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THESIS

TRANSMISSION ELECTRON MICROSCOPY STUDY OF
SUBGRAIN STRENGTHENING OF CARTRIDGE BRASS

by

Thomas Benton Fulton

December 1976

Thesis Advisor:

Jeff Perkins

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TRANSMISSION ELECTRON MICROSCOPY STUDY OF SUBGRAIN
STRENGTHENING OF CARTRIDGE BRASS

by

Thomas Benton Fulton
Lieutenant, United States Navy
B. S. , United States Naval Academy, 1971

Submitted in partial fulfillment of the
requirements for the degree of

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ABSTRACT

The development of substructure in cartridge brass, subjected to cold rolling followed by warm annealing, is characterized as a function of annealing temperature and true strain. Substructure develops and becomes refined as annealing temperature is increased to the point of recrystallization. Dislocation cell structure is also refined as true strain is increased. The variation of hardness with annealing temperature correlates well with substructure development and refinement.

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I. INTRODUCTION

The effect of the development of substructure upon mechanical properties of certain metals and alloys has been reported by numerous investigators [1-5]. It is the aim of this thesis to show correlations which may exist between hardness and subgrain cell structure for cartridge brass (70 Cu-30 Zn). While mechanisms of subgrain formation are still under investigation, it has been determined that stable substructure may be formed by various thermo-mechanical processes, such as warm rolling, creep, and cold rolling followed by warm annealing. The latter process has been used in this investigation.

The particular thermo-mechanical process used determines the nature of the substructural boundaries. For example, as higher temperatures are employed in rolling, for a given subgrain size, the better the subgrain boundary acts as a barrier to plastic flow. Chen and Lytton [6] found that subgrains formed by the process of cold rolling followed by warm annealing, as used in the present work, are less resistant to plastic flow at elevated temperatures than those obtained by warm working.

The effect of subgrain structure on strength is not trivial. Through subgrain refinement, Tilman and Neumeier [7] were able to increase the tensile strength of lead alloys by approximately 21 percent without serious loss of ductility. Young and Sherby [4] achieved 100 percent increase in the low temperature yield strength of AISI 316 stainless steel by reducing the subgrain size from 1 micron to 0.2 micron. These investigators also concluded that

materials with subgrains of 0.4 micron or less appear to be stronger than the same material containing grains of the same size.

Early investigation by Hall and Petch [8] found that flow strength in metals may be related to the grain size. They modeled grain boundary strengthening by assuming that, in the undeformed state, a polycrystalline metal has few dislocations between grain boundaries. As the metal is deformed, generated dislocations glide on well-defined slip planes. These dislocations pile up at grain boundaries and cause a stress concentration which is transmitted through the boundaries to adjacent grains. They proposed that the strengthening effect may be formulated as

$$\sigma = \sigma_0 + k(\bar{d})^m$$

where σ is the strength at a fixed plastic strain, σ_0 is a form of "friction stress", k is a material constant, m is a constant, and \bar{d} is the grain size. They proposed a value of m equal to $-1/2$ which would indicate that the smaller the grain size, the larger the yield strength. In terms of the model, the smaller grain size affords a shorter plane on which dislocations may pile up, and the resulting smaller stress concentration allows a higher yield strength. The stress concentration resulting from the pile up is smaller for a finer grain size and requires a higher applied stress to propagate flow in the structure.

Metallurgists today recognize the Hall-Petch relation to be an empirical relation which is valuable in studying strengthening mechanisms. Many investigators [1,4,5,8] have examined the applicability of this relation to subgrain strengthening and have found that, for ambient temperatures, the relation holds when the subgrain size Δ is substituted for \bar{d} . From these studies, it appears that the constant k

may depend on the nature of the subgrain cell wall and that a better value for m is -1.0 [1,4,5,8,9]. Fujita and Tabata [5] have found that the Hall-Petch relation depends on both the specimen and deformation conditions and have suggested that the usual grain size parameter might be replaced by a more general measure, such as a mean free path for dislocation motion within the specimen that would take into consideration cell obstacles to dislocation motion rather than only high angle grain boundaries.

The purpose of this study is to characterize the strengthening effect of subgrain development in cartridge brass resulting from cold rolling followed by warm annealing. For this investigation, hardness was the mechanical property chosen to characterize the effect of substructure refinement. For the remainder of this report, the term "dislocation cell" will be used to denote the diffused boundary substructure developed, rather than "subgrain", which might imply a more distinct boundary. Alloys with low stacking fault energies, such as brass, tend to form these diffuse boundaries while metals with high stacking fault energies, such as aluminum, tend to form very distinct, well-defined subgrains [10].

II. EXPERIMENTAL PROCEDURES

The samples used in the research were cut from 0.25 inch thick stock of cartridge brass into pieces 0.7 inch by 1.0 inch. The material was received in a half-hard condition, so the samples were initially annealed at 900°F for one hour. The pieces were then cold rolled on a rolling mill, figure 1, to induce true strains ranging from 10 to 90 percent at increments of 10 percent. Specimens of each degree of strain were then annealed at a constant temperature for one hour. Ten annealing temperatures were used, ranging from room temperature to 650°F in increments of approximately 50°F. Mechanical testing was performed on a Rockwell Hardness Tester using the 1/16 inch ball diameter and 100 kilogram weights, resulting in a Rockwell B hardness. Ten readings were taken for each sample, with the highest and lowest disregarded, and the remaining averaged.

When hardness versus annealing temperature was plotted for a given strain, hardness initially increased, followed by recovery at temperatures where recrystallization might be anticipated. Samples were chosen along the 60 percent true strain curve for investigation of their substructure before, at, and after peak hardness was attained. Thinned sections were prepared by first spark cutting on a S.M.D. Servomet Spark Machine, figure 2, using a 0.028 inch wire and a cutting range of 4, producing slices perpendicular to the direction of rolling with a thickness of 0.04 ± 0.005 inch. These sections were sanded with 400 grit paper to 0.03 inch and electrochemically thinned using a 10 percent solution of potassium cyanide at 8 volts AC. The thinned sections were rinsed in distilled water and alcohol. All foils were

examined using a tilting stage in a JEM-7 transmission electron microscope at 100 KV.

III. RESULTS

In figure 4, there is seen a distinct increase in hardness (for a given initial strain) as a function of annealing temperature up to a point. The hardness then falls off rapidly with increasing temperature, as the structure starts to recrystallize. The 60 percent strain series was chosen for detailed substructure investigation because it appeared representative of all samples.

As seen in figure 5, cold rolling with very low annealing temperature does not develop a distinct cell structure. There are bands of dislocation tangles, but no significant, distinct cell structure to act as a barrier to dislocation motion can be identified. There is an area of discontinuity of bands of dislocation tangles at A which is probably the result of a low angle boundary.

Figure 6 shows that a substructure begins to develop for the specimen annealed at 210°F. The cell structure boundary is not distinct, and not easy to quantify, but an ordering of the dislocation tangles is apparent. The hardness of this sample from the 60 percent strain curve from figure 4 indicates that this developing substructure has increased the hardness of the material. Figure 7 shows that annealing at 245°F for one hour allows the dislocations to form much smaller, though still indistinct, cells. The increase in hardness becomes more significant, however, as substructure refinement increases.

Increasing the annealing temperature to 430°F results in the substructure shown in figure 8. The cell structure is more obvious and quantifiable, having characteristic subgrain features with a cell size of approximately 0.2 micron. From figure 4, it is evident that there has been a sharp rise in the hardness of the specimen with the development of the cell structures. Figure 9 indicates that the cell size may reach saturation at some intermediate value of annealing temperature, as there is no noticeable reduction in size of the dislocation cells by increasing the annealing temperature to 525°F. Figure 10 gives a higher magnification view of the cell structure at this peak hardness. While the exact nature of the boundaries cannot be discerned, diffused dislocation tangles are apparent. Reference to figure 4 indicates that the two samples, having approximately the same substructure, also have approximately the same hardness. Figure 11 is indicative of the structure of the material once it starts to recrystallize. The cells are gone, and there appear long flowing dislocation lines with little interruption. As expected, figure 4 shows that this sample not only has not increased in hardness (since it has developed no substructure) but that (due to recrystallization) the material has actually softened from the "as rolled" condition.

Samples of peak hardness at each strain chosen for observation have well-defined cell structures. Figure 12 shows that the cell structure developed by the 20 percent strain during rolling is large and irregularly shaped. The central portions of the cell indicate a low dislocation density while the cell walls are made up of dense tangles. The sequence of figures 12-15 shows the refinement of substructure as a function of increasing strain. This follows because, as deformation increases, dislocation density increases. The smaller cell structure would provide

more boundary surface to take in the generated dislocations. The increase in peak hardness with increasing strain is shown in figure 4. A plot of the log of hardness versus the negative log of cell size can be fitted by linear regression with a line of slope -0.639 . Although this value approaches the value of -1 for m suggested by other investigators, much scatter is inherent in these values, which arises from the determination of the dislocation cell size.

IV. DISCUSSION

Challenger and Moteff, working with AISI 316 stainless steel, have made observations concerning the formation of dislocation cell structure [1]. Their work suggests that subgrains are formed by the intersection and tangling of dislocations lying on intersecting (111) slip planes in the face centered cubic structure. These dislocation tangles would act as barriers to subsequent dislocation glide on the (111) plane and will collect dislocation debris which in turn promotes the formation of dislocation cells whose boundaries lie on the (111) planes. The development of these stable dislocation tangles formed by intersecting slip planes is the Lomer-Cottrell effect.

In other work [11,12], from direct "in situ" observations of aluminum deformed in a 500 kev TEM, Fujita has discovered that these stable tangles do not form effective barriers to dislocation motion. He observed that active dislocations readily climb and slip through these tangles under small applied stresses. In the same work, Fujita observed that after considerable deformation small dislocation loops are formed which are very stable and act to pin the active dislocations. Heavy tangling of dislocations occurs around these loops when the loop density is high. During recovery, these loops anneal out and the dislocations are unpinned.

The present work did not investigate the formation process. In either case mentioned above, the driving force for rearrangement of dislocations into a cellular structure is the reduction of strain energy in the system. In the

study with AISI 316 stainless steel [1], Challenger and Moteff expressed a relationship which showed that a reduction of the average distance between dislocations as they rearrange themselves during annealing results in a corresponding decrease in the strain energy. This reduction in strain energy thus would drive the rearrangement, provided that the dislocations had sufficient mobility. This also presumes the existence of a critical dislocation density beyond which rearrangement will occur. In the present study, it is assumed that the critical dislocation density has been achieved at a small true strain and that the increased dislocation mobility afforded as temperature increases allows the mechanisms of cross slip and climb to form the dislocation cells. The expected temperature dependence of this process is confirmed in this investigation by the observation that the higher the annealing temperature, the better developed are the dislocation cell structures. Vesely [13] has found that dislocation walls formed in deformation prior to forming cells are not continuous throughout the cross section, but that they terminate in the material. He states that the end of a wall represents a discontinuity and is the source of internal stress. Such discontinuities are seen in the present work, and their stress field could result in a barrier to primary dislocation motion. Increased annealing temperatures could also work to relieve these stresses and increase dislocation mobility.

Fujita and Tabata [5] have worked out an interesting relationship between the dislocation cell size and the initial grain size which indicates that the cell size decreases rapidly when the initial grain size becomes smaller than a certain value which is a property of the material. This relation is derived from their work with aluminum and is based on the theory that when the strain increases, the number of active slip systems is increased

due to interaction among the grains. This theory should be valid for all face centered cubic metals.

Most theories on subgrain strengthening are modeled to make the data fit the Hall-Petch relationship. Langford and Cohen [8] investigated the strengthening of iron by subgrains developed by wire drawing. These subgrains were highly elongated and ribbon-like so that their strengthening data may not be applicable to the present work; however, some of their conclusions and modeling are relevant to the strain hardening process. In this model, cell size strengthening is based on the assumption that dislocation sources in the cell walls are readily activated at low applied stresses under the influence of high stress concentrations. The observed strain hardening would result from the stress required to expand each dislocation loop across the glide plane of the cell until it reaches the boundary of the glide plane. The work of deformation per unit cell would be equal to the product of the flow stress and the strain per passage of dislocation. This work is equal to the energy per unit volume expended in forcing the expanding dislocation loop against the friction stress (σ_f) on the glide plane, plus the energy per unit volume required to generate the total length of the dislocation line. The result of their analysis is an equation of the form

$$\sigma = \sigma_0 + k(\lambda)^{-1}$$

From experimental observations, Fujita and Tabata [5] have found that for low strains and no developed substructure in aluminum, the Hall-Petch relation,

$$\sigma = \sigma_0 + k(\bar{d})^{-1/2}$$

is valid, where \bar{d} is the grain size. This would indicate that a kind of dislocation pile-up (upon which the

Hall-Petch relation is based) is formed against the grain boundary in order to transmit the deformation to the next grain. With large angles between grains, it can be assumed that grain boundaries are the major obstacles to dislocation motion. Once the substructure has developed, however, active dislocation motion is retarded significantly by dislocation tangles. Active dislocations must move through the tangles by cross slip and climb. Thus, perhaps the cell structure is dominant in determining dislocation motion by the density and size of the prismatic dislocation loops in the cell walls. The fact that these dislocation loops tend to anneal out [5,11,12,14] may account for the fact that subgrains formed by cold working and annealing are less resistant to plastic flow at high temperatures, as discovered by Chen [6]. It may also help explain why the strengthening from this method was less dramatic than that obtained by the warm rolling done by Sherby [4] and Tilman and Neumeier [7]. Figure 12 gives a good representation of the expected substructure after annealing at a high temperature. The dislocation loops have been annealed out, and so the dislocation tangles become long and flowing in the recovery stages. Thus, it may be deduced from the present work that the nature of the boundary is determined by the conditions under which it is formed.

For intermediate areas where the substructure is developing, the mean free path of dislocations may be a combination of grain size and dislocation tangles. The numerous other possible obstacles to dislocation motion, such as precipitates, solute atoms, and point defects, could lend credence to the idea that rather than having a strength relation based solely on grain or subgrain size, it would be better to use a Hall-Petch type relation with a mean free path of dislocations and recognize where grain boundary and subgrain boundary simplifications would apply.

V. SUMMARY AND CONCLUSIONS

A transmission electron microscopy investigation of the correlation between increasing hardness and the developed substructure of cartridge brass (70 Cu-30 Zn) has been made. The brass was thermo-mechanically treated by cold rolling followed by warm annealing. This process resulted in increased hardness with increasing temperature, for a given true strain, up to the onset of recrystallization. Micrographs of the substructure show that the development and refinement of the dislocation cell structure correlates well with the observed increase in hardness in figure 4.

Fujita [11,12] has theorized that dislocation cell structures are formed by dislocation loops formed during deformation. From "in situ" observations of deformation in a 500 KEV TEM, he has found that these dislocation loops anneal out at elevated temperatures and the dislocations are unpinned, allowing them to move freely. This would help explain why subgrains formed by cold working followed by warm annealing are less resistant to plastic flow at elevated temperatures as observed by Chen [6]. The present study indicates that the cell structure does not form at higher annealing temperatures, as shown in figure 11.

The series of micrographs showing substructure as a function of annealing temperature (figures 5-10) shows a high dislocation density formed during cold work. For annealing temperatures greater than 150°F, most of the dislocations are arranged in more or less complicated cellular walls, and additional dislocations exist as a less dense network within these walls. These features are best

defined in figure 6. This structure is typical of most annealed metals, but the precise distribution depends strongly on the metal, its purity, and the history of the deformation [15]. Also, throughout this series, dislocation density between the cell walls appears to decrease as the dislocations migrate to the walls.

The samples chosen to depict the substructure as a function of true strain were, in each case, taken at the peak of the hardness versus annealing temperature curve and are assumed to represent the most refined substructure. The dislocation cells in each case consist of diffused cell walls with central areas of low dislocation density. As shown in figure 16, the hardness increases with decreasing cell diameter.

While it is evident that cold rolling followed by warm annealing is a viable mechanism for subgrain strengthening of cartridge brass, the boundaries formed are not as effective a barrier to dislocation motion as those of other materials formed by alternative methods [4,7]. Further investigation is needed to see if more effective strengthening may be realized by alternate thermo-mechanical treatment.

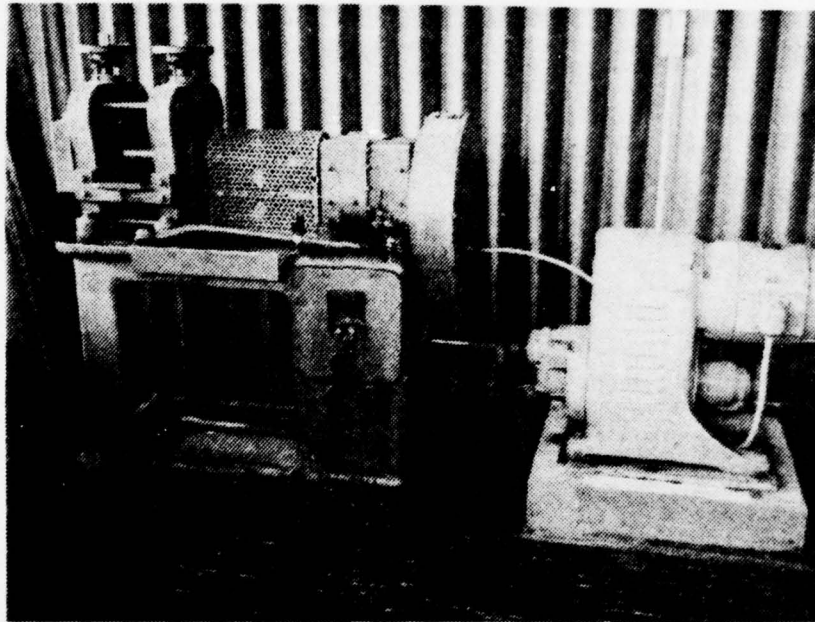


Figure 1 - ROLLING MILL.

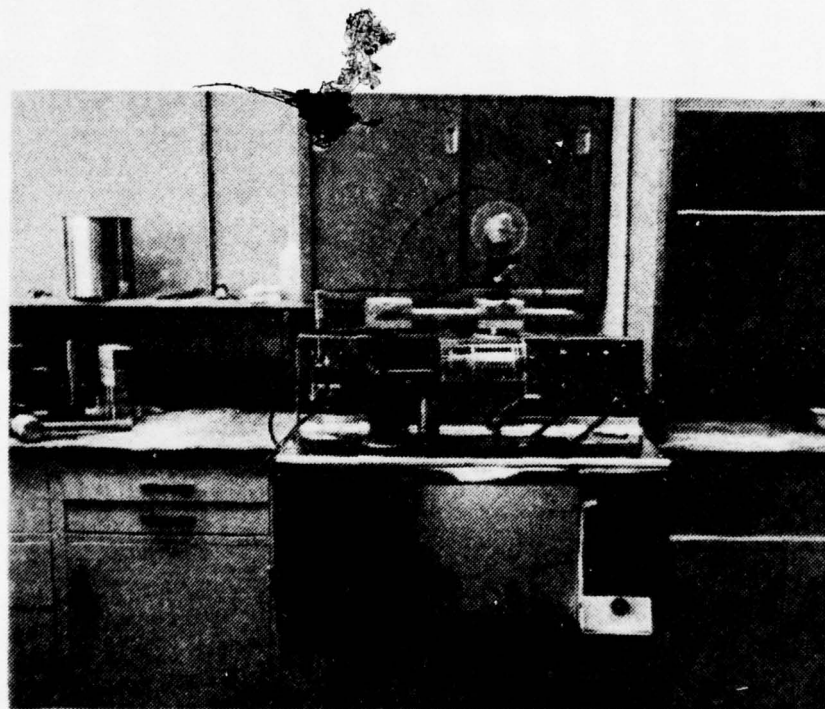


Figure 2 - SERVO S. M. D. SPARK SLICER.

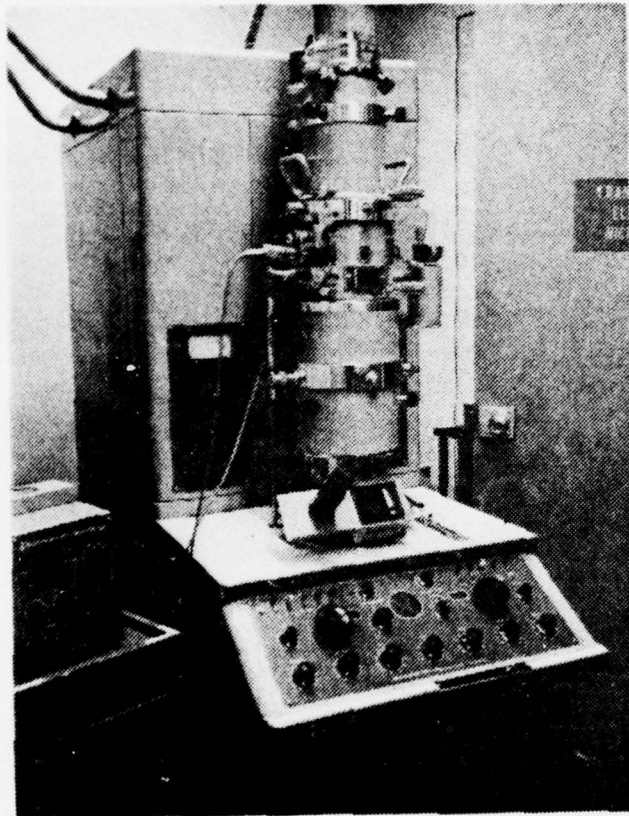


Figure 3 - JEM-7 TRANSMISSION ELECTRON MICROSCOPE.

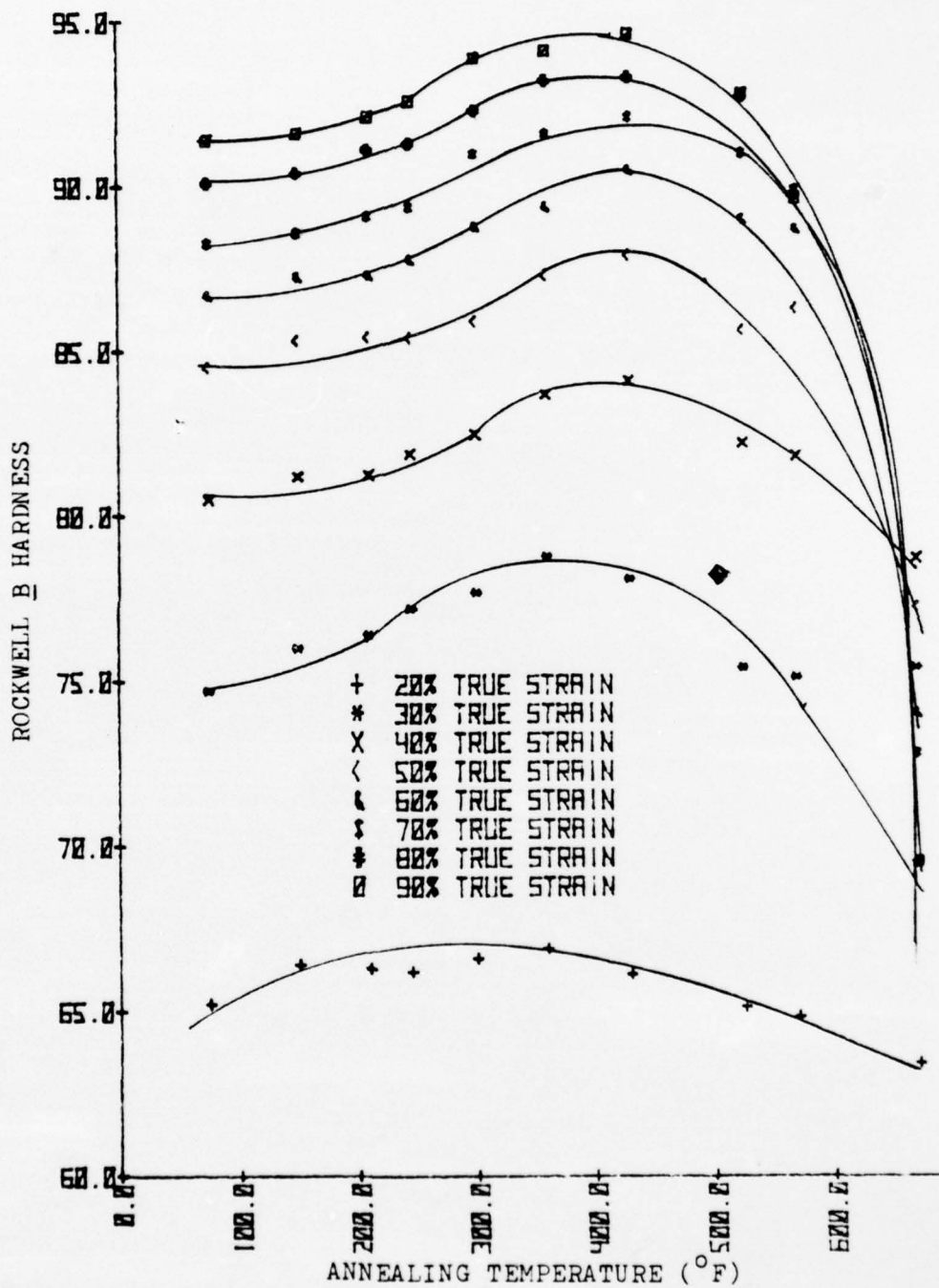


Figure 4 - HARDNESS VERSUS ANNEALING TEMPERATURE FOR CARTRIDGE BRASS AT VARIOUS TRUE STRAINS.

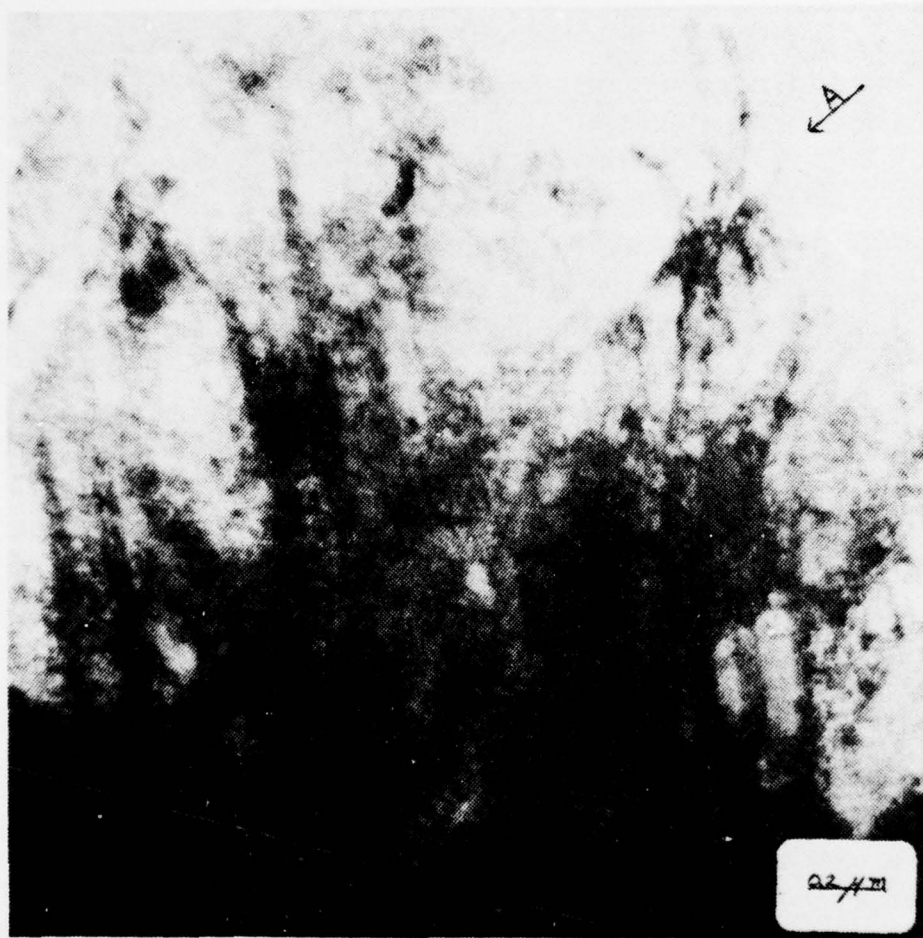


Figure 5 - CARTRIDGE BRASS STRAINED TO 60 PERCENT AND ANNEALED AT 150°F FOR 1 HOUR; BF IMAGE 50,000X.

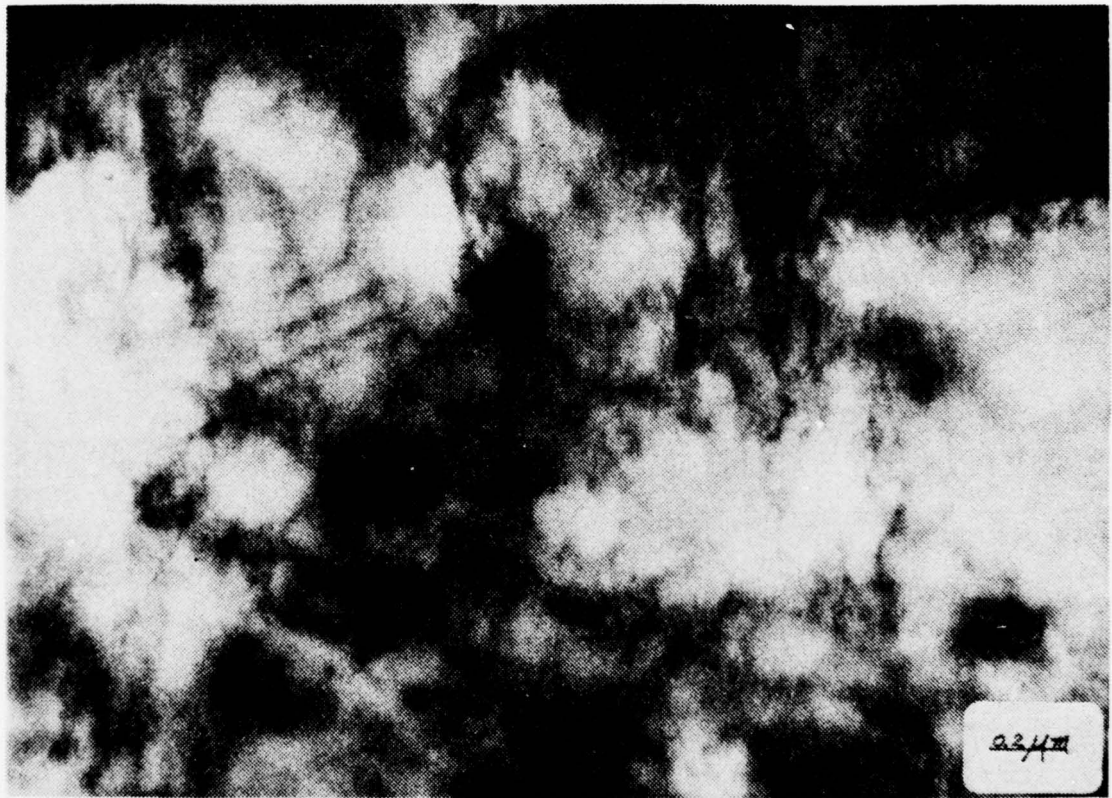


Figure 6 - CARTRIDGE BRASS STRAINED TO 60 PERCENT AND ANNEALED AT 210°F FOR 1 HOUR; BF IMAGE 50,000X.

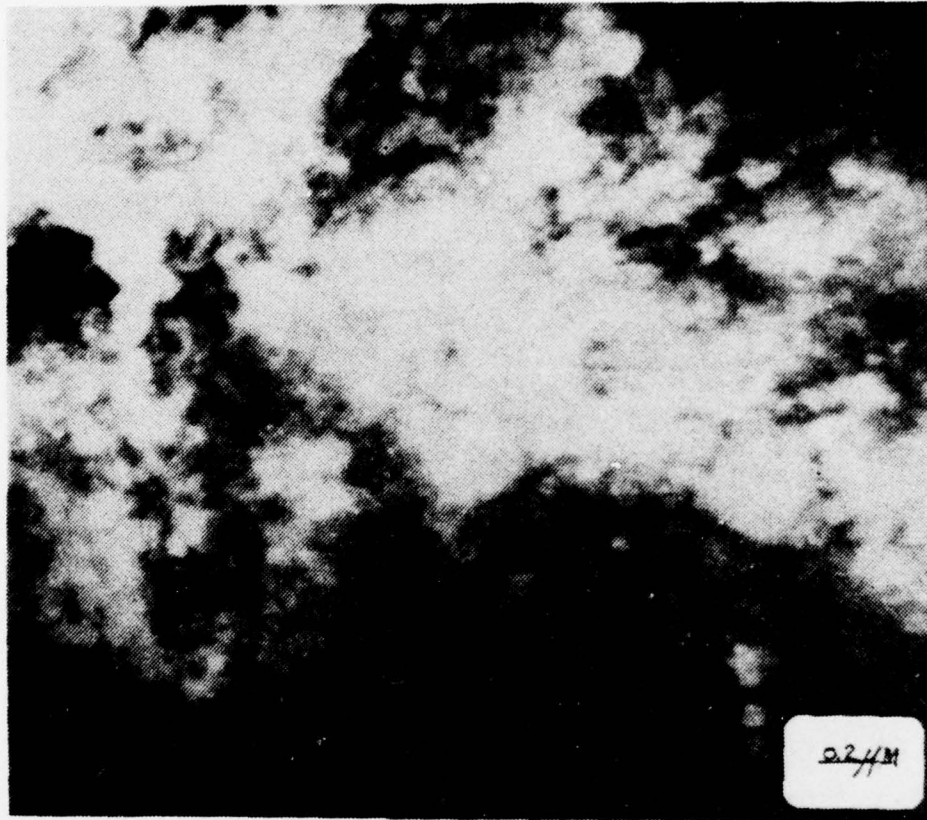


Figure 7 - CARTRIDGE BRASS STRAINED TO 60 PERCENT AND ANNEALED AT 245°F FOR 1 HOUR; BF IMAGE 50,000X.

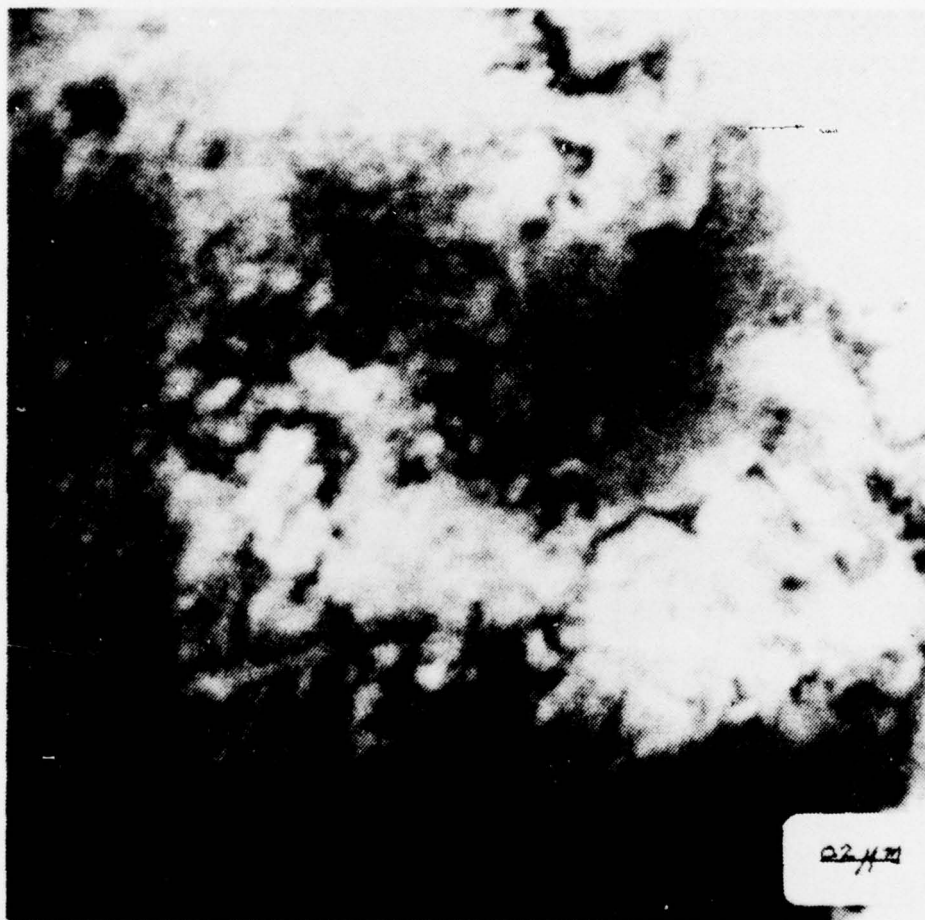


Figure 8 - CARTRIDGE BRASS STRAINED TO 60 PERCENT AND ANNEALED AT 430°F FOR 1 HOUR; BF IMAGE 50,000X.



Figure 9 - CARTRIDGE BRASS STRAINED TO 60 PERCENT AND ANNEALED AT 525°F FOR 1 HOUR; BF IMAGE 50,000X.

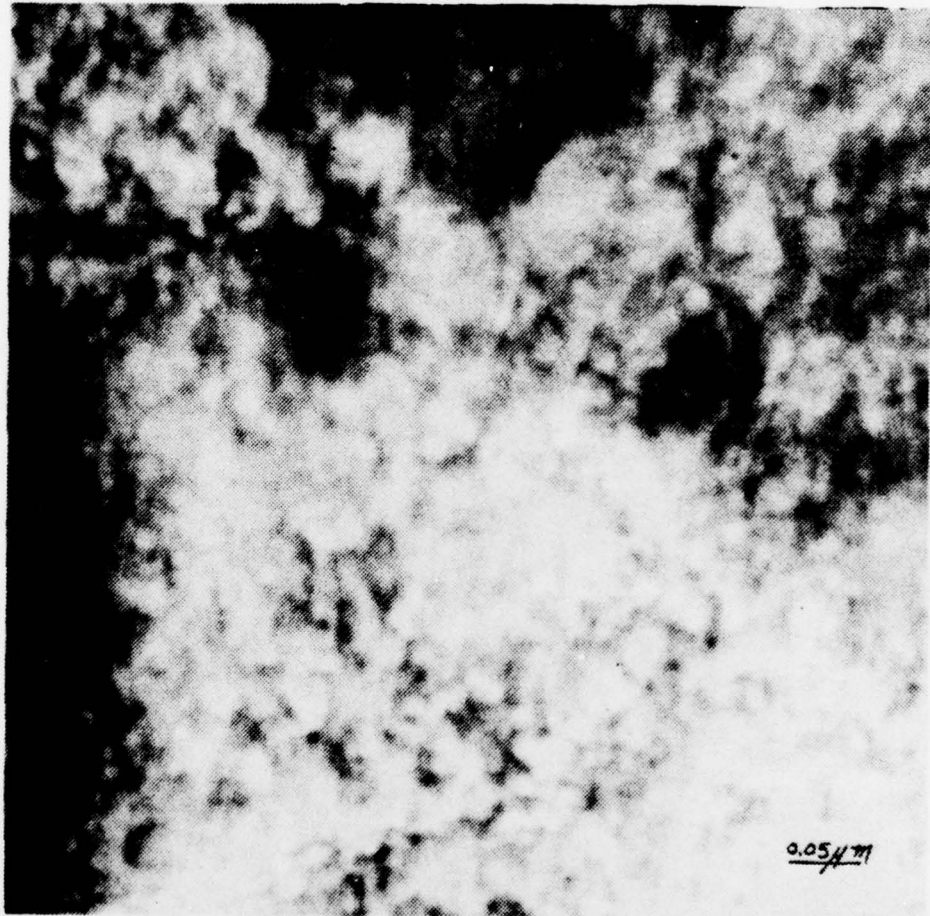


Figure 10 - CARTRIDGE BRASS STRAINED TO 60 PERCENT AND ANNEALED AT 525°F FOR 1 HOUR; BF IMAGE 200,000X.



Figure 11 - CARTRIDGE BRASS STRAINED TO 60 PERCENT AND ANNEALED AT 670°F FOR 1 HOUR; BF IMAGE 50,000X.

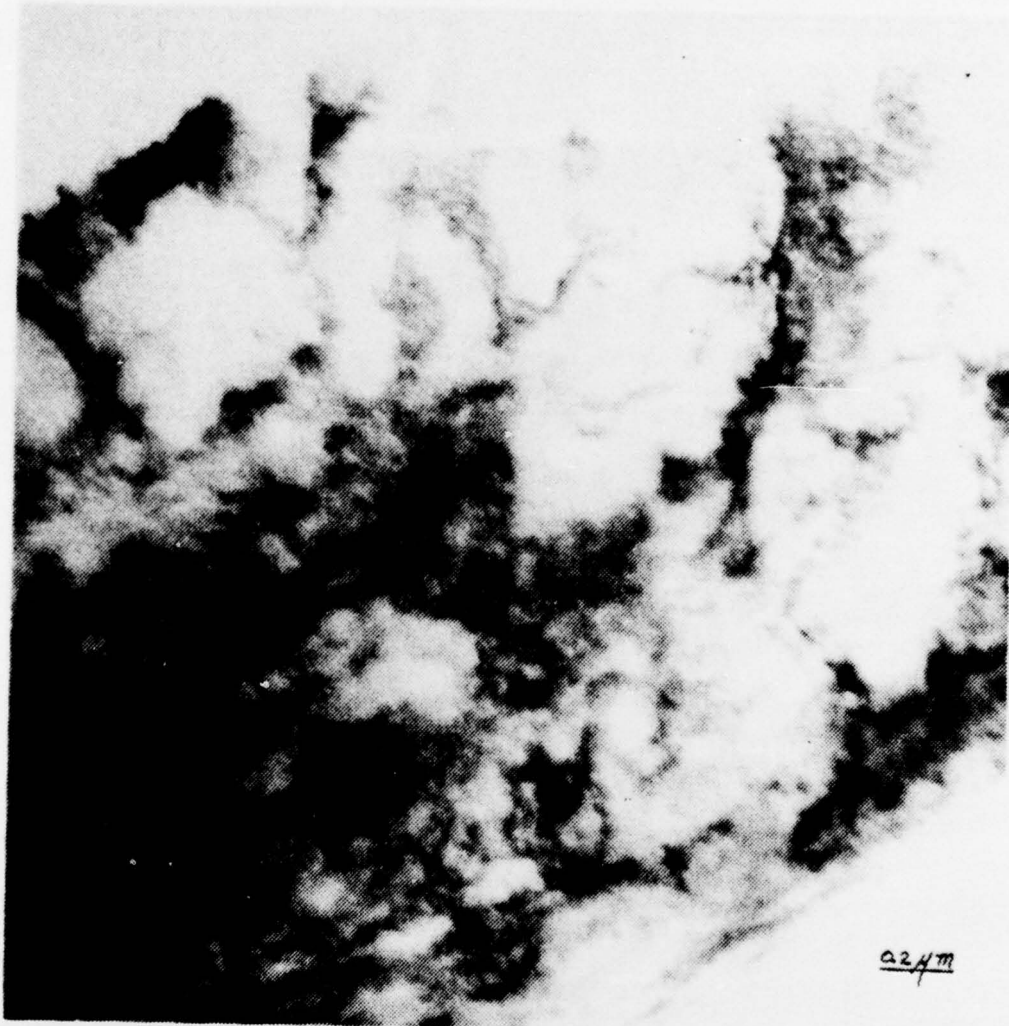


Figure 12 - CARTRIDGE BRASS STRAINED TO 20 PERCENT AND ANNEALED AT 360°F FOR 1 HOUR; BF IMAGE 50,000X.

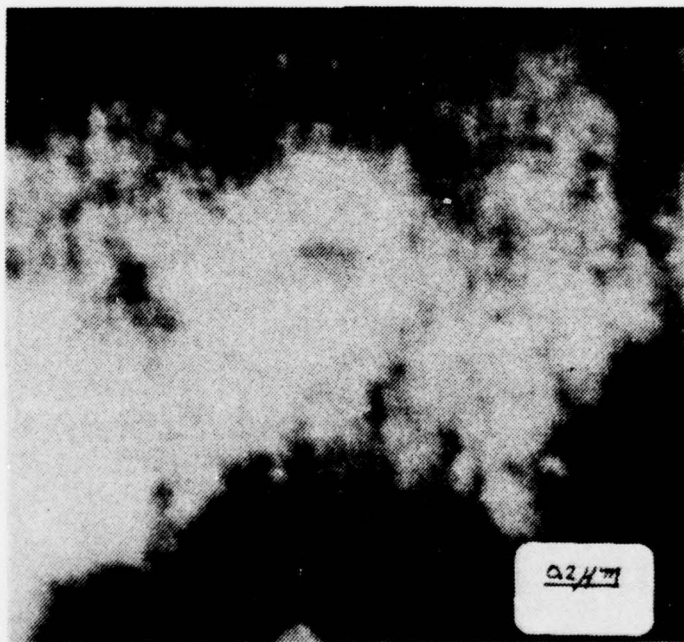


Figure 13 - CARTRIDGE BRASS STRAINED TO 40 PERCENT AND ANNEALED AT 430°F FOR 1 HOUR; BF IMAGE 50,000X.

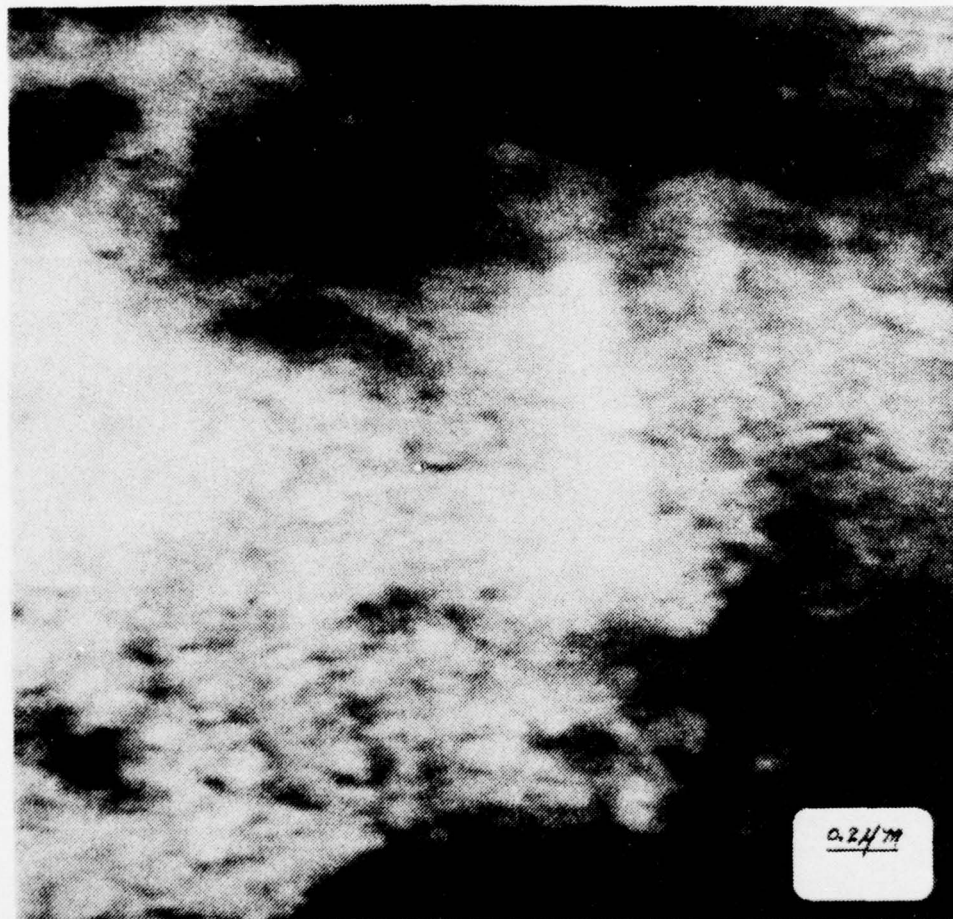


Figure 14 - CARTRIDGE BRASS STRAZNED TO 60 PERCENT AND ANNEALED AT 525°F FOR 1 HOUR; BF IMAGE 50,000X.

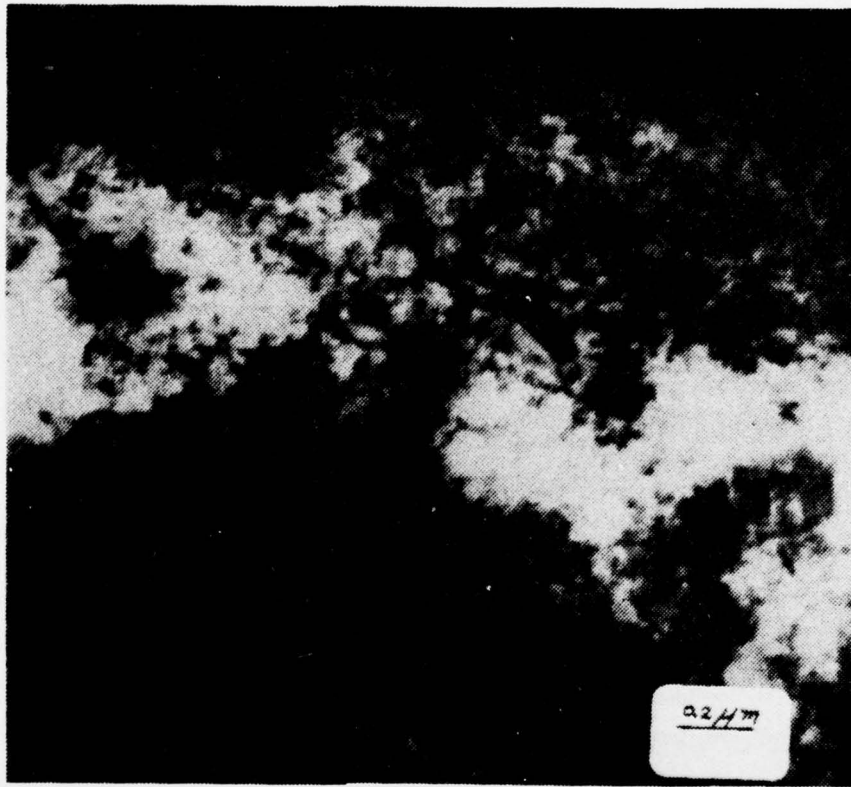


Figure 15 - CARTRIDGE BRASS STRAINED TO 90 PERCENT AND ANNEALED AT 430°F FOR 1 HOUR; BF IMAGE 50,000X.

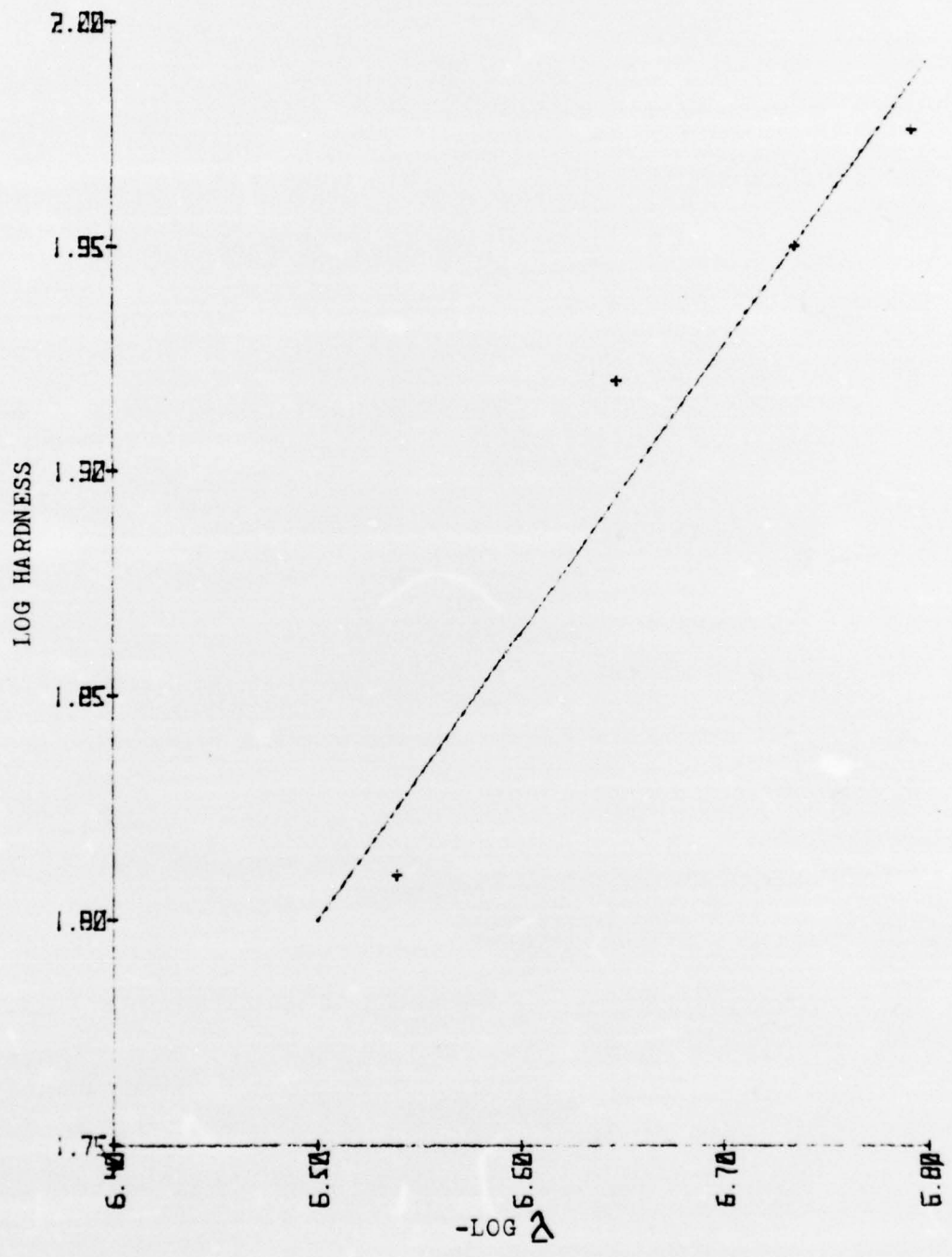


Figure 16 - LOG HARDNESS VERSUS -LOG λ.

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