A STUDY ON THE NATURE OF ANNEALING TWINS

J. R. Hancock, K. L. Keating, D. J. Murphy

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ABSTRACT

An investigation was carried out which was primarily concerned with the properties and phenomenological behavior of noncoherent twin boundaries in copper and copper-base alloys. Direct observation of twinning phenomena was carried out on an evacuated heating stage; the three-dimensional geometry of annealing twins was determined; and the detection of solute segregation by potential probe measurements, the effect of time and temperature of heat treatment and specimen size on twinning frequency, and the effect of mechanical loading on twin boundaries, was investigated.

The investigation has shown the importance of considering the three-dimensional character of annealing twins and of the twinning processes taking place, and the effect of time and temperature, on twinning frequency. Noncoherent twin boundaries were shown to be relatively high energy boundaries extending through a crystal in the form of loops, which, at elevated temperatures, may migrate in either direction parallel to coherent twin boundaries. Migration was observed to occur in a stepwise, discontinuous manner. Noncoherent twin boundaries were moved thermo-mechanically under conditions of severe abrasive deformation, in which case they resembled their counterparts in mechanical twins.
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1.0 INTRODUCTION

Annealing twins are a common structural occurrence in recrystallized face-centered cubic (FCC) metals and alloys, and have come into special prominence in the last decade as a criterion for estimating relative stacking fault energies of different FCC metals and alloys.

An annealing twin is, structurally, a mirror image of the host grain across a crystallographic plane called a twinning plane. In the FCC lattice, the twinning plane is the close-packed octahedral plane and coincides with the plane of contact (also called the composition plane). In this case, the boundary is called a coherent twin boundary since exact matching of atom positions exists across this boundary. The stacking arrangement of planes of close-packed atoms (octahedral planes) may easily be visualized by the following notation. The usual FCC arrangement is ABCABC illustrating the three possible stacking positions atoms may assume. A coherent twin boundary is then ABCBA, where C is the twinning plane, and the atom arrangement on one side of the boundary is the mirror image of that on the other. Coherent twin boundaries have never been observed to move. They are relatively low energy boundaries.

Another type of annealing twin boundary is the noncoherent boundary, a relatively high energy boundary. As the name suggests, exact matching of atom positions across this boundary does not occur. An annealing twin is then often observed as two parallel coherent boundaries joined at each end by a noncoherent boundary (assuming the twin does not
intersect a grain boundary) which may assume any direction. Noncoherent twin boundaries are thermally unstable and at elevated temperatures may migrate in either direction parallel to the coherent boundaries. When the boundaries move in such a direction as to broaden a twin band, the process is called coalescence.

Annealing twins are thought to be produced as a result of accidental incorrect positioning of close-packed planes of atoms during grain growth. The supporting evidence for this view is considerable and is widely accepted. More recently it has been proposed that noncoherent twin boundaries are made up of glissile (i.e., capable of glide) partial dislocations lying on adjacent close-packed planes. This model then projects a three-dimensional image of an annealing twin, viz., a series of adjacent partial dislocation loops. The supporting evidence appears convincing and suggests a more detailed investigation into the phenomenological behavior of noncoherent twin boundaries, since the stability, growth, and disappearance of annealing twins will depend upon the properties of noncoherent twin boundaries, and thus upon partial dislocations. It seems worth mentioning that until 1959 no interpretation or proposals concerning annealing twin phenomena were concerned with a three-dimensional model of a twin, and the theory of twin formation currently favored was formulated entirely on a two-dimensional model. In fact, most text book drawings seem to omit sketching the extension of a twin down into a metal grain. It has become increasingly clear that noncoherent twin boundaries and their properties constitute a major area of inadequate understanding of annealing twin phenomena and impede the formulation of a theoretical explanation of the processes taking place.
Related to the need for knowledge concerning the behavior of twin boundaries is the frequency of occurrence of twins. The frequency of annealing twins (average number of twins per grain) in metals and alloys varies with grain size. This observation, however, is apparently not widely recognized or perhaps even not thought to be significant by several investigators. It has been noted that recent papers reporting twin frequencies make no mention of grain sizes.

It appears that it would be desirable to have more detailed knowledge about such things as the thermal properties and behavior of noncoherent twin boundaries, the effects of mechanical loads on these boundaries, the three-dimensional geometry of annealing twins to aid the spatial conception of the processes operating in the production, growth, and annihilation of twins, the effects of time and temperature on the frequency of occurrence of annealing twins, and whether or not segregation occurs at twin boundaries. It is clarification of such generally inadequate knowledge of the properties of noncoherent twin boundaries and resulting lack of understanding of twin behavior, which is the purpose of this investigation.
2.0 LITERATURE SURVEY

2.1 Summary of Proposed Theories

Several reviews have been published concerning twinning phenomena. These include Burke and Turnbull, 1952; Clark and Craig, 1952; Hall, 1954 and Cahn, 1954. Four major theories of annealing twin production, growth and disappearance have been proposed:

a) The mechanical nucleation hypothesis, (Mathewson and Phillips, 1916; Mathewson, 1928, 1944) predicts the growth of annealing twins from mechanically induced nuclei called twin faults.

b) The growth-accident hypothesis, proposed by Carpenter and Tamura (1926) and developed by Burke (1950), states that annealing twins might form as a result of accidents of growth.

c) The Fullman-Fisher hypothesis (1951) proposes that annealing twins form as a result of a decrease in the interfacial free energy of grain boundaries that would not be achieved in the absence of twinning.

d) The Burgers stimulation process (Burgers, 1946, 1949) proposes that a growing recrystallized grain meets with a dislocation-bearing fragment lying in twinned orientation with it. The fragment discharges its dislocations into the growing crystal, and as a result is able to grow at the expense of the surrounding deformed matrix.

Currently, the growth-accident hypothesis together with the Fullman-Fisher conditions is the most widely accepted mechanism of annealing twin production and growth. Fullman and Fisher (1951) have also postulated the only mechanism for the formation of noncoherent twin boundaries. This theory has been reported to have been confirmed in austenite by Grube and Rouze (1963) who further conclude that the
discontinuity mechanism of twin nucleation which had been accepted by Burke and others is not, in fact, necessary. The major conclusions reached in order of chronological development are as follows:

a) Burke (1950)

1) Twins can form whenever a grain boundary migrates.

2) Annealing twins usually form at or near the corners of grains during grain boundary migration.

3) The number of twins per unit volume is greater in a recrystallized metal following heavy deformation than in lightly deformed specimens, suggesting retention of some kind of fault nucleation mechanism.

4) Since twins may form by grain boundary migration, deformation is not a necessary prerequisite.

5) The width of twins and their frequency are said to be functions of grain size, with finer grain sizes producing narrower and more frequently-occurring twins.

6) Twin broadening occurs by:
   a) Simple grain boundary migration, and
   b) Coalescence (Mathewson, 1928), i.e., by movement of noncoherent twin boundaries parallel to 111 twinning planes.

7) At least three conditions must be met in order for annealing twins to form:
   a) The advancing grain boundary must approximate an octahedral plane of the growing grain at the position where the twinning accident occurs.
   b) The boundary must encounter a discontinuity which will induce a twinning accident.
   c) The twinned orientation must present advantages (e.g., for growth) in order to be propagated for an observable distance.

b) Fullman-Fisher (1951)

1) The presence of annealing twins at the corners of certain grains corresponds to a lower total interfacial free energy than would the absence of twins.
2) New stationary annealing twin boundaries appear at moving grain corners whenever a decrease in the overall interfacial free energy would result.

3) It is proposed that noncoherent twin boundaries form during grain boundary migration when a coherent twin boundary becomes tangent to a migrating grain boundary.

c) Grube and Rouze (1962 and 1963)

1) Annealing twins in austenite are nucleated during grain growth only at corners of grains as the junction migrates.

2) Annealing twins grow both by broadening and by lengthening when the twin boundary coincides with a migrating grain boundary.

3) A twin band will continue to be lengthened by migration of a grain boundary as long as the direction of grain boundary motion has a component parallel to the coherent twin boundary.

4) A twin may be induced to grow by forces acting outside its boundaries.

5) Twins may broaden by coalescence as suggested by Burke.

6) The Fullman-Fisher mechanism of noncoherent twin boundary formation is operative in austenite.

7) Annealing twins are annihilated by grain boundary migration and by noncoherent twin boundary migration parallel to coherent twin boundaries.

8) Twins in austenite which terminate at grain boundaries are thermally stable, but those which terminate within a grain are thermally unstable and anneal out easily.

2.2 The Nature of Annealing Twins

In 1959, Votava and Berghezan postulated a dislocation model in which an annealing twin is made up of glissile partial dislocation loops lying on adjacent \{11\} planes, thus producing the twinning stacking arrangement. Later papers (Votava and Hatwell, 1960; Votava,
1961) provided additional experimental support for the theory. Thus, noncoherent twin boundaries are the emergence points of imperfect dislocations called twinning dislocations, and are confined to the octahedral twinning planes. The authors concluded that the partial dislocations are independently mobile and the noncoherent twin boundary cannot be associated with a particular lattice plane. This view explains the divergent results of attempts to index the noncoherent boundaries. Greninger (1936) reported this boundary to be \{110\} planes common to both the parent and twin crystals, and Fullman (1951) observed that the stereographic locations of poles of noncoherent boundaries corresponded to a \{113\} pole of one crystal and a \{335\} pole of the other. Very recently (Dash and Brown, 1963) this boundary has been indexed as either \{531\}, \{353\}, or \{110\}. Similarly, the above dislocation model may be applied to explain a number of other properties of annealing twins, viz., growth and disappearance of twins parallel to octahedral planes, preferential etching of noncoherent twin boundaries, and preferential precipitation at twin boundaries (Hatwell, Votava, 1961).

Fullman (1951a, 1951b) has measured both the ratio of interfacial free energy of coherent twin boundaries and noncoherent twin boundaries to the average grain boundary free energy in copper. The mean values found were $0.035 \pm 0.006$ and $0.80 \pm 0.015$ respectively. Similar results (Fisher, 1952) of the ratio of interfacial free energy of coherent twin boundaries to the average grain boundary free energy for aluminum gave 0.21. Thus, experimental evidence indicated that the inequality of the following expression may be satisfied in the majority
of cases:

\[ L_{ab}T_{ab} + L_{ac}T_{ac} + L_{aA}T_{aa} < L_{ab}T_{ab} + L_{ac}T_{ac} \]

where \( L \) is the length and \( T \), the interfacial free energy of the boundaries indicated by the subscripts and illustrated in Figure 1, Appendix A. The Fullman-Fisher hypothesis predicts that the frequency of twinning should decrease with increasing ratio of twin boundary to grain boundary free energy, and, thus, with increasing extent of preferred orientation.

2.3 The Frequency of Annealing Twins

The frequency of annealing twins is reported to be independent of annealing time and temperature as such are to depend upon these parameters only insofar as they affect the resulting grain size (Hu and Smith, 1956; Lauta, 1960). Twinning frequencies in copper and copper-base alloys have been reported by Hibbard, Lui and Reiter (1949, 1950), Hu and Smith (1956), Lauta (1960), and Mack, Murphy, and Hancock (1962). The results of the latter show that annealing twin frequencies increase with grain size up to some maximum value, then decrease, with all twin frequency curves of twins/grain vs. grain size showing a maximum of twins/grain at some intermediate grain size. The subsequent decrease in twins/grain could be due to any one or a combination of the following: Coalescence, preferred orientation, increasing temperatures of heat treatment used to obtain the larger grain sizes, and/or dissipation of the twin-producing mechanism at large grain sizes. Hu and Smith (1956) attribute a similar effect in their work (below 700°C.) and in Hibbard et.al.'s work to changes in
preferred orientation with grain size.

A correlation of the effects of alloying elements on twinning frequency is inconclusive at present. The results suggest further investigation concerning the effects of time and temperature or twinning frequency as will be discussed in a later section.
3.0 OBJECTIVES OF THE INVESTIGATION

The objectives of this investigation have been as follows:

a) To study the production, growth, and disappearance of annealing twins by direct observations of the microstructure on an evacuated heating stage.

b) To study the effects of mechanical loading on annealing twin boundaries.

c) To determine the three-dimensional geometry of annealing twins.

d) To investigate the possibility of solute segregation at annealing twin boundaries.

e) To determine the effects of time and temperature on annealing twin frequency.

f) To determine the effect of specimen size on annealing twin frequency.

3.1 Direct Microstructural Observations Using an Evacuated Heating Stage

Vacuum heating stage attachments have been applied successfully to the observation of microstructural behavior of metals. The use of such an attachment for direct observation of twinning phenomena offered the possibility of clarification of details concerning the dynamics of annealing twin production, growth, and disappearance, the possibility of activation energy determinations for noncoherent twin boundary motion, and direct verification of theoretical principles. For these purposes, and to obtain more detailed knowledge about the behavior of noncoherent twin boundaries, the Reichert Vacutherm heating stage was employed in the investigation.
3.2 **Effect of Applied Mechanical Loading on Annealing Twin Boundaries**

The recently proposed dislocation model of noncoherent twin boundaries suggests that these boundaries may be mobile under suitable conditions of applied mechanical loading. Partial dislocations must necessarily exist on each close-packed plane in order that the FCC stacking arrangement be produced within the twin. This arrangement may be expected to impart rather unique mechanical properties to the boundary, particularly in view of their mode of formation. It might be expected that one of the conditions of mechanically induced mobility would be load application at low temperatures, by analogy with mechanical twinning phenomena. On the other hand, once the boundary is formed, the Peierls force (force necessary to move a dislocation from one crystallographic potential valley to the next) may preclude motion at low temperatures. Furthermore, an arrangement of contiguous partial dislocations, recently suggested to be made up of partials with all three possible Burgers vectors (Dash and Brown, 1963), might be expected to be a rather low energy, stable arrangement. Measurements by Fullman (1951b), however, have shown that the energy of a noncoherent twin boundary in copper is 0.8 of the average grain boundary energy, a rather high value. Thus, one might reason that contiguous partial dislocations, with the possible Burgers vectors equally and randomly distributed, result in a high boundary energy primarily due to atomic mismatch, i.e., the high energy is not associated with a shear stress concentration at the boundary as in mechanical twins. The exact nature of this mismatch has not been discussed in the literature.
The movement of noncoherent twin boundaries under applied mechanical loading would support the dislocation model, and together with the experimental conditions, could lead to further deductions concerning the relative energy of the boundary and extent of atomic mismatch.

3.3 Three-Dimensional Geometry of Annealing Twins

It is perhaps surprising that prior to 1959 no three-dimensional model of annealing twins was proposed. The failure to visualize annealing twins in three dimensions, as well as the dynamic processes involved in twinning behavior, appear to be major obstacles to an accurate understanding of annealing twin phenomena. Early attempts to index noncoherent twin boundaries as crystallographic planes perhaps indicate that such boundaries were largely thought of as being planar. Following the proposed dislocation model, the determination of twin profiles (three-dimensional geometry) is a next obvious step to resolve remaining controversy. Twin profile determinations might also be expected to yield information concerning the effects of interactions with twins and with grain boundaries and on annealing twin stability at elevated temperatures.

3.4 Segregation of Solute Elements to Twin Boundaries

If noncoherent twin boundaries are made up of partial dislocation loops, solute segregation would in some cases, be expected to take place and thus affect the growth, disappearance, and thermal stability of annealing twins. A Suzuki type of chemical interaction might also be expected at coherent twin boundaries.
At each type of twin boundary the disruption of the lattice will bring about an increase in the lattice energy due to the hexagonal layer of atoms at the twin plane, and due to atomic mismatch at the noncoherent twin boundary. Under these circumstances solute atoms (in brasses) could segregate at twin boundaries. Any change in lattice energy at the twin boundaries should change the bulk resistivity at the anomaly. These lattice disturbances would be, in effect, free electron barriers which impede the normal flow of electrons.

The objective of this part of the investigation is to determine if solute segregation exists in the vicinity of twin boundaries in commercial brasses. By making voltage traverses across coherent and noncoherent twin boundaries with a microprobe, slight changes in the resistivity of the material should be observed. Such a traverse made on copper would only reveal inhomogenieties in the physical structure. A similar traverse made on a copper-zinc alloy would show a combination of physical structure and solute segregation effects. Thus, if solute segregation exists at twin boundaries, such segregation may be detectable by these means.

No determinations were made using an electron-probe X-ray microanalyzer due to the inavailability of the apparatus.

3.5 **Effect of Time and Temperature on Annealing Twin Frequency**

The effect of time of heat treatment on annealing twin frequency has not been rigorously investigated. The effect of temperature is not clear and no systematic study has been carried out, though it may be concluded from published literature that little or no effect is to be expected (e.g., Hu and Smith, 1956). Lauta (1960) concludes that the
twinning frequency for a given metal or alloy depends only upon grain size. Hu and Smith further state that textural changes are believed to account for the decrease in twinning frequency at larger grain sizes.

Since no extensive work has been carried out, systematic investigation appeared to have fruitful possibilities. Furthermore, since three distinct processes (i.e., production, growth, and disappearance) take place simultaneously, it would be desirable to suppress or isolate one or more of the processes in order to obtain a representative value of the isolated process (twin production values, for example). Decreasing the time of heat treatment offered the possibility of suppressing twin annihilation by noncoherent twin boundary migration.

3.6 Effect of Specimen Size on Annealing Twin Frequency

Preliminary work by Mack et. al. (1962) indicated the possibility of a specimen size effect on annealing twin frequency, with smaller specimens yielding lower twin frequencies. It is not clear if such an effect is in fact present, and for this reason this possibility also offered investigative promise.
4.0 EXPERIMENTAL PROCEDURES

The materials, apparatus, and experimental procedures employed in this investigation are described in the following sections.

4.1 Materials

The purity of the materials used are tabulated below:

a) Tough Pitch Copper (99.92 Cu)
b) 99.99+ Percent Copper
c) 99.99+ Percent Silver
d) 99.99+ Percent Gold
e) 99.996+ Percent Aluminum
f) 99.97+ Percent Tin
g) 99.95 Percent Nickel (Electrolytic, Contains Unremoved Cobalt)
h) 99.999+ Percent Indium
i) 99.999+ Percent Silicon
j) 70-30, 90-10 Commercial Wrought Brass

The commercial brasses were obtained from Anaconda American Brass Company in either cold-rolled and annealed sheet or extruded and cold-drawn rod. Detailed chemical analyses reported by the suppliers are given in Table I, Appendix B.

4.2 Alloy Preparation

Melts weighing approximately 350 grams were prepared of pure copper (99.99+ Percent) and copper containing nominally 1.5-2 atomic percent of silver, gold, zinc, aluminum, indium, tin, nickel, and silicon, respectively. Melting was done in high purity graphite crucibles. All of the alloys except Cu-Zn were melted under a vacuum of approximately $5 \times 10^{-2}$ mm Hg in an induction furnace powered by a 10 KW Westinghouse, 450 KC high frequency generator. Figure 3, Appendix A is a photograph of the apparatus. The ingots were allowed
to solidify in the crucible and were subsequently remelted (except the Cu-Zn alloy) and cast in vacuo in high purity graphite multiple cavity molds (See Figure 4, Appendix A), and allowed to solidify in the mold under vacuum. In this manner sufficient specimens of the same composition were obtained for the separate phases of the investigation. The Cu-Zn alloy was prepared in a high purity graphite crucible in a resistance heated furnace under an atmosphere of dry argon. The zinc addition was made in the form of commercial brass containing 10.09 w/o zinc. This addition resulted in the addition of 0.005 percent impurities to the pure copper. It was not necessary to recast this alloy since it was not examined on the vacuum heating stage because of the high volatility of zinc. All alloys were subsequently annealed 24 hours in dry argon at 850°C.

4.3 Chemical Analyses

The chemical analyses on the alloys were performed by Ampco Metal Company, Milwaukee, Wisconsin, and are tabulated in Table II, Appendix B.

4.4 Direct Microstructural Observations Using an Evacuated Heating Stage

Pure copper and copper alloys of Ag, Sn, Si, and In were investigated on a Reichert Vacutherm Heating Stage. Specimens were specially prepared by machining cast, homogenized, and cold-pressed rods to ten millimeters in height and seven millimeters in diameter. A supporting ledge, which rests on a quartz ring in the heating stage, was machined at the bottom and a thermocouple well drilled in the side (Figure 5, Appendix A). The heating stage is shown in Figure 6,
Appendix A. The heating stage was operated to a ultimate vacuum of $6 \times 10^{-5}$ mm Hg and at temperatures approaching the melting points of copper and its alloys. The heating stage was operable to a maximum temperature of 1600°C. A typical experimental run usually extended over a period of several hours, with variation from specimen to specimen, depending on whether significant and interesting structures developed. Photomicrographs were recorded on either 35 mm film or on 4 x 5 inch sheet film. It was learned that no practicable rate of thermal etching was achieved for copper and its alloys until temperatures above 700°C were realized. For this reason, and since the objective was to observe boundary motion directly, all observations were made above 700°C in order to reduce the time necessary for thermal etching. The estimated time lag for thermal etching at 700°C was of the order of a few seconds. Temperatures were recorded to an estimated ± 20°C determined by recording melting temperatures of copper and a copper-tin alloy.

In most specimens, recrystallization was carried out in the heating stage; in a few, however, recrystallization was carried out prior to examination in the heating stage. In the latter specimens, grain growth was rather sluggish and considerably less boundary motion was observed.

4.5 Effect of Applied Mechanical Loading on Annealing Twin Boundaries

Pure copper (99.99 Percent) and dilute copper-base alloys of silver and aluminum were examined in this part of the investigation since previously determined twinning frequencies (Mack et al., 1962)
indicated these alloys to have twin boundary energies higher and lower, respectively, than that of copper. As-cast and homogenized specimens were cold-pressed to 30 percent reduction in thickness in a small hydraulic press, machined to 1 1/8 x 1/4 x 3/32 inches, and mechanically polished on three surfaces for subsequent microexamination. The specimens were then recrystallized and annealed at 950°C in dry argon for three hours. This treatment resulted in large grains and twins. The specimens were lightly repolished and etched, loaded in three point bending on an Instron testing machine while immersed in liquid nitrogen. They were then unloaded, removed, and examined metallographically both before and after a light etch. A flow-diagram of the procedure is given in Figure 7, Appendix A. Pure copper (99.99+ Percent) was loaded similarly at room temperature.

Each of the specimens was also abraded on 80 grit emery paper on a belt sander both immediately after immersion in liquid nitrogen and at room temperature. In the first case the abrasion was limited to 1-2 second intervals and the specimens were re-immersed in liquid nitrogen between abrasions. In the latter case, the specimens were abraded for 5-6 second intervals, to cause them to become hot, and were plunged into water between abrasions.

4.6 Three-Dimensional Geometry of Annealing Twins

A flow-diagram of the procedures adopted in this phase of the investigation is shown in Figure 8, Appendix A. As-cast and homogenized ingots of pure copper were hot-pressed in three mutually perpendicular directions in a small hydraulic press immediately after being heated to
800°C for 3-5 minutes in a neutral salt bath. The ingots were then sectioned into one-half inch cubes, and cold-pressed to 50 percent reduction in thickness. A specimen was then recrystallized and heat treated for 30 minutes at 800°C in a neutral salt bath to produce twins. It was then mechanically polished, electropolished, and electroetched in standard Disa Electropol D-2 electrolyte (250 ml phosphoric acid, 500 ml distilled water, 250 ml ethanol, 2 ml Vogel's sparbeize, 50 ml propanol, 5 g urea).

The thickness of material removed from the surface after each electropolishing and electroetching operation was measured with the vertical focusing mechanism on a Reichert metallograph. An average of five depth readings were taken at a magnification of 520x. The maximum range of the five readings never exceeded three microns. Electropolishing at a current density of approximately 0.75-1.0 amps/cm² for 40 seconds usually removed 10-15 microns of material. The dimensions of twins at successive depths were measured at a magnification of 400x on a Leitz microhardness tester using microindentations as reference points. Microindentations were used as reference points as a means of determining the direction followed by the boundaries through the crystal. Following these measurements, the specimen was again electropolished and electroetched, and the cycle repeated. By this means twin dimensions were readily measured to less than ± 1 micron, as determined from a series of such measurements. There was an estimated maximum error of ± 4 microns in locating boundary positions after successive polishes due to the difficulty in locating the center of the microindentations after polishing. Grain orientations were
determined by the measurement of angles between coherent twin boundaries. These angles were measured periodically to determine whether polishing had proceeded perpendicular to the specimen surface.

4.7 Segregation of Solute Elements to Twin Boundaries

A microprobe was used to determine if solute segregation existed at twin boundaries. The probe measured slight changes in the resistivity of the material by a series of voltage measurements large twinned grains were desirable.

Various techniques were used to make large twinned grains in copper and high brass alloys. Samples of commercial alloys were cold rolled to about 75-80 percent reduction. Best results were then obtained by annealing these in an inert atmosphere of argon at a temperature very close to the fusion point. Very large grains were made in copper specimens; relatively small grains (about 1 mm) were obtained in the brasses. Samples of 90-10 and 95-5 brass seemed to make the most satisfactory samples for these measurements. The lower brasses were subject to de-zincification at the high temperatures used for recrystallization.

A probe was constructed so that a given thin sample could be moved precisely along x-y coordinates under a flame-polished tungsten point. After positioning the sample the probe point was lowered onto the surface and a measurement was made.

The measurements were made by observing the voltage drop across a traverse made on the surface of the sample. Voltage measurement was made on a Rubicon Type "B" using an external galvanometer. The circuit used for these measurements is shown in Figure 9, Appendix A.
A constant potential was impressed across a thin sample of copper or brass. This voltage was checked frequently by measuring the voltage drop across the one ohm standard resistor (Bureau of Standards Type). A current of about 28 milliamps was maintained in the sample loop. When the DPDT switch was in the opposite position, the voltage drop across the left half of the sample was measured.

Traverses were made on pure copper samples as well as copper-zinc alloys. Thus, a comparison could be made between effects not due to segregation (in the copper sample) and effects possibly due to segregation (as seen in the brass sample).

4.8 Effect of Time and Temperature on Annealing Twin Frequency

Three series of pure copper and one series of tough pitch copper specimens were used in this part of the investigation. As-cast ingots were hot-pressed in three mutually perpendicular directions in a small hydraulic press. Specimens were immersed in a neutral salt bath at 800°C for 3-5 minutes, removed, pressed, and returned to the salt bath until the ingot was approximately cubical. Each hot-pressed ingot was sectioned into 5/8 inch cubes. The cubes were cold-pressed in three mutually perpendicular directions to 50 percent reduction in thickness in a Tinius Olsen hydraulic press. These specimens were then cold-rolled to 50 percent reduction in thickness in the case of the pure copper to 70 percent reduction in thickness in the case of the tough pitch copper specimens.

A series of the pure copper specimens were recrystallized in a neutral salt bath at each of the following bath temperatures: 650°C,
750°C, and 830°C for times varying from 1-2 seconds to several minutes, in order to produce a range of grain sizes. The heat treatments were carried out by immersing the specimens in the salt bath for the desired time and removing and quenching them in water. The tough pitch copper series was processed in the same manner at 750°C. All specimens were mounted in bakelite and prepared for microexamination by standard metallographic procedures. All specimens were etched by swabbing in a 2:1 mixture of NH₄OH:H₂O₂.

Grain sizes were determined by comparison with ASTM standard grain size chart E-79. Twin counting procedures adopted were those previously developed and consisted of individual and systematic examination of grains on a Reichert metallograph. Previous results (Mack et. al., 1962) indicated that fewer total counts could be made with little or no sacrifice in statistical accuracy, and therefore, total counts were reduced from 100 to 80 grains. The following arbitrary rules were set up to standardize the procedure:

a) Four samples of 20 grains each, totaling 80 grains were counted in each specimen.

b) Grains were selected for counting by translating the stage of metallograph a predetermined distance between each grain examined. The grain to be examined would then be positioned under the midpoint of a cross hair in the ocular. The stage translations were chosen to be larger than the average grain diameter. The procedure was to examine 20 grains in a straight line, then to translate the stage two to four times the average grain diameter in a direction perpendicular to the straight line and to repeat the procedure until 80 grains had been examined.

c) No grains were examined within three grain diameters of the edge of the specimen.

d) All twins were counted in the particular grain being examined, i.e., secondary and higher order twins were also counted. The number of twin families in a particular grain was also recorded.
e) Abnormally large or small grains, i.e., those clearly deviating from the average grain size, were not counted.

f) Grains in which structural details could not be positively identified were not counted.

g) If the twin count could be made in such a way as to yield two different but consecutive values, the lower value was recorded.

The average number of twins/grain, twins/twinned grain, and twin families/twinned grain were calculated.

4.9 Effect of Specimen Size on Annealing Twin Frequency

Commercial wrought 70-30 brass was used for this part of the investigation. Sheet, 0.05 inches thick, as received in the cold-rolled and annealed condition, was cold-rolled to 20 percent reduction in thickness. Specimens were cut to a final size of 0.04 x 1/4 x 5/8 inches. Rods, 5/8 inches in diameter, as received in the extruded and cold-drawn condition was sectioned into 1/2 inch lengths for one series of specimens, and sectioned and ground to 0.04 x 1/4 x 5/8 inches for another series (identical to the size of the sheet specimens). All specimens were recrystallized for one hour at temperatures ranging from 398-759°C in a neutral salt bath. Twinning frequencies were then determined as described in the previous section, and twins/grain, twins/twinned grain, and twin families/twinned grain were calculated.
5.0 RESULTS AND DISCUSSION

The results of this investigation are presented and discussed in the following sections.

5.1 Direct Microstructural Observations Using an Evacuated Heating Stage

Thermal etching is a time dependent process and takes place in metal specimens as a result of evaporation of material from higher energy regions such as grain boundaries and twin boundaries. For a given material, the rate of evaporation depends upon the combination of vacuum and temperature employed. During observation at elevated temperatures, only those boundaries which are momentarily stationary ordinarily etch sufficiently to render them visible. Upon continued boundary migration, the previously etched boundary position tends to smooth out and become less distinct with time. These are referred to as "ghosts" in this report. Due to the nature of thermal etching, it was not possible to determine activation energies for motion of noncoherent twin boundaries since the time of initiation and cessation of motion could not be accurately recorded.

The following results were obtained from direct observations of twinning processes taking place at elevated temperatures.

5.1.1 Noncoherent Twin Boundary (henceforth abbreviated NTB) Motion Takes Place, Stepwise, in a Discontinuous Manner at Constant Temperature, and can Migrate in Either Direction Parallel to Coherent Twin Boundaries, Though Usually the Boundary Migrates so as to Annihilate the Twin.

Figures 10 and 11, Appendix A, are series of photomicrographs of a Cu-2 a/o Si specimen taken during heating in the evacuated stage, and
illustrate several characteristic phenomena. NTB's, as well as grain
boundaries, were observed to move discontinuously. NTB motion was
also observed to occur in steps; however, the previously existing steps
did not always remain intact. Figure 10 and 12 show illustrations that
NTB's can migrate in either direction parallel to coherent twin
boundaries. Both figures show the large influence that NTB's can exert
on an intersecting grain boundary, indicating that a relatively large
"tension" is to be associated with NTB's.

The discontinuous nature of NTB migration might reasonably be
expected to be the result of a number of factors. Among these would be
interaction with immobile forest dislocations and dislocation networks,
pinning by inclusions, grain boundaries, and other twins (see section
5.3), segregation effects, and the detailed nature, structure, and
properties of NTB's themselves, about which little is presently known.
It is at present not possible to decide which, if any, of these factors
are the more influential.

Under equilibrium conditions and disregarding other complicating
factors, NTB's would be expected to migrate with such a direction of
motion as would annihilate the twin, which indeed is the most commonly
observed behavior. There is no doubt, however, that elongation of a
twin can and does occur. One simple explanation is that the twin
(e.g., in Figure 12) intersects a grain boundary below the polished
surface and results in a lower interfacial free energy at the intersection.
The adjacent portions of the twin are then attracted to the grain
boundary, which, on the surface of the specimen, appears to be
uninfluenced twin elongation (Figure 12f). This possibility is also
suggested in Figure 12a through 12f by the fact that a grain boundary was migrating toward the twin, indicating that a twin-grain boundary intersection could have occurred below the plane of polish prior to the observed twin elongation. A schematic illustration is shown in Figure 13a, Appendix A. Similar effects could be expected at twin-twin intersections. Additional supporting evidence is pointed out in Section 5.3.

The limited elongation and subsequent reversal of motion observed in Figure 10a through 10e (boundary marked B-B') can be explained in terms of surface effect and dislocation line, or boundary, tension. If the NTB intersects the surface in such a way that the length of the boundary could be decreased by boundary movement, then twin elongation and subsequent reversal of motion could be observed on the specimen surface as illustrated in Figure 13b, Appendix A.

The reason that NTB's migrate in segments is not yet clear. This phenomenon could be intimately associated with the suggestion of Dash and Brown (1963) that the Burgers vectors of the partial dislocations making up the NTB need not be identical, but that all possible Burgers vectors for a given \{111\} plane occur equally and randomly. As previously mentioned, segments of NTB's do not necessarily remain intact, but can break up into new segments of shorter lengths, or alternatively, combine into larger segments. The driving force for annihilation by stepwise migration of NTB's would be the elimination of the high energy boundary, and when this occurs, the coherent twin boundary energy would not contribute, since another coherent boundary would be formed simultaneously. The break-up or combination of segments
occurs frequently and, apparently, randomly. Though the details governing this behavior have not been worked out, other factors which most probably contribute include the following:

a) A possible influential factor in the behavior of NTB's is that every third close-packed plane is capable of matching perfectly with the parent lattice across the NTB.

b) The break-up of segments occurs between close-packed planes in which the interacting forces between partial dislocations are repulsive or negligibly attractive.

5.1.2 No Twins or NTB's Were Observed During Formation

No twins were observed directly during formation; however, the small twin labeled A in Figure 12, Appendix A, possibly formed during the passage from left to right of the three-grain junction in the photomicrograph. The "ghost" of the three-grain junction is readily apparent in Figures 12a and 12b, where the migrating boundaries had stopped long enough for thermal etching to take place.

The NTB of twin B, Figure 12 could have been formed by the Fullman-Fisher mechanism (Figure 2b, Appendix A), however, this cannot be definitely established since the formation was not observed directly.

Many favorable sites for twin formation, i.e., migrating three-grain junctions were observed during heating in the vacuum stage. The fact that no twins were observed during formation is largely attributed to the relatively large grain sizes produced at the high temperatures necessary for rapid thermal etching. Grain growth was relatively slow in the large grain sizes, whereas fast rates of grain growth are thought to be conducive to twin formation. A question might also be raised concerning the likelihood of twin formation at a free
surface, though twin formation at a free surface has been observed by Grube and Rouze (1963) in austenite.

A possibility which might be expected at a free surface is illustrated in Figure 13c, Appendix A. In this case, if a twin were to form below the polished surface and, due to grain boundary interactions (see Section 5.3), were to expand and penetrate the surface near the center of a grain, it would give the erroneous impression of "spontaneous" twin nucleation.

5.1.3 Twin - Grain Boundary Intersections Inhibit Grain Boundary Migration

Grain boundary intersections with coherent twin boundaries, as well as NTB's, frequently exert a "drag" on the adjacent portions of a migrating grain boundary and inhibit grain boundary migration. An example may be seen in Figure 10a at A. Other examples may be found in Figures 12, 14, and 15, Appendix A. Similar effects have been observed in austenite by Rouze and Grube (1962). A striking illustration of this effect is the ease with which very large grains can be grown in essentially twin-free aluminum.

5.1.4 Decrease in the Interfacial Energy Resulting From Twin - Grain Boundary Intersections can Vary Considerably

Figure 14, Appendix A, illustrates the effect of the intersection of a twin and a grain boundary on the interfacial energy. It is readily apparent from the relative extent of thermal etching that has taken place that the interfacial energy has been lowered considerably as a result of the twinned orientation. The resulting decrease in interfacial energy can vary considerably and by way of illustration is
substantiated in Figure 10 and 12, Appendix A, where similar intersections resulted in lower energy reductions.

In view of the wide variation possible in interfacial energies at twin - grain boundary intersections, it is likely that twins can frequently dissociate from grain boundaries in a direction parallel to the coherent twin boundaries as suggested by Fullman and Fisher in 1951 and observed by Grube and Rouze in austenite (1963). This is primarily a result of interfacial energy changes due to the acquisition of new neighboring grains of different orientation during grain growth. Thus, as a new, differently oriented grain is acquired as a neighbor, the interfacial energy changes. If the energy of the twin - grain boundary intersection is raised relative to the remainder of the grain boundary, then the twin may pull away from the grain boundary.

5.1.5 Significantly Fewer Twins were Observed in the Cu - 2 a/o Ag Alloy Than in Pure Copper or in the Alloys of Cu - 2 a/o Sn, Cu - 2 a/o Si, or Cu - 2 a/o In

It was observed that very few twins were formed, or at least survived, in the Cu - 2 a/o Ag alloy during recrystallization and heating in the vacuum stage. These results concur with previous findings (Mack, et. al., 1962) that the twinning frequency in Cu - 2 a/o Ag is below that of pure copper (except at the largest grain sizes in the previous study).

5.1.6 Apparent Lateral Motion of a Coherent Twin Boundary, and Apparent "Dragging" of a Twin Through the Lattice by a Migrating Grain Boundary was Observed in a Cu - 2 a/o Si Alloy

A series of photomicrographs taken of a Cu - 2 a/o Si alloy is shown in Figure 15, Appendix A, in which a grain boundary and a twin
appear to be migrating together. The pinning effect of the inclusion on the grain boundary is also to be noted.

Of primary concern is the observation that the coherent twin boundary was displaced with a surface component perpendicular to itself (Figure 15e). Coherent twin boundary migration has never been observed previously, with one possible exception (Dash and Brown, 1963). Dash and Brown observed twin broadening by growth of stacking fault "packets" emanating from a grain boundary by glide on adjacent \{111\} planes; however, their observation was confined to extremely small twins in thin foils, growing into an unrecrystallized matrix, which may well turn out to be a specialized set of circumstances applicable only to the very early stages of recrystallization. The influence of thin foils on the observed phenomena could also be important and is discussed in Section 5.3 in regard to another observation. In any event, the application of this mechanism to explain Figure 15 would require that stacking fault "packets" be added to one side and removed from the opposite side, which does not seem likely. Another possibility is that the lateral motion is only apparent motion, and is properly accounted for in terms of surface removal by evaporation. However, in order that this may explain a lateral movement of approximately seven microns, the coherent twin boundary would have to intersect the surface at a rather small angle. In order to check this possibility, a back-reflection Laue pattern was taken of the grain in question and the pole of the grain surface was found to lie 12 degrees from a \{111\} pole. This angle would result in an evaporation rate of approximately 1500 close-packed atomic layers per minute which does not seem too unreasonable (950°C at a vacuum of $10^{-4}$ mm Hg).
Surface removal by evaporation may also account for the apparent "dragging" of the twin through the grain by the grain boundary in a direction parallel to the coherent twin boundaries. At present, however, it does not seem unacceptable that a grain boundary could exert such a force on the partial dislocations so that a "dragging" effect would be observed. This effect appears to be nothing more than NTB migration promoted by twin - grain boundary interaction.

5.1.7 Annealing Twins may be Broadened or Lengthened by Grain Boundary Migration

Annealing twins may be broadened or lengthened by grain boundary migration when the appropriate twin boundary coincides with the migrating grain boundary in agreement with the observations of Grube and Rouze (1963) in austenite. Examples of twin broadening by grain boundary migration may be seen in Figures 10 and 12, Appendix A, and examples of twin elongation by grain boundary migration are shown in Figures 10 and 14.

5.2 Effect of Applied Mechanical Loading on Annealing Twin Boundaries

The following results were obtained in this part of the investigation.

5.2.1 The Bending of Pure Copper and Copper Alloys Containing 2 a/o Al and 2 a/o Ag Respectively in Three-Point Loading at Liquid Nitrogen Temperature did not Result in Twin Boundary Motion. The Bending of Copper at Room Temperature Likewise Resulted in no Twin Boundary Motion.

Copper, Cu - 2 a/o Al, and Cu - 2 a/o Ag were loaded in three-point bending to a maximum normal stress of 24 Kg/mm², 22 Kg/mm²,
and 40 Kg/mm² respectively, calculated by using the relationships

$$\sigma = \frac{Mc}{I}, \text{ and}$$  \hspace{1cm} 5.1

$$I = \frac{bh^3}{12}$$  \hspace{1cm} 5.2

where M is the bending moment, I the moment of inertia, b the base and h the height of the rectangular specimens, and c taken as h/2. It was possible to load the Cu - 2 a/o Ag specimen to a higher stress level due to its having a higher yield strength. These stresses resulted in a similar amounts of specimen distortion. A cross-head speed of 0.02 inches per minute was used on the Instron testing machine. Specimens were examined for twin boundary movement on both the tension and compression sides and on the side containing the neutral axis; however, none was detected.

5.2.2  The Abrasion of Pure Copper and Copper Alloys Containing 2 a/o Al and 2 a/o Ag Respectively, at Liquid Nitrogen Temperature did not Result in Twin Boundary Motion; However, Abrasion at Room Temperature Caused Some NTB's to Move. The Resulting NTB Configuration Resembled Their Counterparts in Mechanical Twins

Abrading the specimens immediately after immersing in liquid nitrogen gave no indication of NTB motion. However, abrasion at room temperature, during which the specimens were allowed to become hot, indicated that NTB's did move in copper and in the Cu - 2 a/o Al alloy, apparently due to the combination of severe surface abrasion and thermal activation. No motion was apparent in the Cu - 2 a/o Ag alloy; however, it was observed that the Cu - 2 a/o Ag alloy contained significantly fewer twins and fewer possibilities for this type of motion, with most twins extending entirely across grains and intersecting the grain.
boundaries. Figure 16, Appendix A, shows examples of such NTB motion, in which the NTB's resemble the lenticular shape of mechanical twins. This boundary configuration suggests a shear stress concentration at the tip of the twin similar to that present in mechanical twins, and indicates that one of the major differences between the two types of NTB configuration is the mechanism of formation. If the partial dislocations in annealing twins all have identical Burgers vectors, as would be the case in mechanical twins, the NTB should be relatively easy to move, assuming the nucleation stress level to be significantly higher than the stress necessary to move a previously formed boundary. This situation, however, apparently is not the case with annealing twins, since severe abrasive deformation was necessary. In addition, thermal activation probably assists in the motion. These results then tend to support the contention of Dash and Brown that all of the possible Burgers vectors occur in an annealing twin in a somewhat random manner.

Since the NTB is suitably oriented for glide motion, the results indicate that an explanation is required whereby these boundaries are rendered immobile at low temperatures but are mobile at about room temperature and above. When one examines the nature of the atomic misfit at NTB's on the basis of a hard sphere model, it becomes apparent that the mismatch is rather severe. It is, in fact, necessary that in a \(<110>\) direction, for example of the parent lattice, at least one atom in a twinned orientation must be omitted, or severe compression results. It seems likely that during formation of a NTB that atoms would indeed be omitted under the circumstances by which these boundaries are formed, i.e., the Fullman-Fisher mechanism illustrated...
in Figure 2. Furthermore, in the case of alloys, the NTB's should be particularly favorable sites for the segregation of solute elements, especially in the case of solutes having substantial size factors. Conceivably then, atoms could be either omitted or "extra" atoms added during the growth process. If such "fitting" of atoms into favorable sites does occur during boundary formation, it should have the effect of inhibiting glide motion of the dislocations.

5.3 Three-Dimensional Geometry of Annealing Twins
The results of the twin profile determinations are plotted in Figures 17 through 22, Appendix A. Figures 17 and 18 include a series of photomicrographs taken at successive depths below the initial surface. The depth in microns below the initial surface is recorded in parentheses under each photomicrograph, and the small letters correspond to those on the plots.

5.3.1 NTB's Appear in the Form of Loops in the Crystal
NTB's appear in the form of loops in the crystal, and support Votava and Berghezan's (1959) proposal that annealing twins are bounded by partial dislocation loops. Implicit in these results is that NTB's are not, in general, planar. It seems likely that the planar NTB's observed by Dash and Brown (1963) were a result of the thin foils necessary in their work. Assuming that NTB's consist of an array of partial dislocations, the NTB must be in the form of dislocation loops. It should not be surprising, however, if these loops tend to align along certain crystallographic directions, i.e.; along crystallographic potential valleys after long annealing treatments. Indeed, the planar boundaries observed by Dash and Brown were only seen in the final stages
of growth. It seems likely that such boundaries would be apparent only over relatively small distances, and where the NTB intersects a top and bottom surface of a thin foil, it would be expected in a number of cases to be planar, or approximately so, due to the line tension associated with dislocations.

In summary, NTB's are generally high energy boundaries as evidenced from their chemical reactivity, thermal etching characteristics, and their retarding effect on migrating grain boundaries, and extend down through a crystal in the form of loops. In well-annealed specimens these loops might be expected to lie along certain low energy crystallographic directions, probably only over relatively small distances. Contiguous partial dislocations, however, do interact to give short and approximately straight line segments (microscopically) in directions approximately perpendicular to the coherent twin boundaries.

5.3.2 Annealing Twins were Frequently Observed to Contact Grain Boundaries and Other Twin Boundaries Below the Initial Surface

Frequent interactions were observed between twins and grain boundaries and between twins and other twins, as illustrated in the photomicrographs and twin profile plots. Out of 11 twins appearing in the photomicrographs of Figures 17 and 18, only one twin is completely free of any boundary interactions, and it is likely that this twin experienced similar interactions above the plane of polish. Out of a total of 21 twins observed in this manner (including those appearing in the photomicrographs along with the twin being measured) only two showed no interactions with other boundaries. These two, however, were quite shallow, and as stated above, probably experienced such interactions above the plane of polish.
The three-dimensional character of annealing twins and of the processes taking place during twin production, growth, and disappearance have not been generally appreciated. These results show the enormous influence exerted on twin stability and the resulting twinning frequency by twin-grain boundary and twin-twin pinning interactions. These interactions would account to some extent for the discontinuous nature of NTB migration. In a well-annealed specimen it appears that only rarely are twins completely free of all boundary contacts. It appears, therefore, that interactions with other boundaries is an important factor in the retention of high twinning frequencies, and indeed, for the retention of any twins at all at large grain sizes.

5.3.3 Annealing Twin Boundary Interactions With Grain Boundaries Resulted in Distortions in the NTB's

Figures 17 and 18 both indicate that twin-grain boundary interactions have the effect of distorting the NTB loops (partial dislocation loops). It is not possible to ascertain from these figures whether the twins were in the process of dissociating from the grain boundaries or were being attracted to them. Either is considered to be a possibility, depending upon the relative interfacial free energies involved. In both possibilities the interaction would affect NTB migration. If, for example, the twin plotted in Figure 18 were to be observed on a polished surface at some point above the twin-grain boundary intersection, and the twin was in fact being attracted to the grain boundary, twin elongation might be observed on the surface, as was suggested in Section 5.1.1.
5.3.4 Apparent Twin Shape is Influenced by the Orientation of the Twin with Respect to the Plane of Observation

The dislocation model of annealing twins explains why annealing twins are occasionally observed that apparently have curved "coherent" boundaries. Figure 23, Appendix A, is a schematic illustration of how the apparent twin shape can change with its orientation with respect to the plane of observation. Figure 23a is the normally observed twin shape. Figure 23b is possible when the plane of observation does not intersect both coherent twin boundaries. Several examples of this twin shape were observed during the twin profile determinations. One such example is shown in Figure 17c. Figure 23c is only possible when neither coherent twin boundary is intersected by the plane of observation. The resulting apparent twin shape would probably be difficult to recognize and likely would not be distinguishable from a grain during a casual observation. In addition, etching contrast would probably be small. If such a possible twin shape were located, it would be a simple matter to orient the grain and determine if the crystallographic relationships were consistent. No photographic evidence of this latter twin shape can be presented at this time; however, reflection on past observations by one of the authors (JRH) suggests that such twin shapes have been seen (though rarely) but that the significance was not appreciated at the time.

These twin shapes indicate further that the dislocation loop model of annealing twins is correct.

5.4 Segregation of Solute Elements to Twin Boundaries

The data from several representative potential probe traverses are presented in Figures 24 through 28, Appendix A. The scales used
all plots are the same for ease of comparison. The absolute voltages measured were not given since they varied from one twin area to the next depending upon the gross position of the twin in the sample. The voltages measured have been normalized to relative values since only the change in voltage from point to point is of significance. Similarly, the position of the probe point on the sample, as shown by the abscissa in each plot, is relative only to the specific area being probed.

The following results were obtained from this part of the investigation.

5.4.1 A Linear Change in Resistivity was Observed in a Potential Probe Traverse Across a Single Crystal of Copper

Figure 24 is a plot of a potential probe traverse across part of a large single crystal of 99.99 percent copper. The crystal was in a large sheet (15 mm x 50 mm x 0.4 mm thick) of copper rolled and annealed as described in Section 3.4. The measurements shown in Figure 24 were obtained in the center of this crystal at least five millimeters from a twin or grain boundary. The single crystal extended completely through the specimen and was clearly visible from the other side. Thus, the measurement made could be assumed to be in a semi-infinite single crystal of copper. With the exception of a few points, the plot shows a nominally linear change in potential, in accordance with Ohm's Law, as the probe measurements progress across the specimen.

5.4.2 A Potential Drop was Observed at Coherent Twin Boundaries in Both Copper and 90-10 Brass

Figures 25 and 26 are plots of potential probe traverses across coherent twin boundaries in a pure copper specimen and in a 90-10
commercial brass. In each case there is a marked potential drop at
the twin boundary. This may be a manifestation of the energy anomaly
associated with a twin boundary in an otherwise perfect lattice. Since
this occurs in both specimens, this potential drop cannot be associated
with solute segregation. The rise in potential after the traverse
passes through a twin boundary is the result of current short circuiting
around the ends of the twin boundary. Otherwise the rise in potential
would indicate an area having a negative resistance. This potential
variation at coherent twin boundaries was observed on several samples,
and the measurements obtained were reproducible.

5.4.3 A Difference in Behavior was Observed at NTB's in Copper
and 90-10 Commercial Brass

Potential probe traverses were made across NTB's in copper and
90-10 brass samples. Figures 27 and 28 are examples of such traverses.
In the case of the traverse through the twin in copper a definite drop
in potential was observed just before the twin with a sharp rise at the
twin boundary. Thus, an area of high resistivity is present to the
right of the twin with a low resistivity point at the twin boundary. A
negative resistance at the twin boundary is not possible, and the only
explanation for the rise in potential at the twin boundary is that the
current is short circuiting through the boundary from some other part
of the specimen. The indication is, however, that the NTB is a low
resistivity region even though the material on either side is of higher
resistivity. These effects must all be due to crystallographic
variations of the lattice since no segregation can take place in the
copper specimen (unless some results from the minor impurities in the copper).

By comparison, the potential traverse through the NTB in brass describes only a sharp change in slope at the boundary. The slope to the left of the twin boundary is small indicating a region of comparatively low resistivity. On the right of the boundary the increased slope indicates a higher resistivity region. There are no sharp variations in the traverse as were present in other potential traverses.

The brass specimens had a grain size of about one millimeter. This made the ratio of the grain size to the specimen thickness about three. Therefore, it would be possible for the twin boundary being observed in the brass specimen not to penetrate clear through the specimen. The twin boundary may have changed direction immediately below the viewing plane (see Section 5.3). It was not possible to locate a twinned grain on both sides of the sample sheet because of this small grain size.

The copper specimens all had grain sizes of one centimeter or more. The ratio of grain size to specimen thickness was about 30. Traverses made on the copper can be assumed to be made in a semi-infinite grain, whereas the same cannot be said about the brass samples. Clearly, then, it is desirable to make probe measurements on brass specimens which have much larger grains than the ones reported here. This could not be done in the course of this investigation. There is an apparent limit to the grain size of brass samples which may be controlled by the sample thickness. Using larger samples, large
grains could be obtained in brass samples. These thicker samples, however, do not lend themselves to potential probe measurements since the greater thickness precludes the consideration of samples being semi-infinite sheets.

5.5 Effect of Time and Temperature on Annealing Twin Frequency

The following results were obtained in this part of the investigation.

5.5.1 The Frequency of Annealing Twins can be Altered by Selective Heat Treatment Schedules

Twin frequency curves for copper are given in Figure 29, Appendix A, and the data are tabulated in Table III, Appendix B. The results clearly demonstrate that the frequency of occurrence of annealing twins in copper can be altered by selective heat treatment schedules. The short, isothermal-bath heat treatments resulted in higher twinning frequencies, which decreased with increasing temperature, in the range 650°-830°C. Utilizing temperatures such that desired grain sizes can be achieved in times of the order of a few seconds result in much faster rates of grain growth. In all cases, heat treatment times longer that 1-1 1/2 minutes resulted in a decrease in the twinning frequency for the temperature employed.

The effectiveness of short times in increasing twinning frequencies, as evidenced by the rapid decrease in twinning frequency at the longer times of heat treatment, is thought to be a result of both an increase in the twin production rate and a decrease in the rate of twin annihilation by NTB migration, with the latter probably playing the greater role. The increase in twin production rate is a result of the
rapid rates of grain growth and probably also indicates that the
twin producing mechanism is more prolific at smaller grain sizes, as
might be expected since a greater number of migrating three-grain
junctions would be available for twin production. Evidence that grain
growth rate may be effective in altering twinning frequency may be seen
by comparing the grain growth curves of Figure 30, Appendix A, with the
twin frequency curves of Figure 29. The decrease in the number of twins
annihilated by NTB migration is probably a result of the discontinuous
nature of NTB migration and an increased number of twin - twin pinning
interactions.

The data also show that, for the short heat-treatment, increased
temperatures are effective in decreasing the twinning frequency. Since
the grain growth rate increases with temperature, this effect of
temperature is attributed to an increase in twin annihilation by NTB
migration (as opposed to fewer twins forming) with increasing
temperature, i.e., NTB migration is a thermally assisted process. In
addition, the higher temperatures should promote twin - grain boundary
dissociation and thus contribute further to twin annihilation.

An example of the profusion of twinning obtained in short-time
heat-treated specimens is shown in Figure 31, Appendix A. This specimen
was heat treated for 12 seconds at a bath temperature of 750°C and water
quenched. One additional point should be made. The temperatures
reported are those of the molten salt bath. The specimen temperature,
therefore, cannot be taken as that of the bath at the shortest times
involved.
To summarize, time appears to be the most effective parameter in increasing twinning frequency in copper, and its effect is thought to be manifested largely in the suppression of NTB migration for short times. Twinning frequency decreases with increasing temperature and is a result of an increase in thermally assisted NTB migration. The increased temperatures probably also promote twin-grain boundary dissociation.

5.5.2 The Twinning Frequency of Tough Pitch Copper is Significantly Lower Than That for 99.99 Percent Copper

The twinning frequency of tough pitch copper is considerably lower than that of 99.99 percent copper, and may be attributed to the effects of impurities in the tough pitch copper. No preferred orientations could be detected by X-ray back-reflection pinhole camera techniques; however some differences may exist between the 99.99 percent copper and the tough pitch copper since the former was cold-rolled to 50 percent reduction in thickness and the latter to 70 percent reduction in thickness. Further investigation is being pursued in this area.

5.5.3 Triangular Etch Pits Were Observed in the Short-Time Heat-Treated Copper Specimens

Triangular etch pits whose sides were parallel to coherent twin boundaries were apparent in the 99.99 percent copper specimens as shown in Figure 32, Appendix A. The etch pits were obtained with a 2:1 mixture of NH$_4$OH:H$_2$O$_2$. At longer times of heat treatment (greater than about two minutes) the pits were no longer produced upon etching. Etch pits were usually triangular, though sometimes slightly distorted or
elongated. The pits appeared to be randomly distributed and of variable size, and were never observed to be in a sub-boundary configuration.

A tentative explanation of the origin of the pits may be similar to that discussed by Young (1961). Impurities (< 0.01 percent) may have collected at dislocation sites, which increased with time at temperature. When the "Cottrell Atmosphere" reached a critical value, the etch pits were no longer formed due to a decrease in the dislocation core energy and the strain energy in the immediate vicinity of the dislocation.

5.6 Effect of Specimen Size on Annealing Twin Frequency

The results of the twin frequency determinations in three series of commercial 70-30 brasses are tabulated in Table IV, Appendix B, and plotted in Figure 33, Appendix A. The results indicate that specimen size, within the limits of sizes used in this investigation, has little or no affect on twinning frequency. The differences noted at the smallest grain sizes may be attributable to the differences in extent and type of deformation. The rod specimens were extruded and cold-drawn (from 3/4 to 5/8 inch diameter), while the sheet material had been cold-rolled and annealed (0.05 inch thick as received) and cold-rolled to 20 percent reduction in thickness.
6.0 CONCLUSIONS

The following conclusions have been drawn from the results of this investigation.

6.1 Direct Microstructural Observations Using an Evacuated Heating Stage

a) In general, noncoherent twin boundaries are relatively high energy boundaries.

b) Annealing twins may be broadened or lengthened by grain boundary migration when the appropriate twin boundary coincides with the migrating grain boundary.

c) Noncoherent twin boundaries may migrate in either direction parallel to coherent twin boundaries.

d) Noncoherent twin boundary motion occurs stepwise in a discontinuous manner.

e) The lowering of interfacial free energy can occur at twin-grain boundary intersections in accordance with the Fullman-Fisher hypothesis.

f) Annealing twins can probably dissociate from grain boundaries in directions parallel to coherent twin boundaries.

g) Twin-grain boundary intersections inhibit grain boundary migration.

h) One mechanism of twin elongation may be a result of twin-grain boundary interaction.

i) Significantly fewer twins are found in Cu - 2 a/o Ag at relatively large grain sizes than in pure Cu, Cu - 2 a/o Sn, Cu - 2 a/o Si, or Cu - 2 a/o In.

6.2 Effect of Applied Mechanical Loading on Annealing Twin Boundaries

a) Motion of noncoherent twin boundaries can be thermo-mechanically induced in Cu and Cu - 2 a/o Al under conditions of severe abrasive deformation.
b) Thermo-mechanically induced motion results in a lenticular noncoherent twin boundary configuration resembling that of mechanical twins.

c) A major difference in the properties of noncoherent twin boundaries of mechanical and annealing twins arises from the mechanisms of formation.

6.3 Three-Dimensional Geometry of Annealing Twins

a) Noncoherent twin boundaries do not, in general, coincide with any crystallographic plane but form loops through the crystal.

b) Only rarely are annealing twins free of all boundary interactions in well-annealed specimens.

c) Annealing twin stability and resulting twin frequencies are greatly influenced by interactions with grain boundaries and other twins.

d) Intersections with grain boundaries distorts the noncoherent twin boundary loops.

e) Apparent annealing twin shape, as observed on a two-dimensional surface, depends upon the orientation of the twin with respect to the plane of observation.

6.4 Segregation of Solute Elements to Twin Boundaries

a) There is an alteration of the bulk resistivity at both coherent and noncoherent twin boundaries.

b) Solute segregation could not be detected in a 90-10 commercial brass.

c) The potential probe measuring technique can be used to determine minute variations in the bulk resistivity of copper and a commercial copper-zinc alloy.

d) It is difficult to form large twinned grains suitable for potential probe measurements in thin samples of commercial copper-zinc alloys.

6.5 Effect of Time and Temperature on Annealing Twin Frequency

a) The twinning frequency in copper indicates some dependence on grain growth, with faster growth rates producing more twins/grain.
b) Twinning frequency as a function of grain size experiences a maximum value and decreases at large grain sizes.

For tough pitch copper:

c) The short heat-treatment times had little effect on the twinning frequency.

d) The lower twinning frequency of vacuum-melted tough pitch copper from that of 99.99 percent copper is attributed to impurities.

For 99.99 percent copper:

e) Twinning frequency increases with decreasing temperature in the range 650\(^\circ\)C-830\(^\circ\)C for short heat treating times.

f) Decreasing the time of heat treatment (at sufficiently high temperatures) to the range of a few seconds to a few minutes is effective in increasing the twinning frequency.

g) Twin annihilation by noncoherent twin boundary migration is suppressed for short heat treatment times.

h) A decrease in twinning frequency at large grain sizes is to be expected for at least two reasons: (1) Dissipation of the twin producing mechanism, and (2) increased extent of twin annihilation at the longer times involved.

6.6 Effect of Specimen Size on Twinning Frequency

a) Specimen size has little or no effect on twinning frequency in 70-30 commercial brass.
REFERENCES


Figure 1. Schematic illustration of twin formation resulting from a decrease in the interfacial free energy of grain boundaries. Grain boundaries with high interfacial free energies are represented by wide lines. Diagram after Fullman and Fisher (1951).
Figure 2a. Schematic illustration of noncoherent twin boundary formation by twin - grain boundary dissociation in a direction parallel to coherent twin boundaries.

Figure 2b. Schematic illustration of the formation of a noncoherent twin boundary after twin formation at the migrating junction of three grains. The coherent twin boundary becomes tangent to the A/C boundary, and upon further grain boundary migration leads to the formation of a noncoherent twin boundary segment. Diagrams after Fullman and Fisher (1951).
Figure 3. Water-cooled, high frequency vacuum induction furnace used to melt and cast alloys.
Figure 4. Ingot cast in high purity graphite mold.
Figure 5. Specimen dimensions (in mm) for examination on vacuum heating stage.
Figure 6. Reichert Vacutherm heating stage mounted on metallograph for operation.
As cast ingot

A

Cold-pressed to 30% reduction

B

Machined to 1 1/8 x 1/4 x 3/32" 

C

Mechanically ground and polished compression, tension, and one neutral axis side of specimen

D

Annealed three hours in argon at 950°C to produce large grains and twins

E

Repolished and etched

F

Loaded in 3 point bending in Instron testing machine at liquid nitrogen temperature

G

Microexamination of bent specimen

H

Lightly re-etched specimen to expose boundary movements

I

Repeat steps G through I

Figure 7. Flow diagram of procedure adopted in bending tests of Cu, Cu-2 a/o Al, and Cu-2 a/o Ag specimens.
Figure 6. Flow diagram of procedure adopted in determination of twin profiles.
Figure 9. Measuring circuit diagram
Figure 10. Grain boundary migration and noncoherent twin boundary migration in a-Cu - 2 a/o Si alloy as observed on the vacuum heating stage. 150x.
(a) $t = 0; \ T = 840^\circ C$

(b) $t = 14'; \ T = 840^\circ C$
(c) $t = 39'$; $T = 880^\circ C$

(d) $t = 51'$; $T = 880^\circ C$
(e) Cooled to room temperature, reheated; 
$t' = 0; \ T = 910^\circ C$

(f) $t' = 15'; \ T = 950^\circ C$
(g) \( t' = 59' \); \( T = 950^\circ C \)
Figure 11. Noncoherent twin boundary migration in a Cu - 2 a/o Si alloy as observed on the vacuum heating stage. 64x, enlarged three times in reproduction.
(a) $t = 0; \ T = 760^\circ C$

(b) $t = 25'; \ T = 840^\circ C$
(c) $t = 42' \quad T = 840^\circ C$

(d) $t = 1 \text{ hr } 5' \quad T = 880^\circ C$
(e) \( t = 1 \text{ hr 12'} \); \( T = 880^\circ C \)

(f) Cooled to room temperature, reheated; \( t' = 0 \); \( T = 860^\circ C \)
(g) \( t' = 16' \); \( T = 910^\circ C \)

(h) \( t' = 40' \); \( T = 950^\circ C \)
Figure 12. Grain boundary migration, twin broadening, and noncoherent twin boundary motion in copper as observed on vacuum heating stage. a-e, 150x; f, 64x, enlarged three times in reproduction.
(a) $t = 0; \ T = 850^\circ C$

(b) $t = 22'; \ T = 850^\circ C$
(c) $t = 55'$; $T = 910^\circ C$ last 5 minutes

(d) Cooled to room temperature; reheated to $910^\circ C$
(e) Cooled to room temperature; reheated to 980°C

(f) Cooled to 910°C, held 30 minutes
Figure 13a. Attraction between a noncoherent twin boundary and a grain boundary beneath the polished surface, resulting in twin elongation on the surface. Similar effects would be possible at twin-twin intersections.

Figure 13b. Effect of a free surface on twin elongation and subsequent reversal of motion.

Figure 13c. Apparent twin formation (as observed on the specimen surface) by expansion of the noncoherent twin boundary loop due to twin-grain boundary attraction.
Figure 14. Lowering of interfacial free energy as a result of the twinned orientation. 64x, enlarged three times in reproduction.
(a) $t = 0; \ T = 845^\circ C$

(b) $t = 8'; \ T = 1020^\circ C$
(c) $t = 25' \; ; \; T = 1030^\circ C$
Figure 15. Grain boundary and apparent twin boundary migration in Cu - 2 at. % Si alloy as observed on the vacuum heating stage at 950°C. 64x, enlarged three times in reproduction.
(a) $t = 0; \ T = 950^\circ C$

(b) $t = 3^t; \ T = 950^\circ C$
(c) \( t = 8' \); \( T = 950^\circ C \)

(d) \( t = 23' \); \( T = 950^\circ C \)
(e) $t = 28'$; $T = 950^\circ C$
Figure 16. Thermo-mechanically induced noncoherent twin boundary motion in copper and Cu - 2 a/o Al.

a - Cu - 300x; b through e - Cu - 650x;
f - Cu - 2 a/o Al - 225x.
Figure 17. Twin profile determination in a copper crystal. The twin profile is plotted along the coherent boundary A-B. The small letters on the accompanying photomicrographs (300x) correspond to those on the plot. The figures on the photomicrographs are depth in microns below the initial surface.
Figure 18. Twin profile determination in a copper crystal. The twin profile is plotted along the coherent boundary A-B. The small letters on the accompanying photomicrographs (300x) correspond to those on the plot. The figures on the photomicrographs are depth in microns below the initial surface.
Figure 19. Twin profile determination in a copper crystal. The twin profile is plotted along a coherent twin boundary.

Figure 20. Twin profile determination in a copper crystal. The twin profile is plotted along a coherent twin boundary.
Figure 21. Twin profile determination in a copper crystal. The twin profile is plotted along the coherent boundary A-B. The profile of twin 2 is plotted in Figure 22.

Figure 22. Twin profile determination in a copper crystal. The twin profile is plotted along the coherent boundary A-B. The profile of twin 1 is plotted in Figure 21.
Figure 23. Apparent twin shape as influenced by the twin orientation with respect to the plane of observation.
Figure 24. Potential traverse across a single crystal of copper
Figure 25. Potential traverse across a coherent twin boundary in copper.
Figure 26. Potential traverse across a coherent twin boundary in 90-10 brass
Figure 27. Potential traverse across a noncoherent twin boundary in copper
Figure 28. Potential traverse across a noncoherent twin boundary in 90-10 brass
Figure 29. Twinning frequency in copper for short heat treatment times. The time of heat treatment is shown near each point. Data from Mack, et. al., (1962) are for tough pitch copper which was heat treated for one hour at various temperatures.
Mean Twins/Grain

- O 650°C 99.99% Cu
- □ 750°C 99.99% Cu
- ◇ 830°C 99.99% Cu
- Δ 750°C Tough Pitch Cu
- ▼ Data from Mack, et. al., (1962)

Mean Grain Diameter (mm)
Figure 30. Mean grain diameter versus time of heat treatment for copper.
Figure 31. Profusion of twinning in a copper specimen heat treated 12 seconds at 750°C.

Figure 32. Triangular etch pits in a copper specimen heat treated 12 seconds at 750°C.
Figure 33. Twinning frequency in 70-30 commercial brasses. All specimens were heat treated for one hour at temperatures indicated in Table IV, Appendix B.
TABLE I
Detailed Chemical Analyses of Materials Used

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*Detailed Analyses not available*
TABLE II
Chemical Analyses of Copper-Base Alloys

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All elements determined by wet analysis with no element taken as remainder.
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T/G  Mean Twins per Grains
T/TG Mean Twins per Twinned Grains
TF/TG Mean Twin Families per Twinned Grains
### TABLE IV

Twin Frequency Data for 70-30 Commercial Brasses

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**Mean Twins per Grain**

**Mean Twins per Twinned Grain**

**Mean Twin Families per Twinned Grain**