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The effect of annealing on Neumann bands.

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UNIVERSITY OF LOUISVILLE

THE EFFECT OF ANNEALING ON NEUMANN BANDS

A Thesis

Submitted to the Faculty
of the Graduate School
of the University of Louisville
in Partial Fulfillment
of the Requirements
for the Degree of

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

Alex B. Davidson

1940

THE EFFECT OF ANNEALING ON NEUMANN BANDS

Alex B. Davidson

Approved by the Examining Committee.

Director _____

May 22, 1940

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ABSTRACT

The exact structure of Neumann bands is controversial, and there is some doubt as to their behavior under different conditions. This thesis is an investigation of the action of Neumann bands, under different annealing conditions, in an attempt to add to the present knowledge of their structure.

Samples of Swedish iron 0.08 % carbon, containing Neumann bands, were annealed at various temperatures in a small tube furnace. Photomicrographs were made of each sample, before and after annealing, so that the actions of the Neumann bands could be observed.

There was no unique correlation between Neumann bands and recrystallization, and the behavior of the Neumann bands was similar to that of the grain boundaries.

INTRODUCTION

When subjected to mechanical shock low carbon steel and relatively pure iron develop a unique change in grain structure. This change appears on a polished and etched specimen in the form of straight dark lines or bands which are designated as "Neumann Bands". The exact nature of this change is unknown although several theories persist. The purpose of this investigation was to determine the effect of various annealing temperatures on existing Neumann bands in a Swedish iron specimen. Information of this type, if sufficiently complete and correlated, will eventually lead to an exact knowledge of the nature of this structural change.

HISTORICAL

Neumann bands are generally believed to be a form of crystal twin.(10,2) The structure attributed to a twin is, in general, the shifting of a crystal section with reference to its original orientation. It is in other words a type of internal buckling.(2,7) When the reorientating has been effected, the section stands structurally as a mirror image to the original form and is symmetrical with respect to the original twinning plane.(7)

It is recognized that there are two types of twins, annealing twins and mechanical twins. The former are found in face-centered, cubic crystal lattices exclusively and the latter in body-centered lattices. They are considered to be of a similar structural nature differing only in their space lattice relationships.(4,2) Annealing twins are formed by annealing a cold worked specimen while mechanical twins are formed by cold deformation alone.(10)

Neumann bands are held to be either mechanical twins(4,2) or a structural change resulting from localized deformation brought about by rapid application of heavy stress. Rosenhain and McMinn(10) believe the latter theory. They further maintain that Neumann bands are sections through lamellae or thin plates and not of the nature of thin rods.

Neumann bands are definitely an indication of strain in the metal, and as this internal buckling extends through the whole grain, they can not be removed by re-

polishing and reetching.(10) When a material is in such a state of strain annealing may be expected to produce a recrystallization of the grains. This recrystallization may be followed by grain growth, but it is doubtful whether an unstrained metal or a metal in the cast state will exhibit these phenomena.(11,9,8)

THEORETICAL

Since Neumann bands are known to be an indication of strain, it is probable that the grains showing them have been more severely strained than adjacent grains which have no Neumann bands. Also since the internal buckling which forms Neumann bands was caused by this severe stress, it is logical to believe that the adjacent grains, free from Neumann bands, are in a state of internal stress greater than that of the buckled grains, since they have made no rearrangement to relieve their stress. Stress relief may have taken place in these unmarked grains by slip bands which would not be visible because the polishing operation removed them.(10) If this were true there would probably be no great stress differentiation of the grains.

Assuming, however, that the adjacent grains are in a state of internal stress greater than the marked grains, they would be expected to recrystallize or grow before the grains with Neumann bands, for it is known that the greater the previous deformation of a metal the lower will be its recrystallization temperature.(11,8)

Recrystallization upon annealing relieves the internal stresses in the metal and tends to remove the distortion caused by mechanical deformation with the formation of new equi-axed grains.(3) The dimensions of these new grains are affected by the following factors; (1) amount of cold deformation, (2) size of distorted grains, (3) temperature

at which deformation was produced, (4) annealing temperature, (5) length of time at annealing temperature and (6) the rate of cooling from annealing temperature.(10,8)

For most iron and steel there is a minimum recrystallization temperature, below which grain growth will not take place. This temperature is the Ar-1 line in the iron-iron carbon equilibrium diagram(see fig. 1). However Stead showed that there was a range of low carbon steel, 0.04 to 0.12 % carbon, which recrystallized below this Ar-1 line and as low as 450 degrees Centigrade. He also showed that for each annealing temperature in this range there was a definite critical strain value which would produce an abnormally large grain size in the specimen.(1,11,12,6) Since the Swedish iron used in this investigation contained 0.08 % carbon it should have a minimum recrystallization temperature somewhere between 450 degrees C and the upper critical line at 900 degrees C. At some temperature in this range it might show abnormal grain size.

Rosenhain (11) states that the recrystallization of strained ferrite might be explained on the assumption that straining results in the production of amorphous cement or nuclei, which recrystallize on heating below the critical range. (11) If this be true, grain growth would be expected to start at Neumann bands since their buckling

action produces interfaces similar to a grain boundary. It is also known that recrystallization may start inside a grain from nuclei or fragments formed by cold strain, which might not be seen under the microscope.(5,8)

TEMPERATURE DEGREES CENTIGRADE

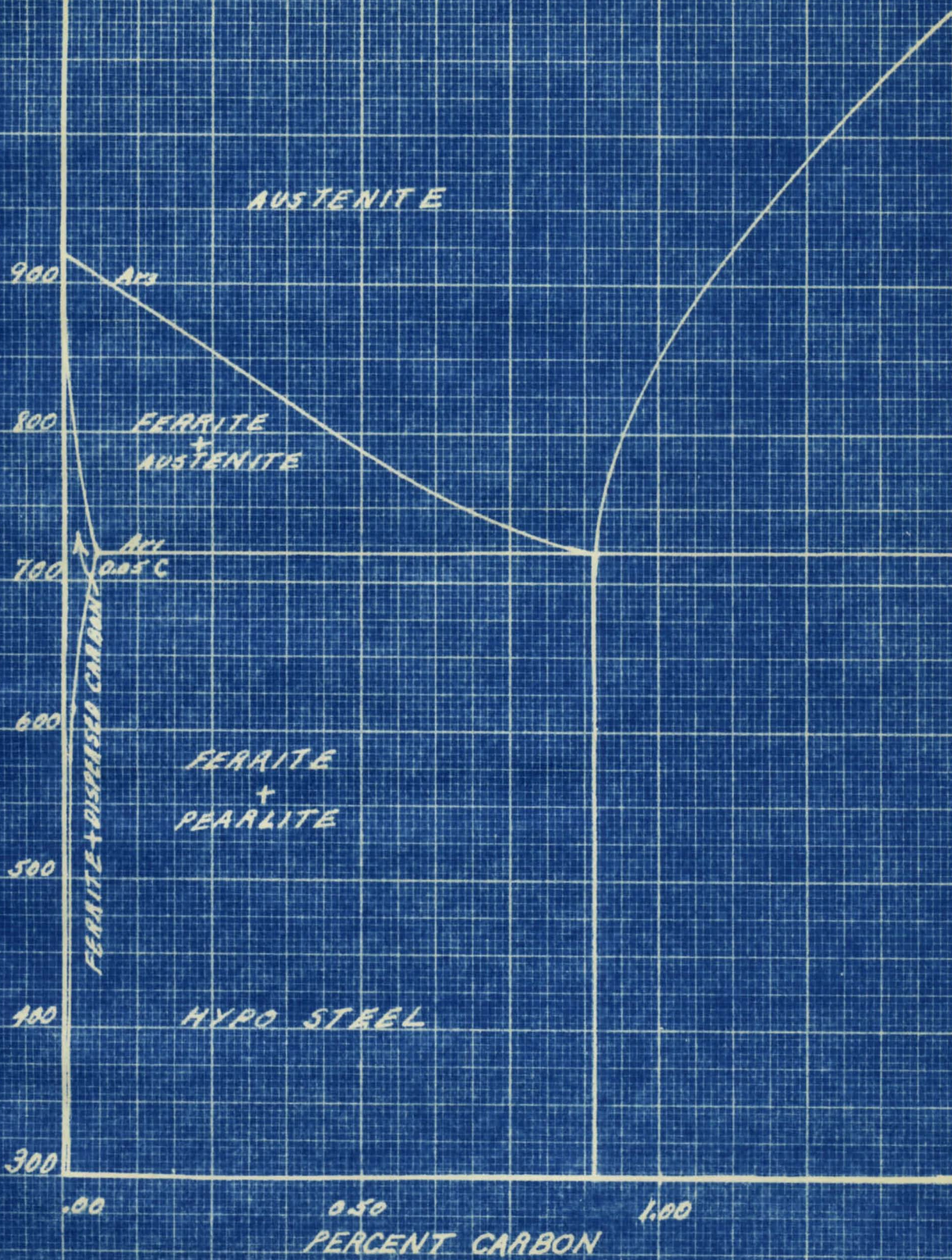


FIG. 1. THE IRON-IRON CARBIDE EQUILIBRIUM DIAGRAM

EDUARD DIEZGEN CO. PHOTODUPLICATION SERVICE, U.S.A. 20 X 20 PER INCH

APPARATUS

A small tube furnace was constructed for annealing the samples of iron. The furnace had a heating element of Chromel C wire wound around a zirconia combustion tube. This was set in a cylindrical shell and the annulus packed with kieselguhr. The maximum temperature for the furnace was 1000 degrees C on a 110 volt circuit, and the temperature was controlled by an adjustable rheostat.

The ends of the combustion tube were necked down, one end piece being detachable with a tight fitting rubber sleeve as a connector. Rubber tubing connected the heating tube to a tank of hydrogen so that a stream of reducing gas could be circulated over the annealing specimen.

A Chromel-Alumel thermocouple was laid lengthwise in the combustion tube. The cold end, which protruded from one end of the tube, was sealed to the neck of the tube with paraffin to prevent escape of the hydrogen gas. The junction was at the center of the tube where the sample would be placed. The EMFs of this thermocouple were read on a standard Leeds and Northrup portable potentiometer.

Photomicrographs of specimens were taken with a Leitz Micro Metallograph "MM-1", 100 diameters magnification, using a 23 mm. objective and a 6x Periplan ocular. A camera bellows extension at 41 cm. was used. Illumination was by an A.C. arc light and a prism illuminator. The speci-

men stage was equipped with movable markers and a calibrated scale so that the spot under observation could be found again if the sample was removed from the stage.

PROCEDURE

The metal specimens were cut from a $\frac{1}{2}$ inch bar of hot rolled Swedish iron, 0.08 % carbon, and were about $\frac{1}{2}$ inch long.

The sudden impact stress, necessary for the formation of Neumann bands, was effected by means of hammer blows on each sample. The specimens were then ground, polished according to standard metallographic practice, and etched in a 5 % nital solution.

A sample was placed on the stage of the micro-metallograph and a spot found which was suitable for further treatment and examination. All scale and holder notations were recorded for further identification procedures, and a photomicrograph was made.

The specimen was next placed in the center of the furnace and a slow stream from a cylinder of compressed hydrogen passed through the annealing tube. The hydrogen tended to prevent oxidation of the polished surface of the sample at the annealing temperature. The furnace was heated to the annealing temperature, and maintained there for the correct period of time by means of the adjustable rheostat.

When the period of annealing had expired the furnace was shut off and allowed to cool. A temperature of approximately 150 degrees C was reached after 4 or 5 hours cooling. The hydrogen was then shut off and the ends of the

annealing tube sealed to prevent the entrance of oxygen. After further cooling to room temperature the sample was removed.

The majority of the specimens developed a thin, blue oxide coat while in the furnace. This coat appeared to outline new and larger grains overlying the original grain structure. A photomicrograph was taken of the spot previously photographed so that the oxide grain outline could be compared with the new grains brought out by repolishing and reetching. This photomicrograph was specifically for the purpose of correlating initial and final structures at the same spot.

The sample was then repolished, reetched, and the same spot reinspected. The new grain formation, in most of the samples, conformed to the outlines of the oxide coat. A photomicrograph was taken completing the series for that sample.

In the case of some specimens, the oxide coat developed beyond the point of grain visibility and a reduction had to be accomplished. This was done with a dilute, acid, stannous chloride mixture and the sample was then handled similarly to those described.

DATA AND RESULTS

Figure 2 (page 19) is a photomicrographic series of a specimen annealed 1 hour at 950 degrees C.

The original grain size in this sample varies through wide limits as shown in plate A. The spot was on the edge of the specimen and this variation was probably due to the hot rolling process.

The three parallel Neumann bands at the bottom of plate A can be faintly seen through the oxide coat, in plate B. However there is no trace of them in plate C. The single band in the upper central portion of plate A can be traced in plate B and it also has disappeared in plate C.

The polishing scratches, which can hardly be seen in plate A, are brought out sharply in plate B by the anneal and even more so in plate C by the light repolish and re-etch.

The oxide coat in plate B has formed in the shape of the new grains in plate C as can be seen by comparing the two. The grain formation in the lower right hand corners of the two plates and the polishing scratches are the best identification marks.

The specimen shown in Figure 3 Page 20) was also annealed 1 hour at 950 degrees C.

In this series of photomicrographs the sample markers were not used, and after the specimen was repolished and reetched, the previously observed spot could not be

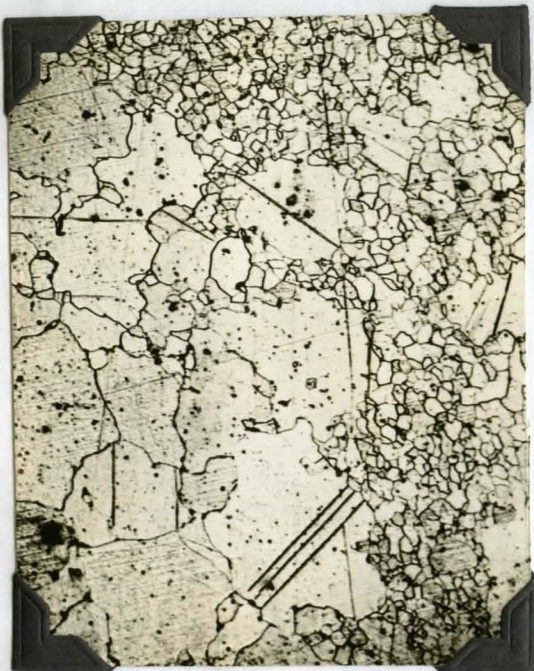


Plate A. Before anneal



Plate B. After annealing
for 1 hour

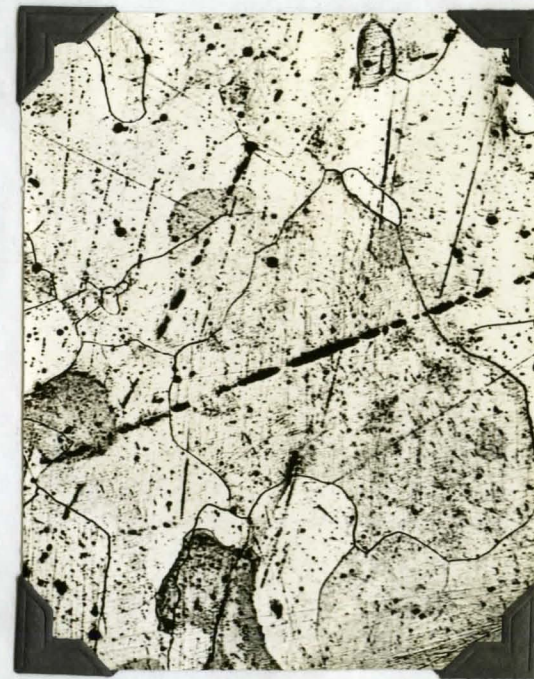


Plate C. Annealed specimen
repolished and re-
etched

Figure 2. Effect of Anneal at 950 degrees C, 100 x.



Plate A. Before anneal

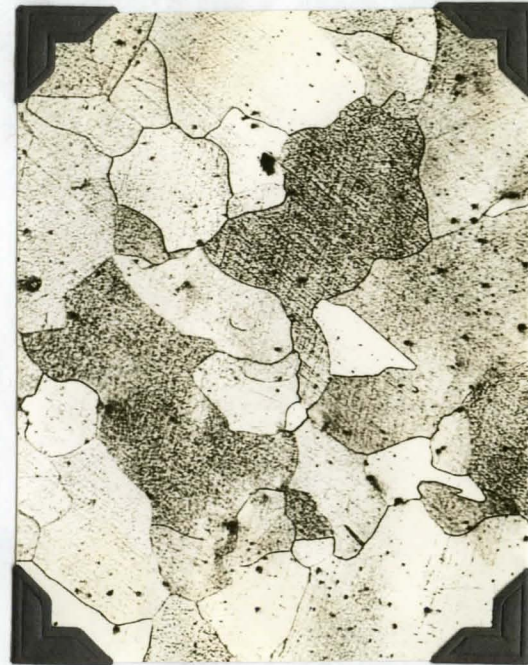


Plate C. Annealed specimen
repolished and re-
etched

Figure 3. Effect of Anneal at 950 degrees C, 100 x.

found. Plate C, therefore, merely shows the average size of the new grains.

The oxide coat was so dark after the anneal that a photomicrograph could not be taken.

This anneal was above the upper critical line and the series shows definite grain growth. A close examination of the entire specimen failed to show a single Neumann band, indicating complete removal by a 950 degrees C anneal although the original grains were not followed in the photomicrographic series.

Figure 4 (page 22) is a series of a sample annealed 1 hour at 850 degrees C. The position of the Neumann bands in this series can be followed, and it is seen that there is no trace of them in plate C. A comparison of plates B and C indicates that in some cases the new grains have formed directly across the Neumann bands. The bands in the upper right corner show this very well.

A comparison of plates A and C also indicates the reversal of relative grain sizes, i.e. the area of fine grains has become that of large grains and vice versa.

Figure 5 (page 23) is another series of a sample annealed 1 hour at 850 degrees C.

The Neumann bands in this series can be followed from plate A to plate B, but plate C shows no trace of them.



Plate A. Before anneal



Plate B. After annealing
for 1 hour



Plate C. Annealed specimen
repolished and re-
etched

Figure 4. Effect of Anneal at 850 degrees C, 100 x.



Plate A. Before anneal



Plate B. After Annealing
for 1 hour

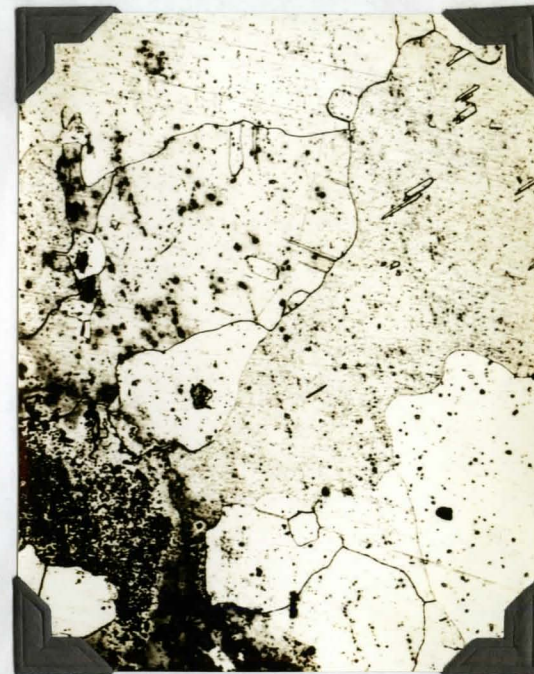


Plate C. Annealed specimen
repolished and re-
etched

Figure 5. Effect of Anneal at 850 degrees C, 100 x.

The large grain in the upper right corner has an orientation which appears to have no relation to the Neumann bands which were absorbed by it.

The average initial grain size of this series was finer than that of Fig. 3, and the final grains are much larger. This condition, however, has no bearing on this specific investigation.

The oxide grain outlines in plate B are good reproductions of the final grains in plate C.

Figure 6 (page 25) shows the effect of a 1 hour anneal 750 degrees C.

The Neumann bands in this series are grouped in the center section of the sample, indicating that this portion received the greatest stress. Plate C indicates a complete effacement of the Neumann bands.

The grain size in plate C is abnormally large, and it is likely that this degree of strain produced germination at the 750 degrees C annealing temperature.

The abnormal grains have followed the oxide outline, which can be seen by comparing the upper edge and lower right corner of plate B with the corresponding spots in plate C.

The sample shown in Figure 7 (page 26) was annealed 1 hour at 650 degrees C.



Plate A. Before anneal



Plate B. After annealing
for 1 hour

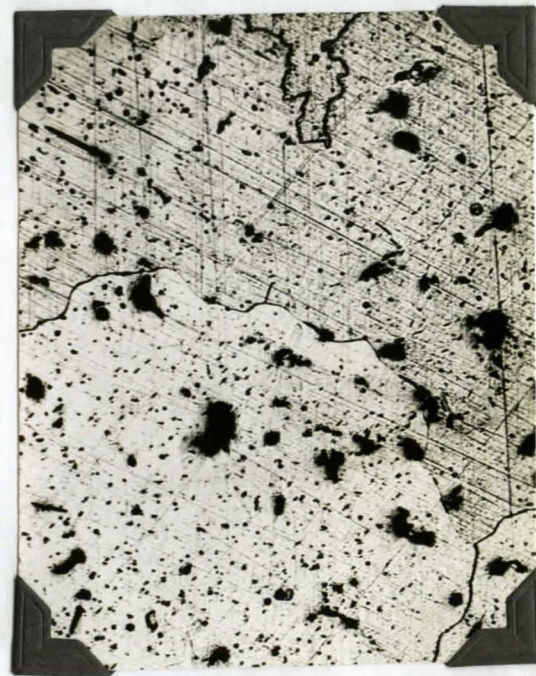


Plate C. Annealed specimen
repolished and re-
etched

Figure 6. Effect of Anneal at 750 degrees C, 100 x.



Plate A. Before anneal

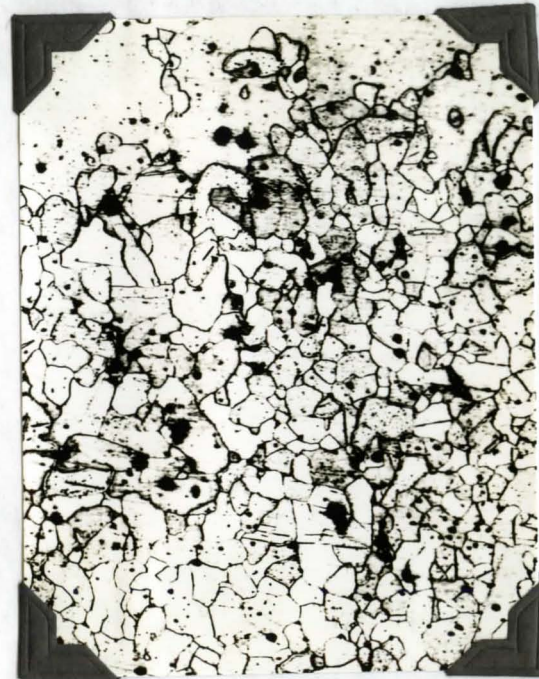


Plate C. Annealed specimen
repolished and re-
etched

Figure 7. Effect of 1 hour Anneal at 650 degrees C, 100x.

Annealing has not wholly removed the Neumann bands in this series. Most of the Neumann bands in the central portion of plate A extend from one grain boundary to the other, while in plate C they have been shortened and no longer reach to the boundaries. Some few scattered bands have been completely removed.

This 650 degrees C annealing temperature is apparently just high enough to start recrystallization. It is evident that the large grains at the upper edge are growing by feeding on the smaller ones. The Neumann in the upper right corner of plate A is being absorbed by the new large grain in that corner. The original crystal which held this band appears to be partially dissolved into the large grain.

This section was a longitudinal section of the bar. The oxide coat after annealing was very dense, and it could not be properly thinned down with the stannous chloride solution.

Figure 8 (page 28) represents the effect of a 3 hour anneal at 650 degrees C on a specimen.

The Neumann bands in this series are entirely removed by the annealing, and they appear to have had no effect on the final grain structure.

The oxide grain outline in plate B is poor, but the sharp pointed grain outline protruding from the right

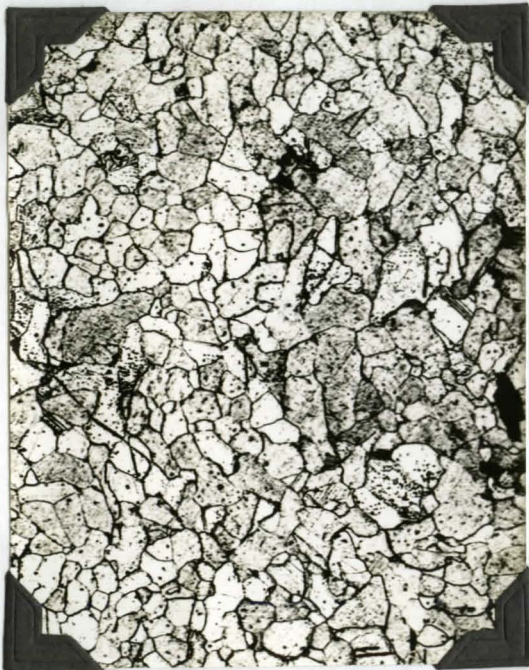


Plate A. Before anneal



Plate B. After annealing
for 3 hours

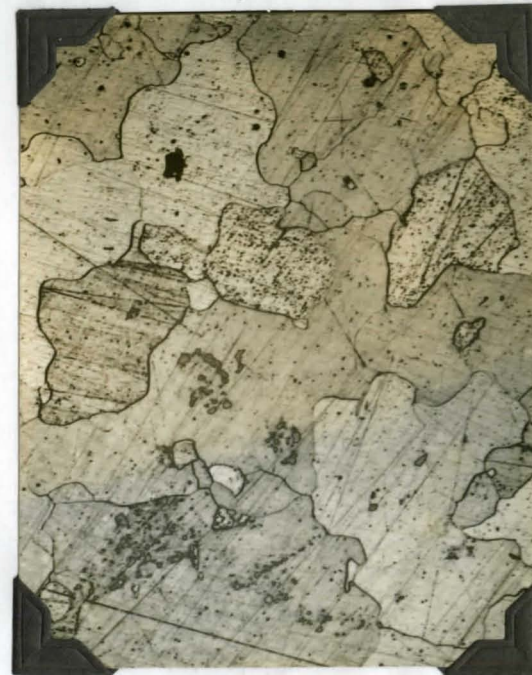


Plate C. Annealed specimen
repolished and re-
etched

Figure 8 Effect of Anneal at 650 degrees C, 100 x.

edge of plate B, about three fourths of the way from the top of the plate, can be seen as a grain in the same position on plate C. The relationship between oxide and final grain can also be seen in the upper right corner.

Although this anneal was the same temperature as that of Fig.6, the longer period of time enabled complete recrystallization and grain growth to take place in this series.

Figure 9 (page 30) is a photomicrographic series of a specimen annealed 3 hours at 550 degrees C.

The Neumann bands in this series were not removed although some of them seem to be shortened and thinned.

Plate B shows definite signs that grain growth is beginning, but the grains with the Neumann bands are not in any way unique.

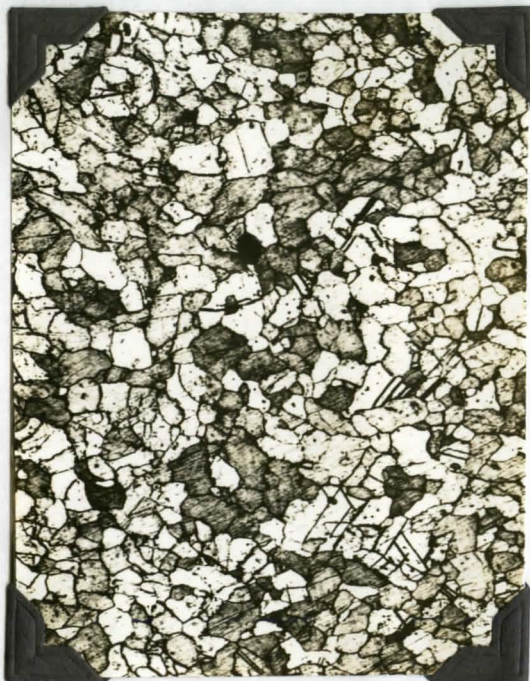


Plate A. Before anneal



Plate C. Annealed specimen
repolished and re-
etched

Figure 9. Effect of 3 hour Anneal at 550 degrees C, 100 x.

SUMMARY

The results of this investigation indicate that Neumann bands are completely removed above the critical temperature, 700 degrees C. Below this temperature they tend to persist and their action is very similar to that of the original grain boundaries. When grains containing Neumann bands are being absorbed, the bands become fainter and thinner, finally disappearing completely.

The orientation of the Neumann bands do not affect the orientation of the growing grains, and there is no difference in the manner of absorption of the marked or unmarked grains. In some cases the new grain boundaries cut the exact lines where the Neumann bands had been originally.

APPENDIX

SAMPLE DATA SHEET

Sample 2Section TransverseAnneal 1hr at 950°C

Camera Settings

Objective 23 mm.Ocular 6x PeriplanIlluminator PrismBellows 41 cm

	Diaphragm	Shutter	Printing Time
A. Before anneal	<u>5</u>	<u>1/50 sec</u>	<u>6 sec</u>
B. After anneal	<u>10</u>	<u>1/2 sec</u>	<u>8 sec</u>
C. After reetch	<u>6</u>	<u>1/50 sec</u>	<u>6 sec</u>

Plates- Wratten and Wainwright Metallographic Dry Plates

Plate Developer - D-70

Print Developer - D-72

Figure 2

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VITA

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Alex B. Davidson, the son of Harold B. Davidson and Violet S. Davidson, was born in Memphis, Tennessee, on April 17, 1917. At the age of three he moved to Louisville, Kentucky, and has lived there from that time.

Mr. Davidson attended the Louisville public schools and finished his secondary school education in Louisville Male High School, 1935. He attended the Speed Scientific School of the University of Louisville from 1935-1939 receiving a degree of Bachelor of Chemical Engineering. His thesis was "The Tantalum Electrode for p^H Measurements."

During his college career Mr. Davidson was active in extra curricular activities culminating in the captaincy of the University of Louisville football squad of 1938.