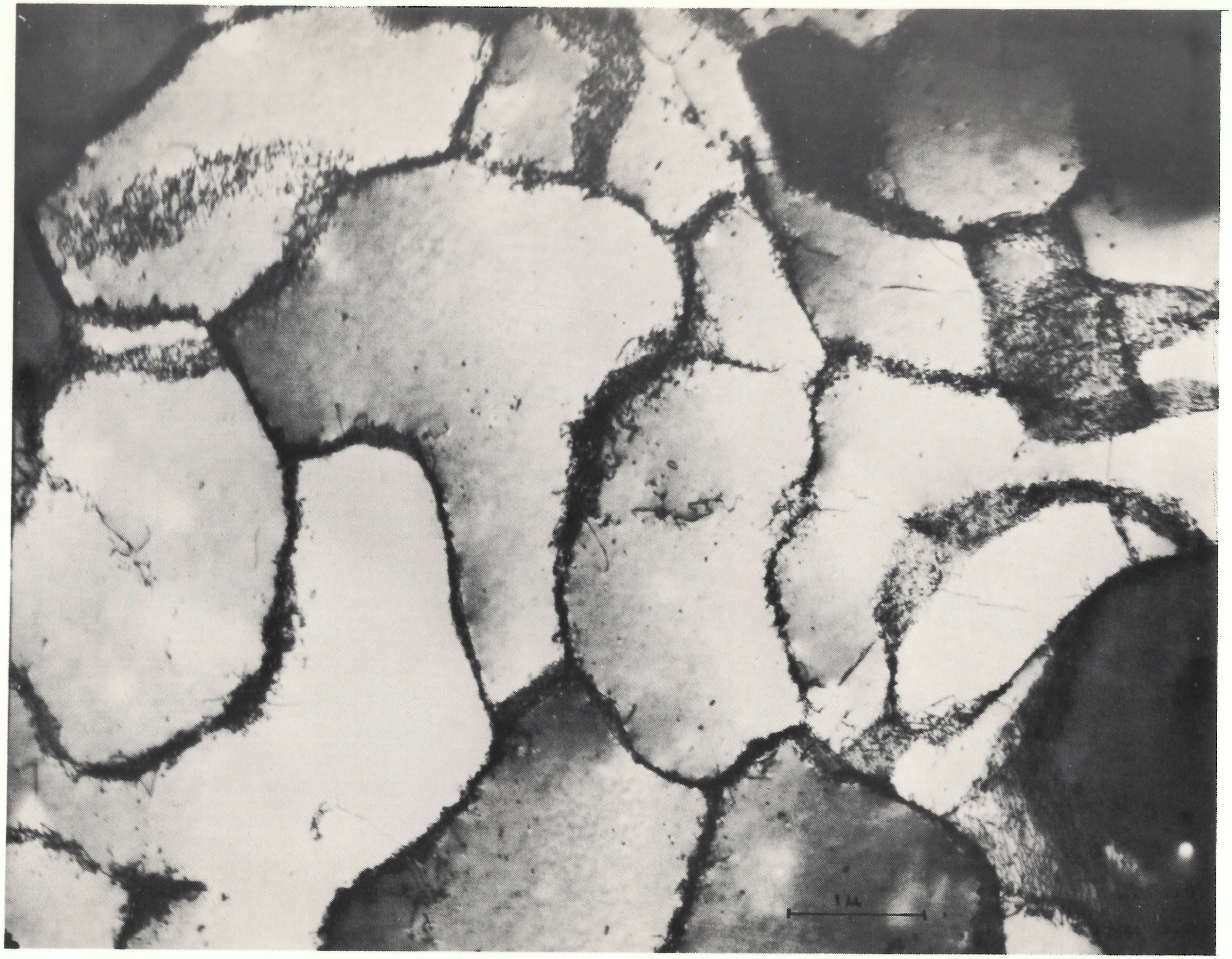

Fig. 6 Dislocation Cell Structure after Strain-Cycling at a Saturation Stress of 9900 psi for 1500 Cycles and then 8000 psi at 295°K. 21000x

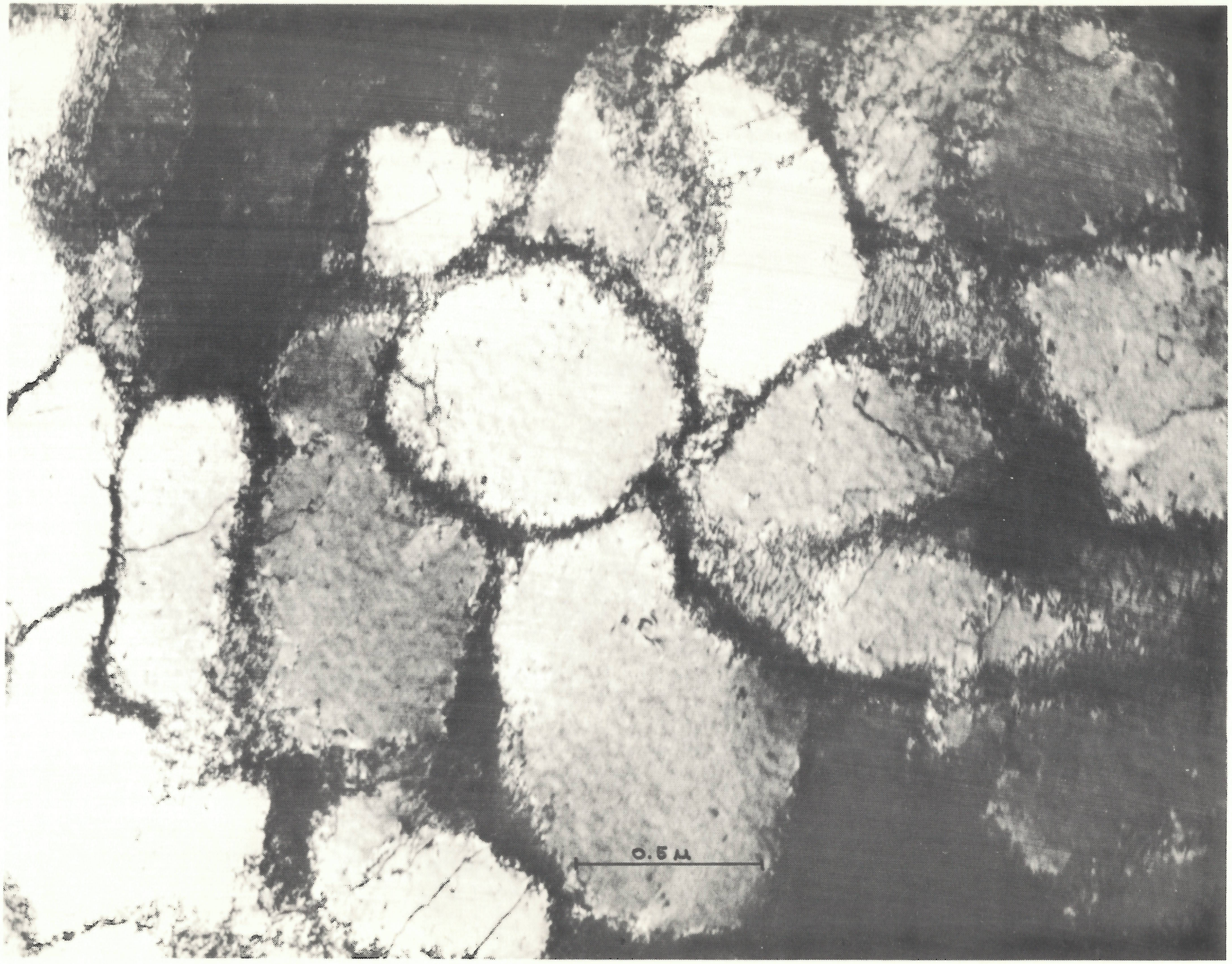

Fig. 7 Dislocation Cell Structure at a Saturation Stress of 16,000 psi at 78°K. 53000x

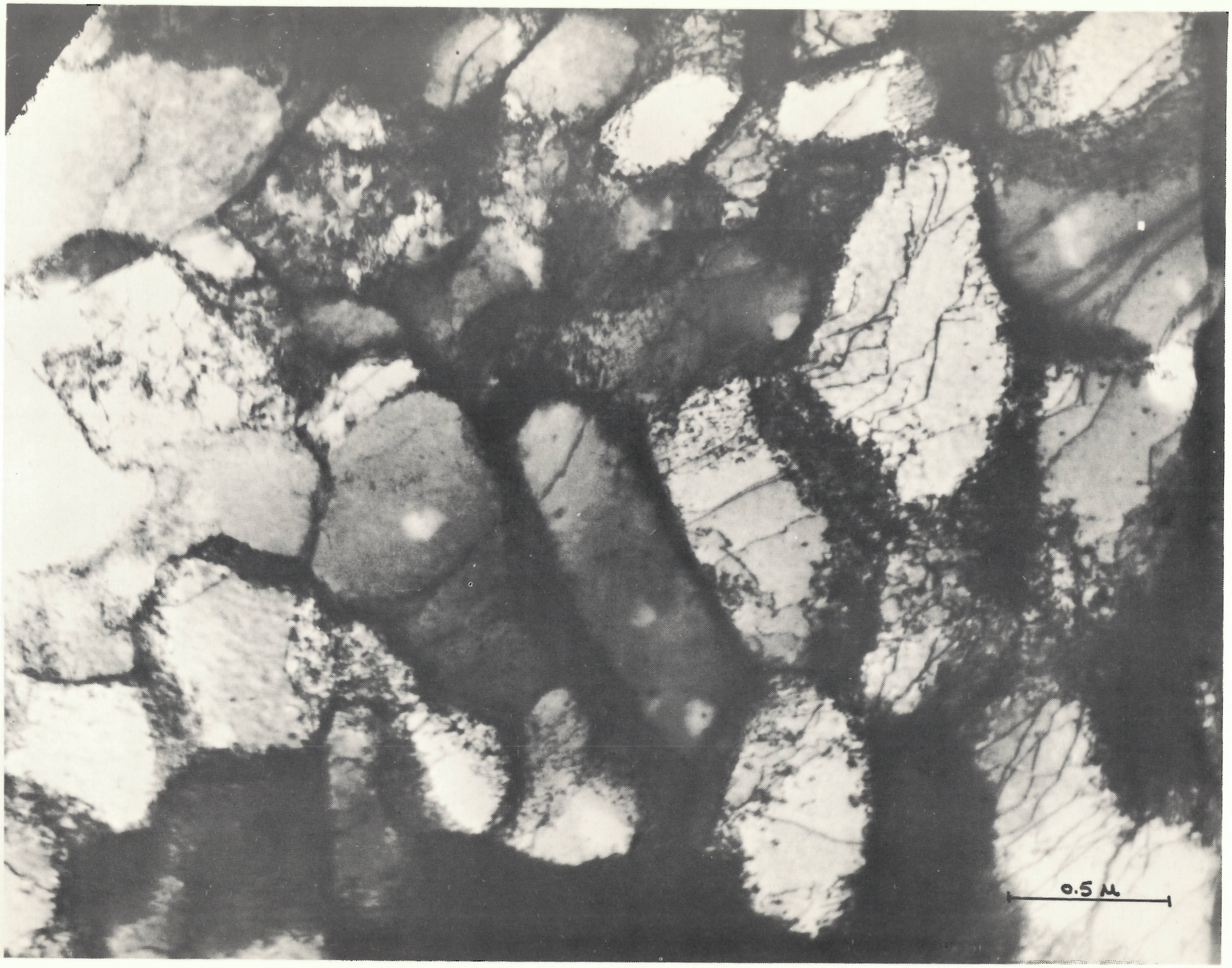

Fig. 8 Dislocation Cell Structure at a Saturation Stress of 16,000psi at 78°K. 48000x

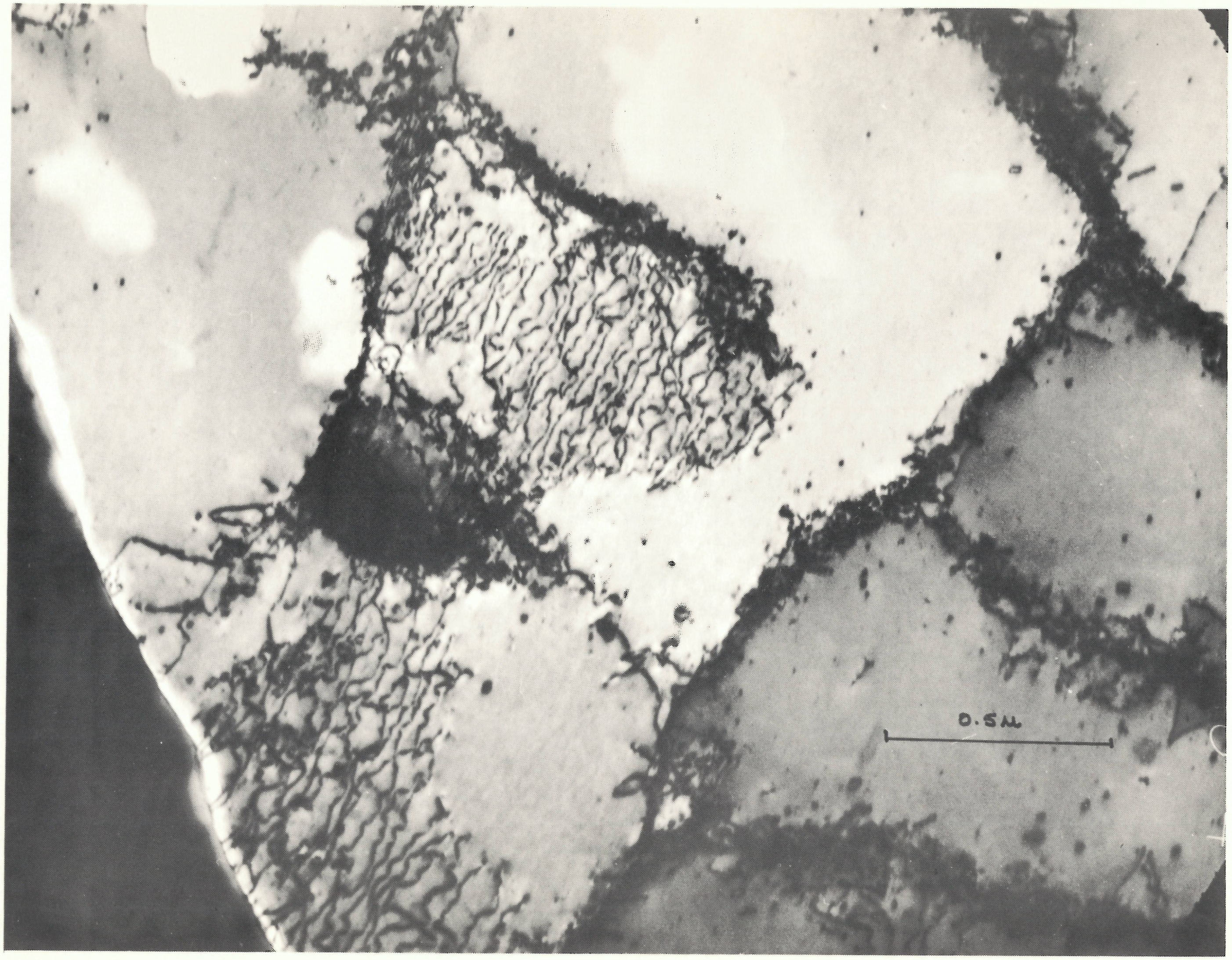

Fig. 9 Dislocation Arrangement in Cell Wall at a
Saturation Stress of 8000psi at 295°K. 67,500x