

SOME EXPERIMENTS ON THE
SOLID-PHASE BONDING
OF LEADED BRASS

by

JERALD OLIVER MCRAE

A THESIS

submitted to


OREGON STATE COLLEGE

in partial fulfillment of
the requirements for the
degree of

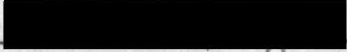
MASTER OF SCIENCE

June 1949


APPROVED:



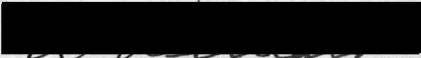
Professor of Mechanical Engineering
In Charge of Major



Head of Department of Mechanical Engineering



Chairman of School Graduate Committee



Dean of Graduate School

ACKNOWLEDGMENT

The author wishes to express his appreciation to Professor S. H. Graf, for his invaluable suggestions and advice concerning many details of this thesis.

Thanks are also due to Professor O. G. Passche for his innumerable helpful suggestions and for the time he spent in taking several of the photographs contained in this thesis.

TABLE OF CONTENTS

| | Page |
|---|------|
| INTRODUCTION | 1 |
| MATERIAL SELECTED | 3 |
| DESIGN OF EQUIPMENT | 9 |
| BASIC THEORETICAL CONSIDERATIONS | 18 |
| EXPERIMENTAL BONDING PROCEDURE | 22 |
| EVALUATION OF THE SOLID-PHASE BONDS | 27 |
| SUMMARY AND CONCLUSIONS | 49 |
| BIBLIOGRAPHY | 54 |

ILLUSTRATIONS

| | Page |
|---|------|
| Figure 1. The Copper-Zinc Constitution Diagram . . | 6 |
| Figure 2. Photomicrograph of Material Selected showing Presence of Directional Properties | 8 |
| Figure 3. Photomicrograph of Material Selected showing Presence of Beta Brass | 8 |
| Figure 4. Leaded Brass Specimens | 9 |
| Figure 5. Testing Machine and Equipment Assembled for Solid-Phase Bonding Experiment . . . | 11 |
| Figure 6. Furnace removed showing Specimen Container in place between Water-Cooled Loading Members | 13 |
| Figure 7. Section thru Assembled Equipment showing Furnace, Specimens, Container, Thermo- couple, and Loading Members | 14 |
| Figure 8. Tensile and Metallographic Specimens . . | 31 |

ILLUSTRATIONS (CONTINUED)

| | Page |
|--|------|
| Figure 9. Graph of Specimen Series One | 33 |
| Figure 10. Macrograph showing Fracture of Specimen 31 | 34 |
| Figure 11. Graph of Specimen Series Two | 36 |
| Figure 12. Graph of Specimen Series Three | 37 |
| Figure 13. Photomicrograph of Specimen 25 | 38 |
| Figure 14. Photomicrograph of Specimen 14 | 40 |
| Figure 15. Photomicrograph of Specimen 13 | 41 |
| Figure 16. Photomicrograph of Specimen 19 | 42 |
| Figure 17. Photomicrograph of Specimen 15 | 43 |
| Figure 18. Photomicrograph of Specimen 18 | 44 |
| Figure 19. Photomicrograph of Specimen 11 | 45 |
| Figure 20. Photomicrograph of Specimen 31 | 46 |
| Figure 21. Photomicrograph of Specimen 31 showing Lead Segregation | 47 |
| Figure 22. Photomicrograph of Specimen 28 | 48 |

TABLES

| | Page |
|--|-------|
| Table I. Results of Tensile Tests of the Specimens | 29-30 |

SOME EXPERIMENTS ON THE SOLID PHASE BONDING OF LEADED BRASS

INTRODUCTION

Welding is usually thought of as a fusion process. The edges of two members are melted either with or without the addition of other molten material, and the members are joined by solidification of the molten material which exists between them at some stage of the process. This is known as liquid phase bonding. In another welding process only the added material is melted and the initial members to be welded remain solid at all points. This is liquid-solid phase bonding, a common example of which is bronze welding or brazing.

Forge welding is another very old art and has on occasion been erroneously thought of as solid phase bonding. In this type of welding, according to Kinzel (6), a low melting phase which is liquid at the forge welding temperatures and comprises primarily iron and iron and other oxides is largely responsible for the bonding. When fluxes are used a larger amount of the liquid phase is present. The function of the hammering is to squeeze out any excess liquid and inspection after welding will show a once molten constituent at the interface of the primary members. Therefore, forge welding is not a solid phase bonding process. In flash welding the existence of a

liquid phase is obvious. In electrical resistance welding there is a liquid phase formed at least locally, which can be verified by the microscope.

Solid phase bonding is the process by which particles or members are bonded by atomic forces without the presence of a liquid phase at any stage of the process. In his history of powder metallurgy, Cyril Smith (8), sets forth the following facts. Solid phase bonding is the oldest of all types of welding. In Egypt iron was used as early as 3000 B.C. This iron was prepared by producing a metal sponge and hammering it to bond the particles together. The products were often unsound and full of slag but occasionally excellent samples were produced.

While the solid phase bonding of metal particles is old the solid phase bonding of structural members is new. Obviously, powder metallurgy is based on solid phase bonding. Solid phase bonding is feasible between identical metals and between dissimilar metals at suitable temperatures and pressures. It is commonly agreed upon that cleanliness of the surfaces is a prerequisite for obtaining a good bond. Durst (4), states that it is generally agreed that the mechanism of solid phase bonding is based on the diffusion process and the pressure employed is chiefly a means of bringing the two surfaces close enough together to get within the range of atomic attraction. This assumption

implies that every conceivable combination of metals will probably be solid phase bondable. Some diffusion occurs even between two metals which are considered to be insoluble in each other.

Thin but tough surface films like oxides and gas absorption films, as normally present on metallic surfaces, are in general sufficiently insulating to prevent diffusion, thereby hindering solid phase bonding. Atomic attraction is weak beyond one interatomic distance; thus gas films, believed to be monomolecular, are an effective stop-off.

Absorption films can be eliminated by heat, while hot oxide films, as a rule, grow in thickness unless oxygen is completely absent from the environment. How much of an obstacle is an oxide or a gas absorption film? What temperatures and pressures are required for producing a solid phase bond? In an effort to provide the answers to some of these questions, the experiments described in this thesis were carried out.

MATERIAL SELECTED

In the selection of a suitable material for experimentation a number of factors had to be considered. As the time available for this thesis was limited, the availability of the material was given first consideration.

In order to obtain a range of sufficient satisfactory data, it seemed advisable to choose a material that could be heated to very near its melting point without necessitating the construction of an intricate and expensive furnace. A simple resistance wound tube furnace can be expected to develop temperatures, within the tube, up to 1000 C. Above this limit a controlled atmosphere, vacuum, or carbon resistance furnace is advisable.

Basic solid phase bonding theory indicates that a material which undergoes a transformation may be satisfactorily bonded at that transformation temperature, which is often well below the melting point. In order to investigate this concept, a material which would undergo a transformation well below the liquidus was required.

It was apparent that considerable machining would be necessary to convert the as-welded specimens to standardized test specimens for metallographic examination and physical testing. In view of this fact a material with good machining characteristics was desired.

After consideration of the above-mentioned factors, the material chosen was a leaded brass of the following nominal composition: 62 percent copper, 35 percent zinc, and 3 percent lead. Heyer (5), states that for all practical purposes copper-lead alloys are of the mechanical mixture type, their solid solubilities being 0.06 percent

copper in lead and 0.02 percent lead in copper. He further states that lead plays essentially the same role in leaded brasses. The melting point of lead is far below the melting point of brass and at any bonding temperature in excess of 327 C a liquid phase will exist. However, as noted above, lead is only very slightly soluble in brass and can usually be expected to be well dispersed within any representative sample. In the interest of obtaining a material which undergoes a transformation below the melting point and also displays free machining characteristics, the leaded brass was still considered most suitable. With the relatively short heating and cooling periods to be employed, lead segregation within the specimens seemed unlikely and its effect on the solid phase bonding of the brass was deemed irrelevant.

In order to place the alloy in the correct position on the copper-zinc constitution diagram, shown in Figure 1 on the following page, it was necessary to run a quantitative chemical analysis on the material. This analysis was carried out in accordance with the standard procedure outlined by the American Society for Testing Materials (1). The composition as determined by this analysis was as follows: copper 60.5 percent, zinc 36.6 percent, and lead 2.9 percent. Considering the brass matrix to be essentially free of lead it would be composed of 62.3 percent copper

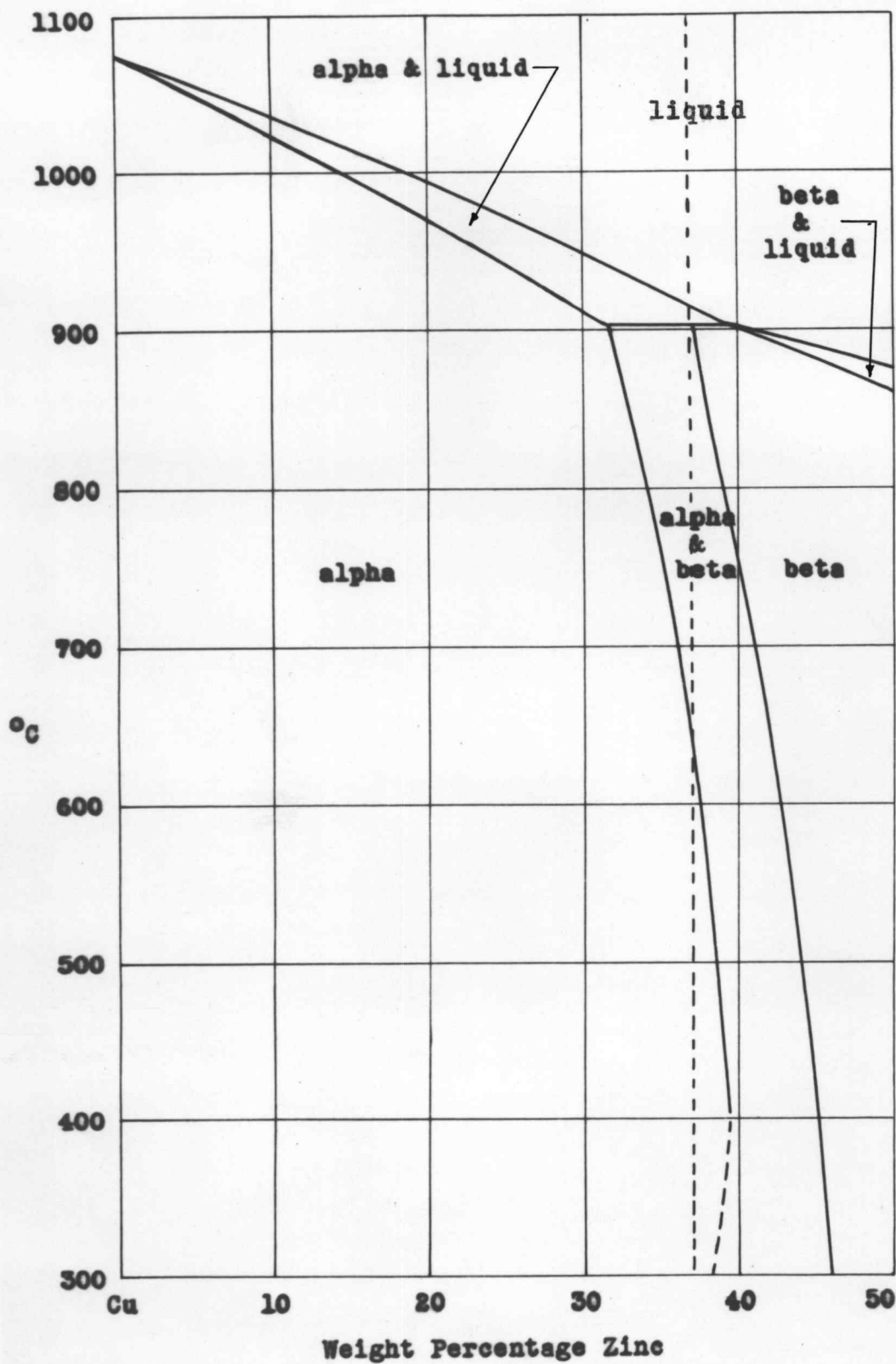


Figure 1 Copper-Zinc Constitution Diagram

and 37.7 percent zinc. From the constitution diagram of Figure 1, the following data are apparent. This alloy undergoes a transformation from alpha brass to alpha plus beta brass at approximately 620 C. The melting point is 903 C. This material fulfilled the chemical composition requirements as set forth by the American Society for Testing Materials (2), and it is assumed that the material was cold rolled to 1/2 hard inasmuch as no hardness was specified. Wilkins and Burns (9), list 1/2 hardness as corresponding to approximately 20 percent reduction by cold working.

By polishing an original sample and etching it with ammonium hydroxide and hydrogen peroxide the microstructure displayed marked directional properties as shown in Figure 2. By polishing the specimen and etching it with a solution of ferric chloride, some beta brass was shown to be present as seen in Figure 3. The presence of the beta brass at room temperature indicates that the material was cooled, during production, at a rate in excess of that required for complete transformation from alpha plus beta brass to alpha brass. The lead present in the specimen was shown to be well dispersed as expected.



Figure 2. Original sample of leaded brass. Etchant used was 43 cc NH_4OH and 6 cc of H_2O_2 . 200x.

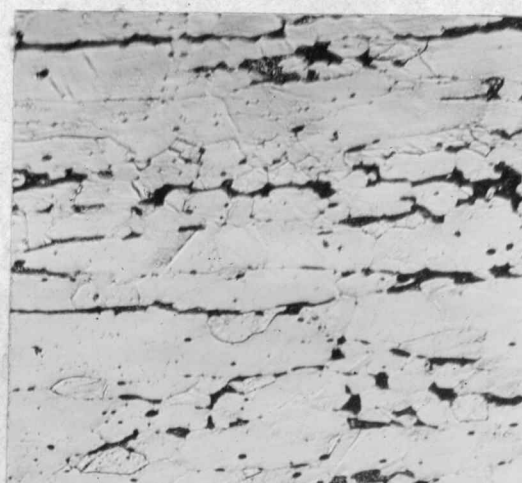


Figure 3. Original sample of leaded brass. Etchant used was FeCl_3 . 200x.

The leaded brass was obtained in rod form and was 1 1/8 inches in diameter. The specimens were machined from 1 1/2 inch lengths of the rod and one specimen of each pair was drilled to receive a thermocouple. The interface surfaces of the specimens were 3/4 of an inch in diameter. They were finished on number one emery paper and cleansed in alcohol before being inserted into the containers. The containers are described in detail in the following section of this thesis. A cross section of a pair of specimens, as they appeared within the container, is shown in Figure 4.

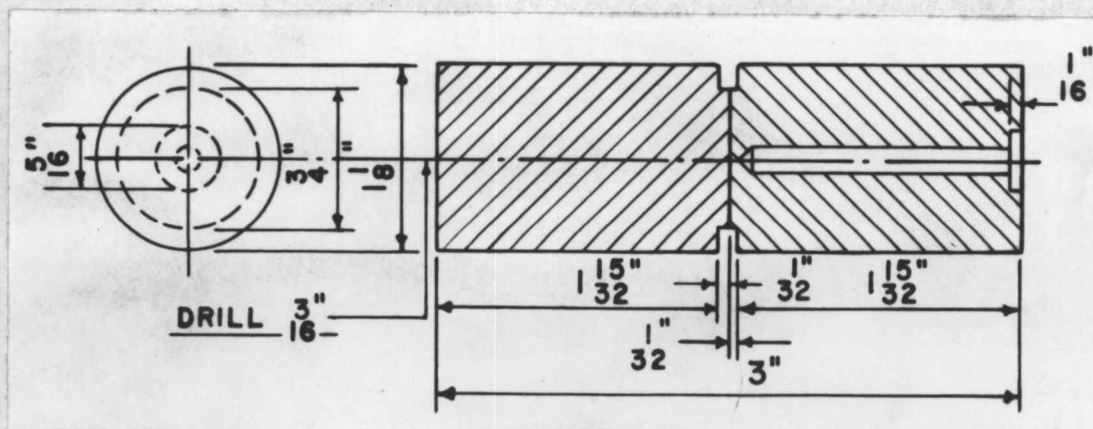


Figure 4 Leaded Brass Specimens

DESIGN OF EQUIPMENT

The equipment to be used in these experiments was designed to meet the following requirements.

1. To provide a means of applying a measured compressive load to the specimens at some predetermined temperature.
 2. To provide a means of holding the specimens in alignment during the bonding process.
 3. To provide a means of controlling oxidation of the specimens by the use of a vacuum or an inert atmosphere.
 4. To provide a means of uniform heating of the specimens with an upper limit of at least 900 C.
- To further provide a heat source whereby the specimens could be heated rapidly in order to

minimize the oxidation of the specimens during the heating cycle.

5. To provide a means of measuring the temperature at or near the interfaces of the specimens.
6. To provide high temperature materials or adequate cooling of less heat resistant materials, in order to impart durability to the equipment under high temperature loading.
7. To provide a simple method for inserting and removing the specimens in the apparatus.
8. To provide equipment as simplified as possible.

In order to fill the above requirements, the following selections and decisions were made.

A standard testing machine was chosen to provide a means of applying a measured compressive load at a predetermined temperature. This machine is shown in Figure 5.

It was decided to include an evacuated specimen container in the apparatus for the following reasons.

1. To hold the specimens in alignment during bonding.
2. To control oxidation of the specimens by the use of a vacuum and also to provide a means for obtaining a variation in oxidation as desired in one series of specimens to be run during the experiments.

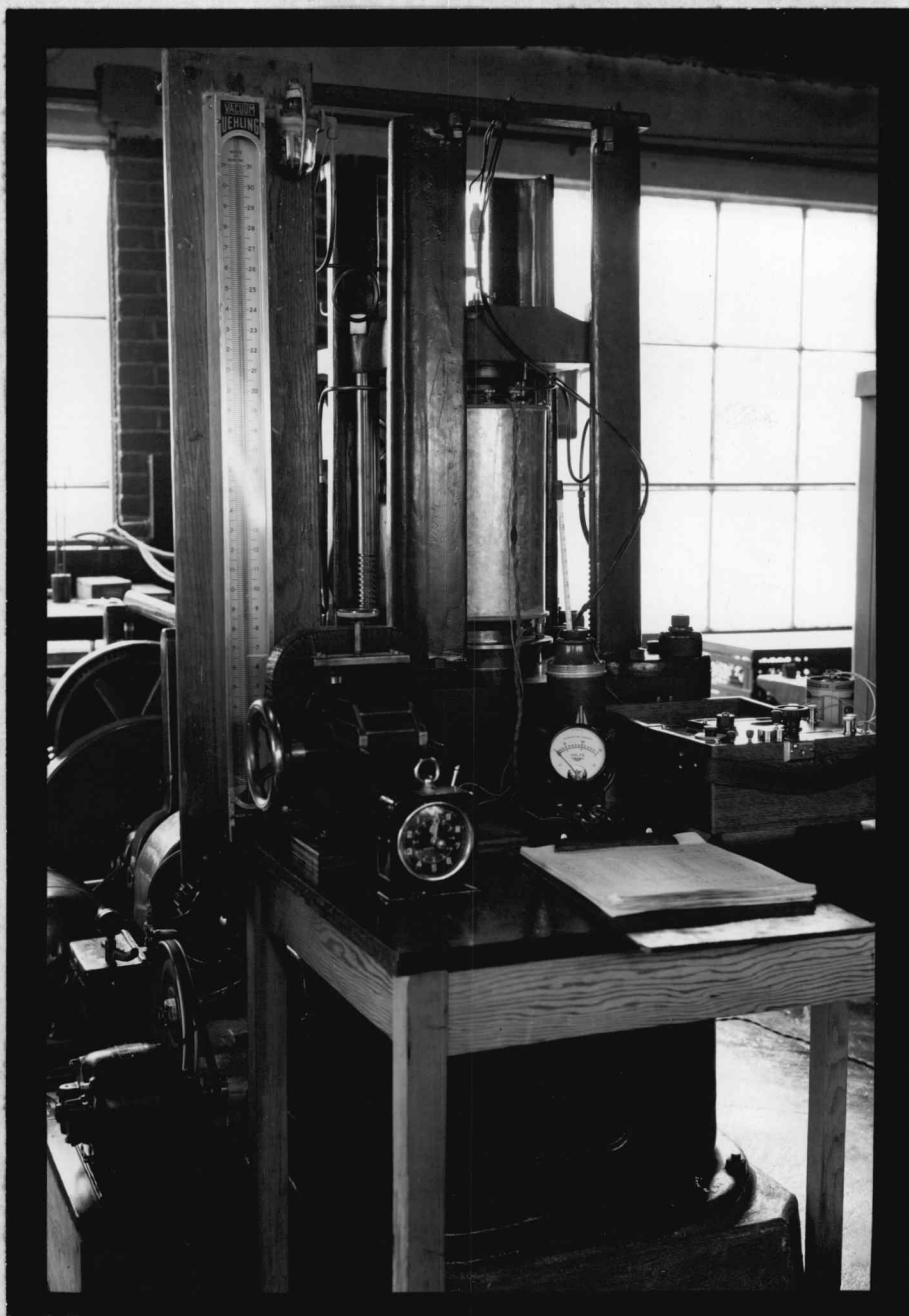


Figure 5 Testing Machine and Equipment
Assembled for a Solid Phase
Bonding Experiment

3. To help provide uniform heating of the specimens by diminishing the effects of any temperature gradient which might exist within the furnace tube.
4. To provide a simple means of inserting and removing the specimens from the furnace by assembling them as a unit.
5. To render a simply wound resistance tube furnace adequate as a heating unit.

In order to simplify the construction of the container it was decided to place the thermocouple within the vacuum tube. A chromel-alumel thermocouple was acceptable for measuring the temperatures to be employed.

Due to the fact that high temperature alloys were not readily available, it was decided to construct the various components of mild steel and to provide water cooling of the members used to apply the compressive load. These two members were also provided with inconel tips to resist oxidation and high temperature loading. The inconel tips were seated by a pressed fit and the members were fabricated by welding as shown in Figures 6 and 7.

The specimen containers were also made of mild steel. The body of the container was a 3 inch length of 1 1/4 inch thin wall steel tubing. The containers were of welded construction as shown in Figures 6 and 7. In order to reduce

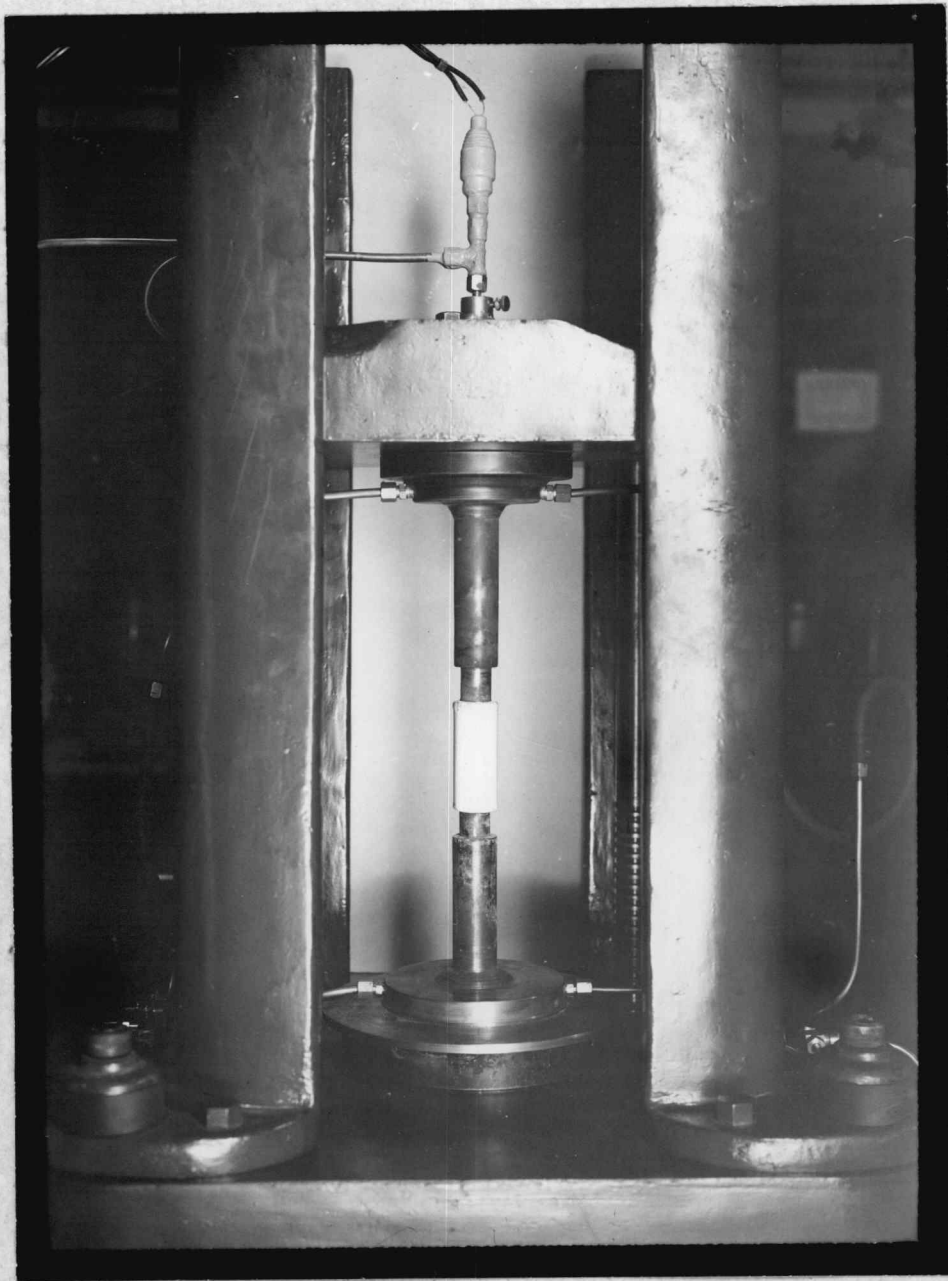
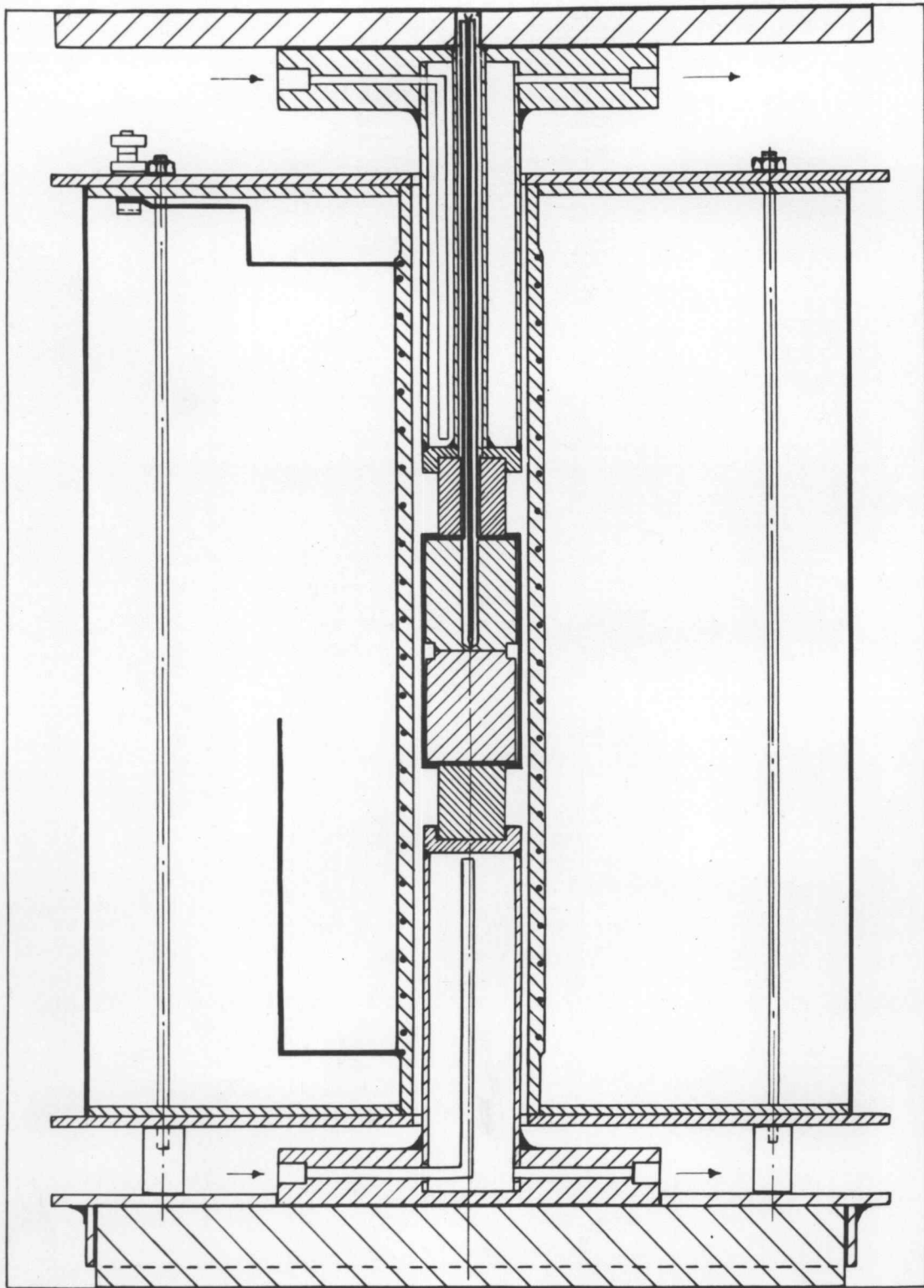
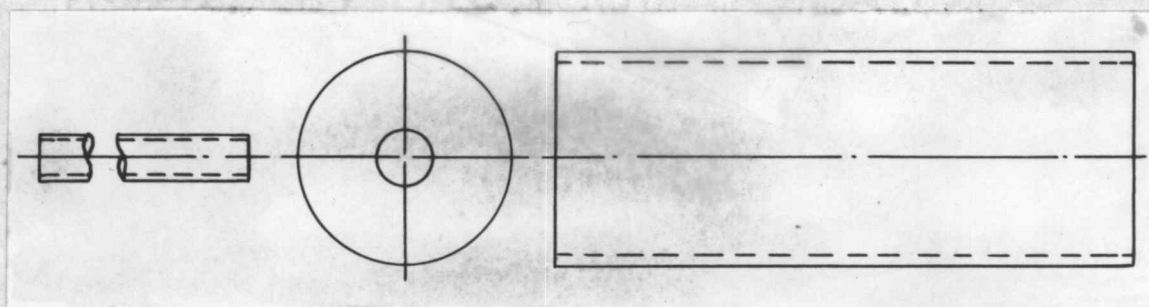


Figure 6 Furnace Removed Showing Specimen Container in place between Water Cooled Loading Members. At top thermocouple is seen emerging thru seal from vacuum tube.



**Figure 7 Section through Assembled Equipment
Showing Furnace, Specimens, Container,
Thermocouple, and Loading Members.**

the effect of the container in regard to the load applied for bonding, the containers were constructed as follows:

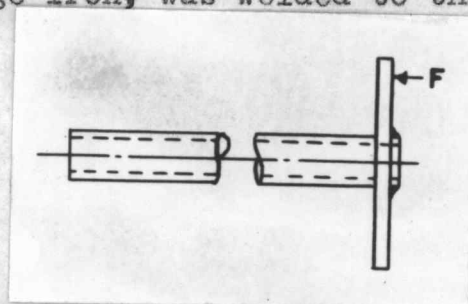


Part A

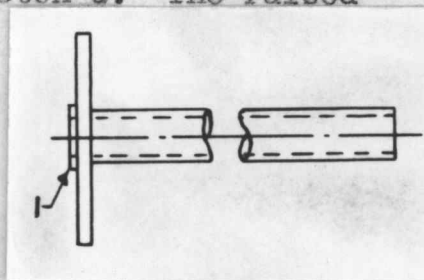
Part B

Part C

Part B, which was cut from 20 gage iron, was welded to the vacuum tube A as shown in sketch E. The circumference was then machined to conform to part C. The surface F was machined to provide a flat surface perpendicular to the tube designated A above. After welding and machining, parts A and B exhibited a section as shown in sketch G. The raised portion I, shown in sketch G, formed a re-enforcement for the tube and end weld. The barrel C was then attached by the use of a butt weld. This type of construction gave a container which provided proper



Sketch E



Sketch G

alignment of the specimens during bonding and also proper alignment of the container within the furnace.

The containers were lined completely with a thin sheet of mica approximately 0.015 of an inch thick. The mica was used for the following reasons:

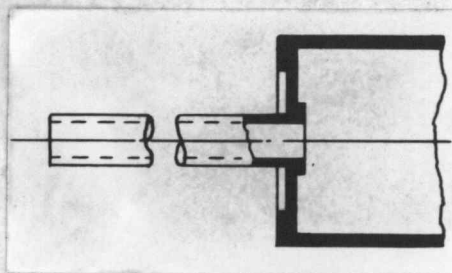
1. It prevented contamination of the specimens by any minute particles of oxide scale which might be present within the container.
2. It prevented bonding or seizure of any surfaces of the specimens with the exception of the interfaces and as a result it aided in the easy assembling of the specimens for bonding and their subsequent removal after bonding.
3. It further diminished the effects of any temperature gradient which might exist within the container thereby providing a uniform heat supply to the specimens.

After assembling the specimens in the containers a disc of mica was inserted and a cap of 20 gage iron welded on. This weld was made in an off-the-work manner whereby the flame was directed off the work rather than on it as is customary in gas welding. This method was used in order to prevent undue heating of the specimens and subsequent preliminary oxidation. The presence of the mica as insulation made this weld possible and helped to localize the heating

employed for welding.

Realizing that any loss of load due to the presence of a container would be in proportion to the amount of deformation obtained, the containers and specimens were assembled, as a unit, with as small a longitudinal clearance as possible. After completing the assembly the containers were machined on the end surfaces as shown in sketch H. The machined portions of the ends of the containers were $1/32$ of an inch thick after the final machining operation. The

containers were designed with an end diameter of $1\frac{1}{4}$ inches even though



Sketch H

the loading members were only $7/8$ of an inch in diameter. In this manner the thin end pieces could deform during loading rather than necessitating the collapse of the steel barrel walls. The effect of the container on the load applied for bonding was further minimized in this manner. The specimens were to be removed from the containers after bonding by using a lathe and a cut-off tool. This operation would result in the destruction of the container. However, it was believed that an attempt to use a container for successive experiments would result in experimental difficulties and unreliable data.

The furnace was of the simple resistance wound tube type. It employed a heating element composed of approximately 18 feet of chromel-A wire of gage 18 wound on an alundum tube and imbedded in alundum cement. A cross section of the furnace is shown in Figure 7. The furnace was controlled by the use of a reactor to obtain a variable potential supply.

The temperature of the specimen interface was determined by the use of a calibrated chromel-alumel thermocouple and the electromotive force developed by the couple was measured with a potentiometer. An ice-water cold junction was employed with the thermocouple.

The evacuation of the container was obtained by the use of a vacuum pump and the vacuum obtained was measured by differential readings between a vacuum manometer and a standard barometer. The equipment noted above is shown assembled at the testing machine in Figure 5.

BASIC THEORETICAL CONSIDERATIONS

According to Durst (4), it is generally agreed that the mechanism of solid phase bonding is based on the diffusion process. There is only a very limited amount of experimental data available for the determination of diffusion coefficients. However, enough has been done so that it is logical to speculate that for most metals the diffusion coefficient is too small for transfer sufficient for good

bonding, except as the temperature approaches the melting point. Here the diffusion coefficient increases rapidly and becomes of sufficient order to permit good bonding.

The change in structure with the accompanying movement of atoms in a phase change also greatly increases the coefficient of diffusion. One can readily visualize surfaces being brought into intimate contact under moderate pressure, so that in the course of the transition the surface atoms may be said to diffuse across the interface. These atoms are in the process of reorganization and if they find themselves equidistant from their neighbors on the right and on their left (relative to the original interface) they can be considered to reorganize without regard to their original position in regard to the interface. Furthermore, by bonding, the original state of stress at the interface is eliminated.

There is likewise a parallelism in solid phase bonding and the production of large metallic single crystals. Durst (4), states that generally to produce single crystals stresses must be first relieved by heating and then a small critical stress which varies with temperature and time is applied. Significantly parallel to solid phase bonding is the observation that single crystal growth is greatly expedited by successive passes through an allotropic phase change. All of this indicates that any transformation will play a major role in solid phase bonding.

ADVANCE BOND

It is quite obvious that in certain cases recrystallization of the material to be bonded can have an important effect on the quality of the bond produced. In the writer's opinion recrystallization is certainly a diffusion process. The temperature at which the recrystallization will occur is determined to a large extent by the degree of cold or hot work experienced by the specimen. In other words, by the degree of distortion of the crystal lattices whereby a large distortion will impart a large amount of internal energy to the metal and will consequently bring about recrystallization at some lower temperature.

Several fundamental laws of recrystallization are now well recognized.

1. Recrystallization occurs at a higher temperature, the smaller the degree of deformation or, more accurately, the smaller the degree of strain hardening.
2. Increasing the time of annealing displaces recrystallization to a lower temperature.
3. The final grain size depends chiefly on the degree of deformation and to a lesser extent on the annealing temperature; the final grain size is larger, the smaller the degree of deformation.
4. The larger the original grain size, the greater the amount of cold deformation to give equivalent recrystallization temperatures and times.

In addition to these major factors, it should be noted that (a) the rate of heating to the recrystallization temperature is a further variable, and (b) the amounts of cold work to give equivalent strain hardening vary with the temperature at which deformation is performed.

It has also been established that any impurities or insoluble particles within a metal will greatly influence the recrystallization process.

There are two possible procedures which may be used to effect a solid phase bond. In one procedure the specimens are loaded immediately after being inserted into the furnace. The load is held constant until a predetermined bonding temperature is reached. The load is then removed and the bond is considered to be completed. The other procedure involves no application of load until the predetermined bonding temperature is reached. At this temperature the load is applied for a measured interval of time and the bonding is effected.

The aforementioned decision to use a specimen container immediately recommended the use of procedure 2 whereby the load would not be applied until the container had reached a temperature at which its effect on the load applied for bonding would be negligible.

A preliminary test specimen was run using procedure 1. The fracture of the bond showed that a certain percentage

of the interface area had been in intimate contact throughout the process and as a result no oxidation of these areas had occurred. Therefore, the bond, considered as a whole, was not uniform. In order to prevent the introduction of another variable it was decided to use procedure 2 as described above.

EXPERIMENTAL BONDING PROCEDURE

The first preliminary test runs were carried out by assembling the specimen containers within the furnace and then applying a potential across the winding. The time required to bring the specimens to temperature in this manner was approximately 50 minutes. This extended period of heating increased the oxide film on the interfaces and good bonding was not obtained. As a corrective measure the various accessory connections, including the water lines, vacuum line, and thermocouple, were lengthened to allow the inserting of the container after the furnace was in position and at the temperature desired. This procedure decreased the heating time required to approximately 20 minutes and satisfactory bonding was then obtained. The specimens employed in the preliminary experiments were run for the purpose of standardizing the equipment and its operation, and to determine a starting point for subsequent experimentation.

The deformation of the containers was measured by means of a vertical scale which was mounted on the head of the testing machine.

In order to determine what part of the load applied was supported by the container the following experiments were carried out. A container of the standard type was loaded with a pair of specimens and immediately after being inserted into the furnace it was loaded in compression with a 100 pound load which was held constant. The furnace had been preheated at 80 volts potential for 20 minutes prior to inserting the container. When the temperature of the specimen interfaces had reached approximately 650 C, the ends of the container deformed approximately $1/32$ of an inch after which the clearances within the container were exhausted and the 100 pound load became steady again. A second assembled container unit was inserted into the furnace and a load of 50 pounds was applied as before. The same events took place with the exception that a slightly longer period of time was necessary to complete the $1/32$ of an inch deformation. In view of these experiments it is believed that when the specimens had reached a temperature of approximately 650 C, the container had been heated to some higher temperature and as a result had reached a plastic state whereby it could not sustain any appreciable load for even a short interval of time. By

inserting a bare thermocouple into the approximate central axis of the furnace tube and covering the top opening in the furnace a temperature of approximately 1010 C was measured. Although the actual temperature of a container was never measured while the specimens were at a temperature of 650 C, it is obvious that the container was at some temperature above that of the specimens. After the first initial runs the loading members which were in contact with the containers were slightly oxidized on the surface and developed a slight scale whereby the heat loss through the end surfaces of the container was greatly decreased. This fact was supported in that the initial runs required a much greater supply of cooling water than those runs made later.

The mica lining of the containers tended to provide insulation between the specimens and containers and this fact together with the decreased heat loss noted above would tend to indicate that the containers reached a temperature of perhaps 900 C while the specimens showed a measured temperature of 700 C. It should be pointed out that only one specimen was bonded at a temperature below 650 C. This specimen was run at a temperature of 620 C in order to investigate the effect of a predicted transformation on the bond produced.

In view of the fact that pressure is not the governing factor in solid phase bond, the effect of the container on the load applied was deemed insignificant and pressure values, as tabulated and shown on various curves in the remaining sections of this thesis, are the actual measured loads applied.

The experiments carried out were divided into 3 series. The variables involved were temperature, pressure, vacuum, and time.

Series 1 was made up of 14 specimens which were run holding pressure, vacuum, and time constant. The temperature was varied from 620 C to 870 C for the various specimens. This temperature range began below the transformation temperature and extended to within approximately 30 C of the melting point. The load applied was constant for the series of specimens and was measured as 300 pounds which is equivalent to 725 pounds per square inch of interface area. The specimens were loaded for a period of 30 seconds. The vacuum measured showed a residual pressure of 0.2 of an inch of mercury. This series was made up of the majority of specimens and the corresponding curve showing the relationship between ultimate tensile strength and bonding temperature was verified at several points. This curve will be found in the following section. The 300 pound load was chosen because the specimens would not

support a greater load at temperatures above 800 C without severe deformation. In order to minimize the load supported by the container and to provide a possibility of determining the occurrence of recrystallization during bonding, severe deformations were not desired.

Series 2 was made up of 5 specimens and in this case the pressure was varied. The temperature, the time, and the vacuum were held constant. The pressure was varied in steps of 100 pounds, beginning with a 100 pound load and including a 500 pound load. The vacuum was measured as a residual pressure of 0.2 of an inch of mercury. This was the highest vacuum obtainable with the vacuum pump employed. The specimens were loaded for bonding at a temperature of 800 C and the load was applied for a period of 30 seconds. It should be pointed out that this series was not designed to provide a verified relationship between the ultimate tensile strength of the bond and the pressure applied for bonding. It was designed to determine the trend of such a relationship.

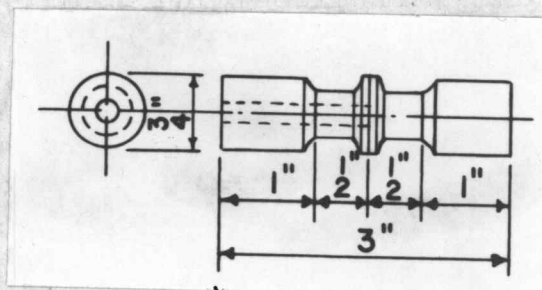
Series 3 was composed of 3 specimens for which the vacuum was varied. The temperature, the pressure, and the time of bonding were held constant. The load applied was 300 pounds and the bonds were made at a temperature of 850 C. The load was applied for 30 seconds. The specimens were run at vacuums measured as residual pressures of

0.2 of an inch of mercury, 8 inches of mercury, and 18 inches of mercury. This series was run to determine the trend of the relationship between vacuum employed and the ultimate tensile strength of the bond produced.

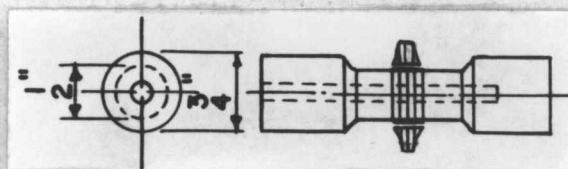
EVALUATION OF THE SOLID PHASE BONDS

The solid phase bonds produced in these experiments were evaluated by physical testing and metallographic examination. Due to the fact that a large number of low strength bonds were expected the only physical test employed was the determination of the ultimate tensile strength of the bonds. The low strength bonds gave no reduction in area and as a result no measure of ductility was possible. The yield point in brass is usually not clearly defined and therefore no yield point determinations were made on the specimens.

In order to provide a tensile specimen and a metallographic specimen the as-bonded samples were machined as shown in sketch J. The metallographic specimens were removed as shown in sketch K. After removal of the metallographic



Sketch J



Sketch K

samples the tensile test specimens were machined in accordance with the specifications set forth by the American Society for Testing Materials (3), with two exceptions. The gage length employed was 1 inch instead of the standard 2 inches. This exception was necessitated by the 3 inch length of the as-bonded specimens. The second exception was necessary due to the presence of the thermocouple hole in one of the specimens as shown in sketch J. In order to give a uniform section throughout the 1 inch gage length the hole was extended as shown in sketch K. The ends of the specimens were threaded to fit the holders of a 60,000 pound Baldwin-Southwark testing machine. This machine was hydraulically activated and provided slow, even loading of the specimens. A photograph of the tensile test specimens and the metallographic specimens is shown in Figure 8. The tabulated results of the tensile tests are shown on the following pages of this thesis.

Specimen 25 was run in order to investigate the transformation expected at 620 C. The ultimate tensile strength did show the presence of the transformation. It should be pointed out that due to the presence of lead in the brass the transformation temperature may not have been 620 C. Therefore it would be erroneous to say that this specimen shows the complete effect of a transformation on the bond produced. However, by comparing the tensile test data

TABLE I

RESULTS OF TENSILE TESTS OF THE SPECIMENS

| Specimen No. | Bonding Temp C | Bonding Load Applied lb | Vacuum--Re- sidual Press. in. of Hg | Ult Tensile Strength psi |
|-----------------|----------------------|-------------------------------|---|--------------------------------|
| Series 1 | | | | |
| 25 | 620 | 300 | 0.2 | 10,900 |
| 10 | 725 | 300 | 0.2 | 0 |
| 12 | 750 | 300 | 0.2 | - |
| 21 | 780 | 300 | 0.2 | 476 |
| 14 | 786 | 300 | 0.2 | 3,340 |
| 20 | 794 | 300 | 0.2 | 7,850 |
| 13 | 800 | 300 | 0.2 | 9,970 |
| 19 | 812 | 300 | 0.2 | 14,800 |
| 15 | 824 | 300 | 0.2 | 17,500 |
| 18 | 832 | 300 | 0.2 | 20,150 |
| 11 | 850 | 300 | 0.2 | 24,500 |
| 31 | 870 | 300 | 0.2 | 17,450 |

TABLE I (CONTINUED)

| Specimen No. | Bonding Temp C | Bonding Load Applied lb | Vacuum--Residual Press. in. of Hg | Ult Tensile Strength psi |
|----------------------|----------------|-------------------------|-----------------------------------|--------------------------|
| Series 2 | | | | |
| 22 | 800 | 100 | 0.2 | 0 |
| 29 | 800 | 200 | 0.2 | 1,900 |
| 13 | 800 | 300 | 0.2 | 9,970 |
| 24 | 800 | 400 | 0.2 | 10,250 |
| 23 | 800 | 500 | 0.2 | 10,500 |
| Series 3 | | | | |
| 11 | 850 | 300 | 0.2 | 24,500 |
| 26 | 850 | 300 | 8.0 | - |
| 30 | 850 | 300 | 18.0 | 0 |
| Specimens Not Bonded | | | | |
| 27 | (See page 32) | | | 46,800 |
| 28 | | | | 47,600 |

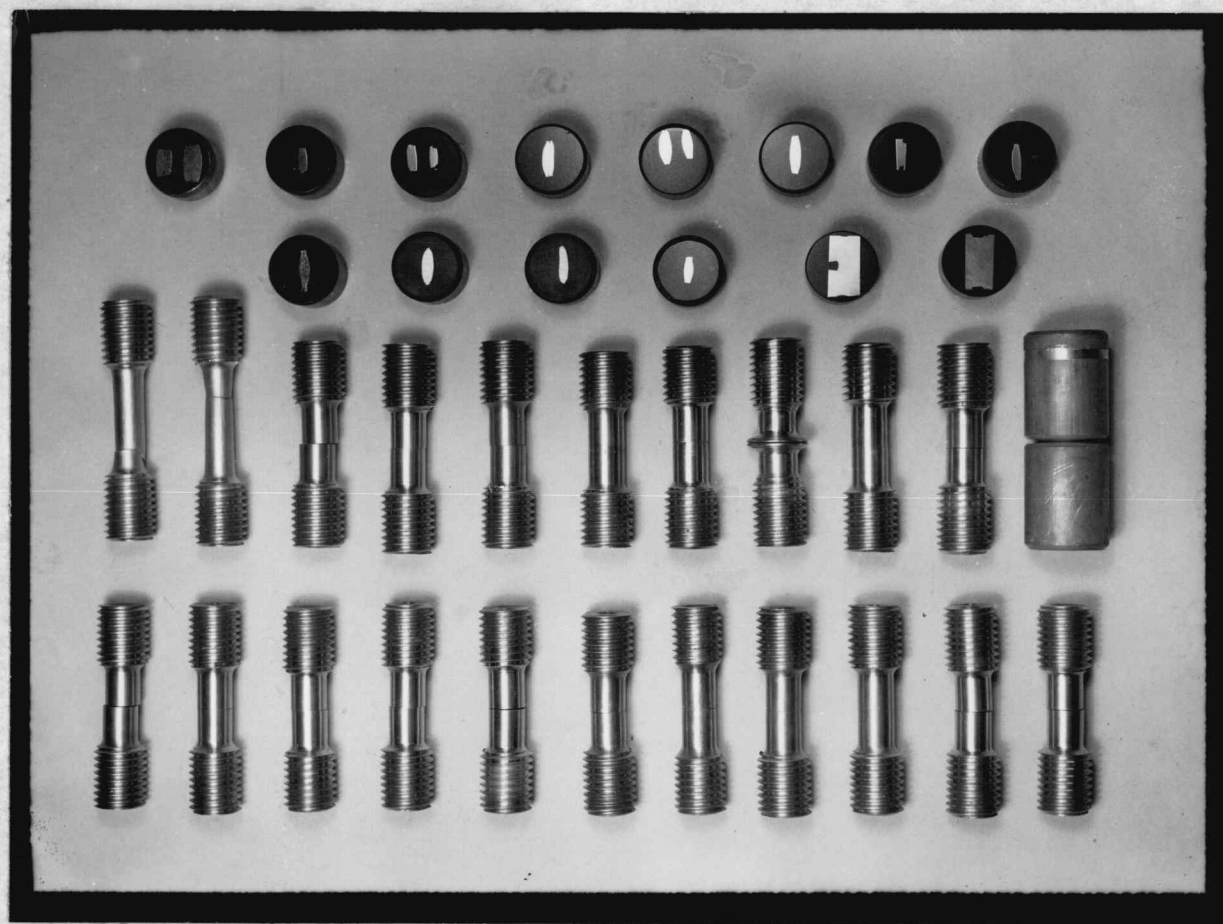


Figure 8 Tensile and Metallographic Specimens

tabulated on pages 29 and 30, it is obvious that the transformation, either partial or complete, did result in a stronger bond than one would have expected without a transformation. Furthermore, the strength of this bond indicates that at least a partial transformation did occur during the bonding. Due to the fact that the temperature of bonding was below 650 C, the container did sustain at least a part of the load applied; this specimen was not contained in the plot of ultimate tensile strength versus bonding temperature shown in Figure 9.

In order to compare the strength obtained in the bond in specimen 11 to that of the parent metal, two experimental runs were necessary. Two containers were loaded with a standard rod of brass 3 inches in length and heated, by the standard procedure, to a temperature of 850 C. They were held at that temperature and then removed and cooled in the same manner as the bonded specimens. The machining operations employed were identical. When these specimens were tested in tension it was found that the bond produced in specimen 11 supported approximately 52 percent of the load required to fracture the parent metal.

The bond produced in specimen 31 was expected to support a greater load than that which specimen 11 supported. However, upon machining specimen 31 it was found that lead segregation had occurred and also that a partial

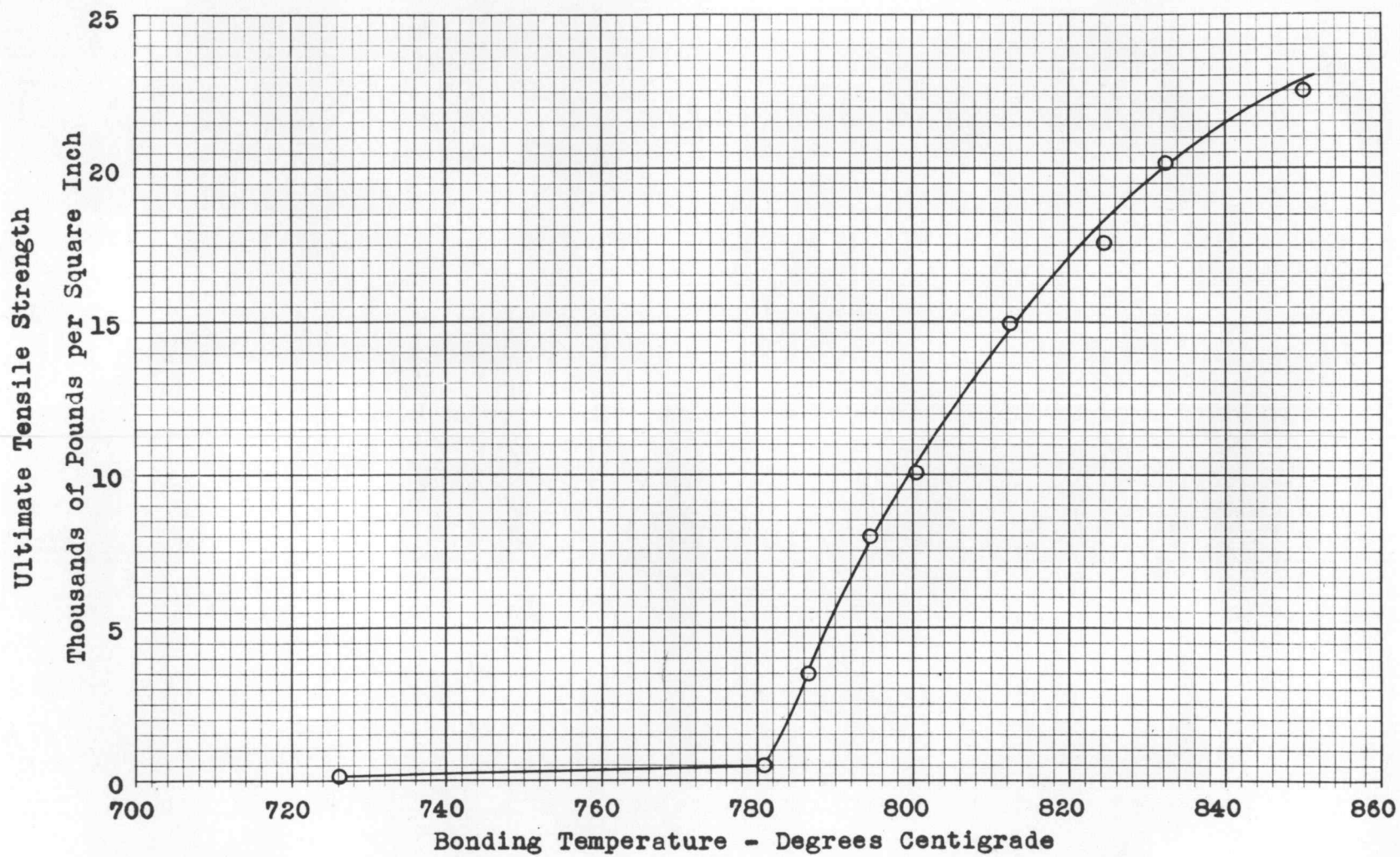


Figure 9 Graph of Specimen Series One

vaporization of the segregated lead had left the material honeycombed with gas holes. To verify this statement a quantitative chemical analysis was run on a sample of the honeycombed material. This analysis was also run in accordance with the standard procedure outlined by the American Society for Testing Materials (1). The analysis showed the sample to be composed of 60.8 percent copper, 36.7 percent zinc, and 2.5 percent lead. This analysis showed that the honeycombed material had lost 0.4 of a percent of lead (or about 14% of the lead originally present). The fracture of the specimen showed a grouping of gas holes located on one side of the specimen as shown by the macrograph in

Figure 10. This grouping of holes undoubtedly set up a stress concentration within the specimen during loading and resulted in low strength of the bond. For these reasons specimen

31 was also

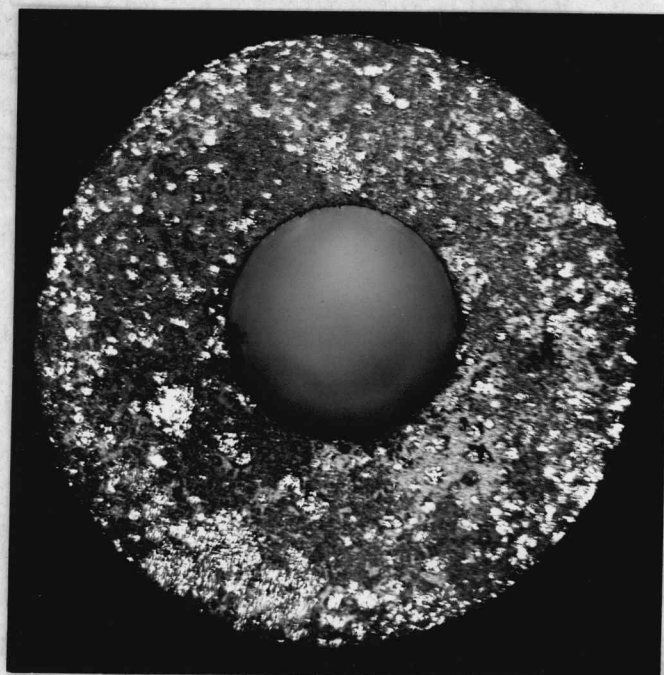


Figure 10 Macrograph showing the Fracture of Specimen 31

excluded from the plot shown in Figure 9.

Series 2 was run holding the temperature, vacuum, and time constant and varying the pressure applied in 100 pound steps. A graph of the ultimate strength obtained in the bond versus the bonding pressure applied is shown in Figure 11. This series was necessarily limited to 5 specimens and therefore the values obtained were not verified.

Series 3 was composed of 3 specimens and was designed to at least partially evaluate the vacuum employed in series 1 and 2. A graph of the ultimate tensile strength of the bond versus the vacuum employed is shown in Figure 12.

It should be pointed out that the tabulated results of the tensile tests give no values of ultimate tensile strength for several of the specimens. These specimens although bonded together upon removal from the containers, fractured under light loads applied during the machining operations. Although these bonds had very little strength it would be erroneous to list their ultimate tensile strengths as 0 pounds per square inch.

The metallographic specimens were processed by the standard methods of polishing and the photomicrographs of a majority of the specimens are shown in Figures 13 to 20, inclusive. These photomicrographs are shown in

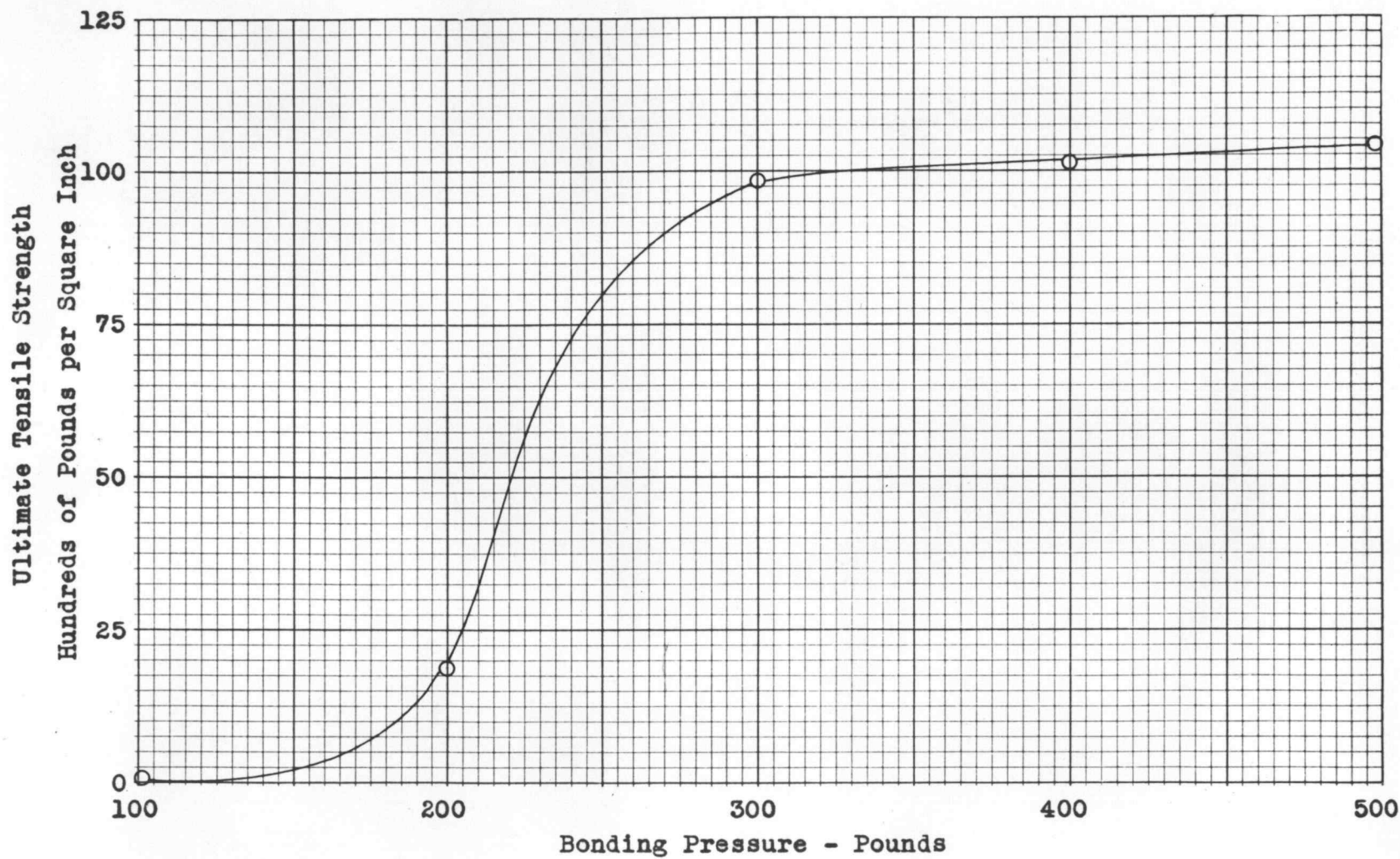


Figure 11 Graph of Specimen Series Two

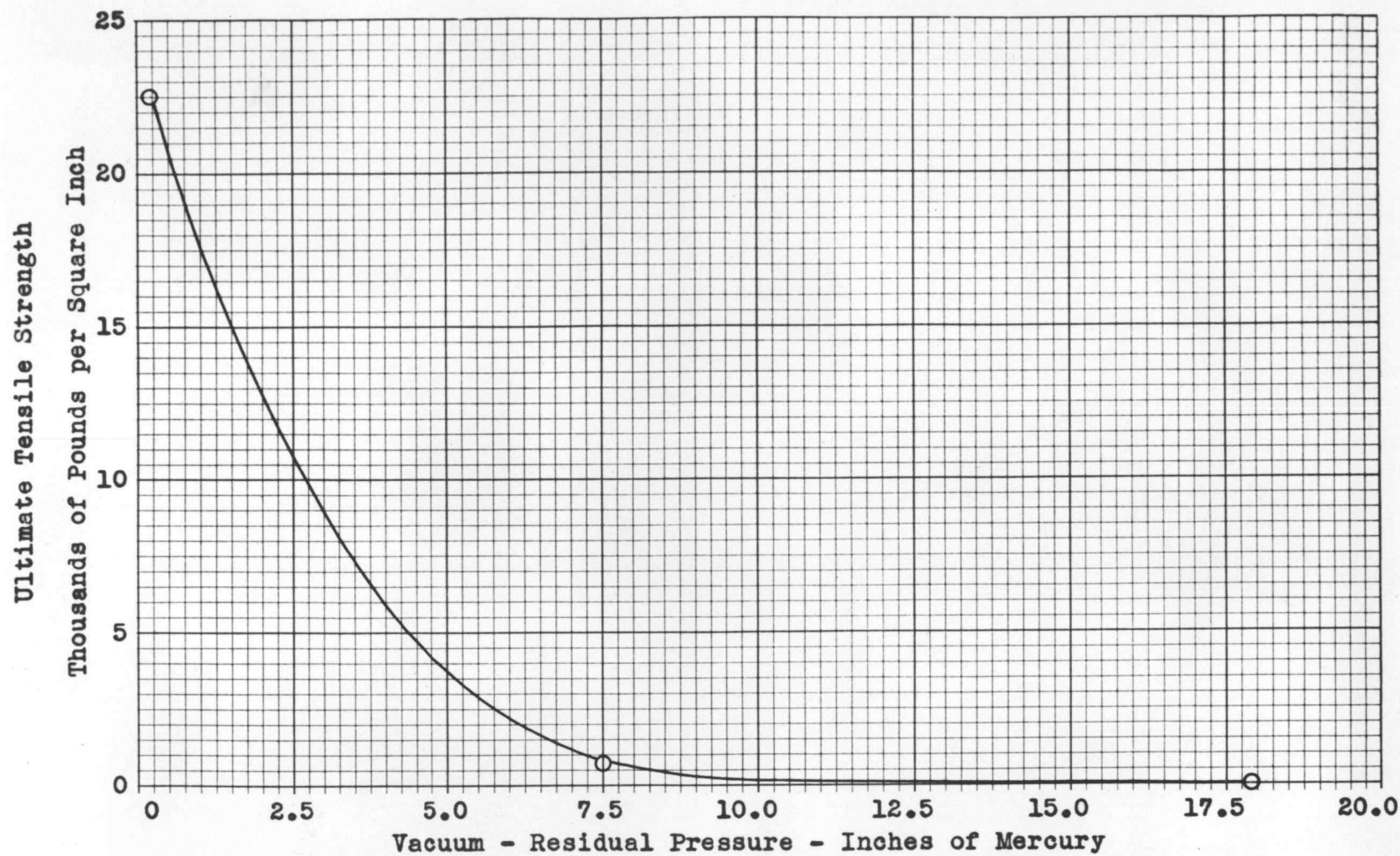


Figure 12 Graph of Specimen Series Three

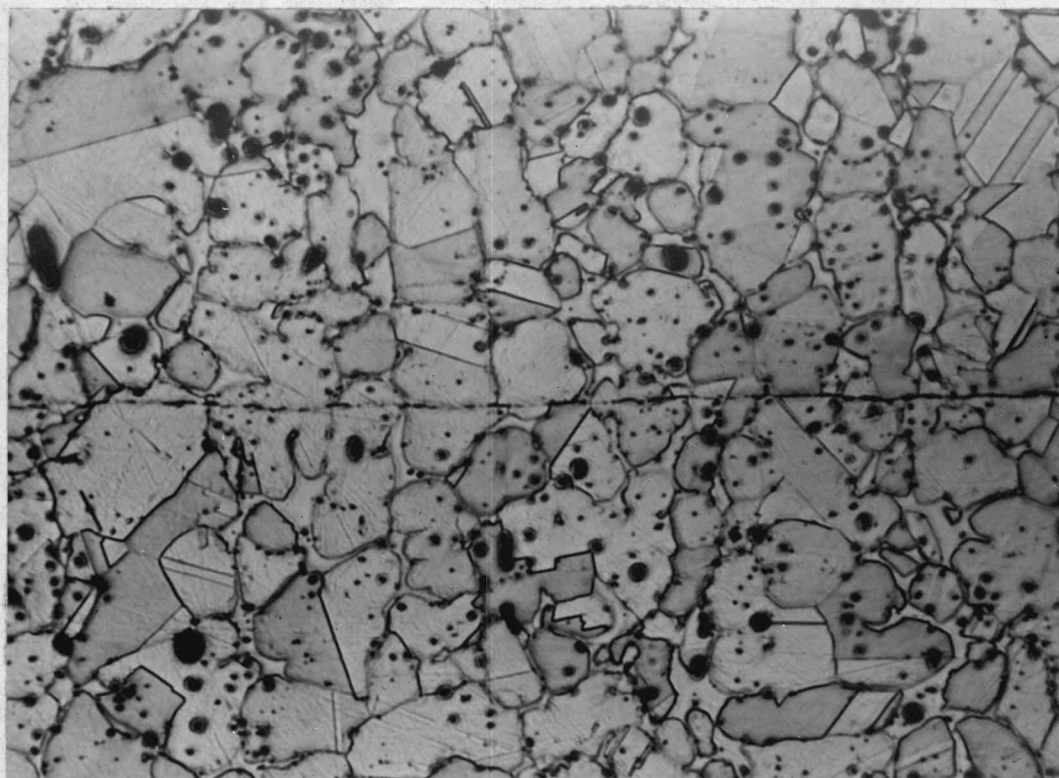


Figure 13 Specimen 25

chronological order with respect to bonding temperature and they represent magnifications of 200 diameters. The interface of the specimens extends horizontally through the center of the photomicrographs. Obviously each photomicrograph shows only a minute portion of the bond, however, the writer did attempt to show representative structures. All the metallographic specimens of the bonds produced were etched with a solution containing 43 cubic centimeters of ammonium hydroxide and 7 cubic centimeters of hydrogen peroxide.

Specimen 25 was bonded to determine the effect of a transformation on the bond produced. Although the interface is clearly defined it is apparent that it is bridged in several places by trans-interface grain growth. From the photomicrograph it appears that a reorganization of the structure has occurred. The presence of a large number of small grains suggests that a partial recrystallization might have taken place. However, the total deformation during loading was only $1/16$ of an inch. This amount is equivalent to approximately 2 percent reduction in the original length. Part of this 2 percent reduction was consumed in the initial deformation of the container, therefore, it seems probable that the maximum amount of deformation actually absorbed by the specimen did not exceed $1\frac{1}{2}$ percent. Many authorities, including Mehl (7), state that recrystallization will not take place at any temperature unless the cold work experienced by the specimen is at least 7 percent. If insufficient cold work is present to bring about recrystallization at some definite temperature, grain growth will occur with applications of intense heat. For these reasons it seems improbable that recrystallization due to hot or cold working occurred. However, it does seem probable that the reorganization of the grain structure was due to the transformation. The presence of the small grains would tend

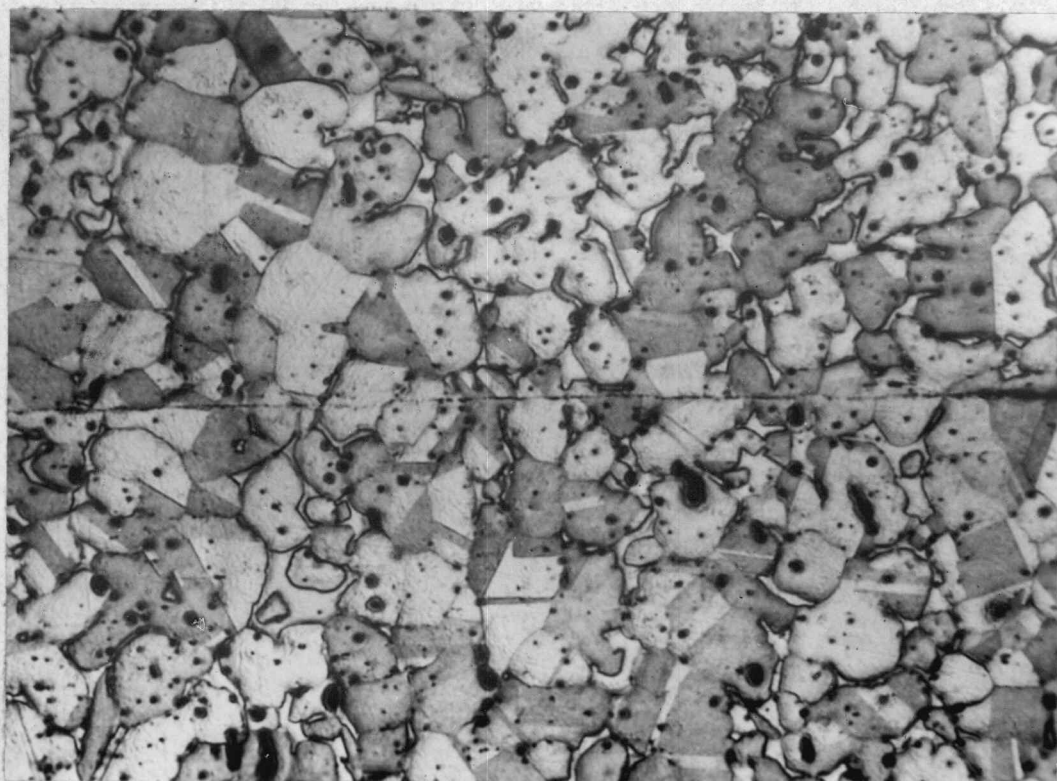


Figure 14 Specimen 14

to indicate that the transformation was not complete.

The photomicrograph of specimen 14 also showed a large number of small grains to be present in the vicinity of the interface. From the standpoint of the temperatures involved in producing the bonds in this specimen and in specimen 25, recrystallization due to hot work seems more probable. This specimen was not loaded for bonding until a temperature of 786 C was reached. Initial recrystallization undoubtedly occurred before this temperature was reached due to the 20 percent cold work present in the

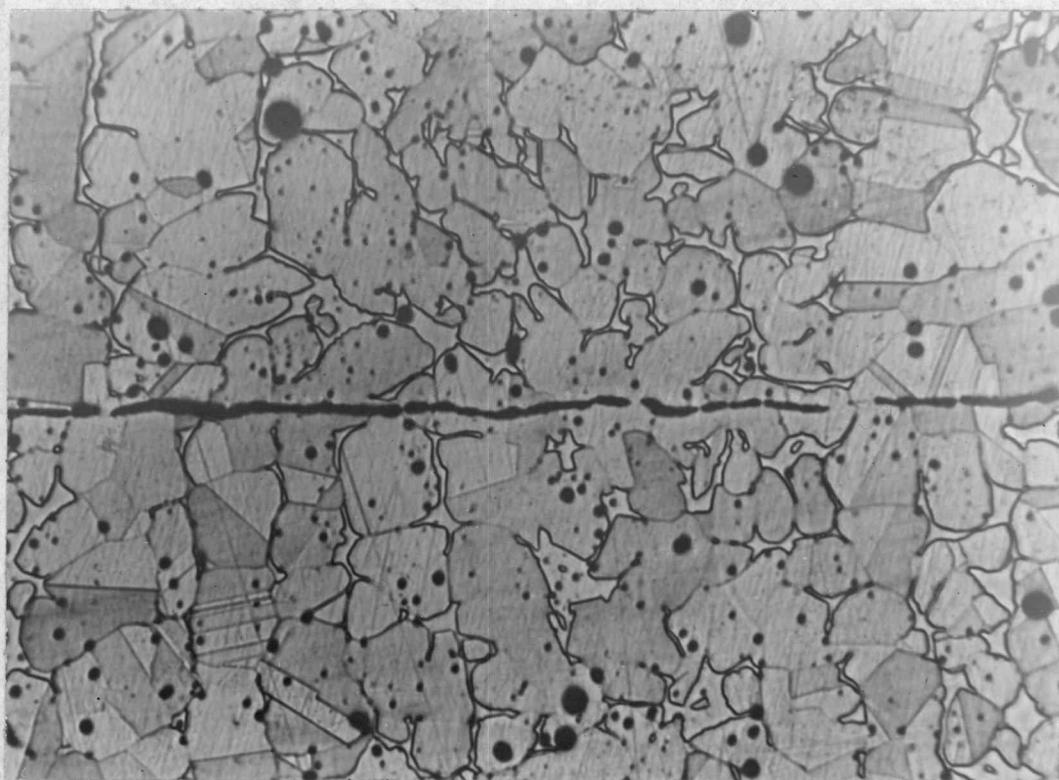


Figure 15 Specimen 13

original brass rods. However, this recrystallization could not have affected the interfaces as a unit because they certainly were not close enough together to allow any diffusion to take place across the interface. Therefore, if recrystallization did have an effect upon the bond produced it was due to a subsequent recrystallization brought about by the 1.5 percent hot work experienced by the specimen during loading. As noted before this seems unlikely. The interface is bridged in several places by trans-interface grain growth. The oxides present within these grains are

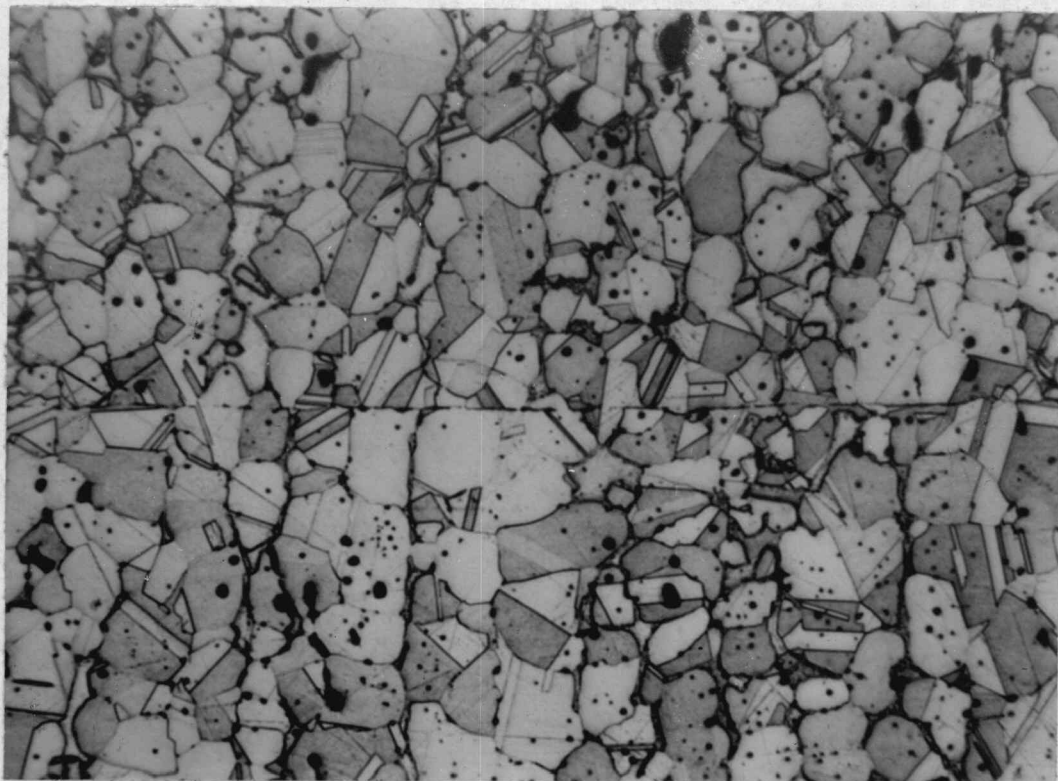


Figure 16 Specimen 19

not completely dispersed.

The interface is clearly visible in the photomicrograph of specimen 13, however, it is bridged by grains in several places. The interface seems to be exceptionally wide in this case and consequently it is believed that it represents a portion of the bond at the extreme edge of the bonded area.

The photomicrograph of specimen 19 shows the interface to be bridged in several places and the oxides present within these grains seem to be quite well dispersed.



Figure 17 Specimen 15

The dispersal of an interface oxide film is also believed to be a diffusion process. The deformation of this specimen under loading also was 1.5 percent. Therefore, recrystallization due to hot work during loading seems improbable.

The photomicrograph of specimen 15 shows a definite increase in the amount of trans-interface grain growth present over that present in the preceding specimens. The oxides present in several of the bridging grains are completely dispersed. No substantial evidence is present to

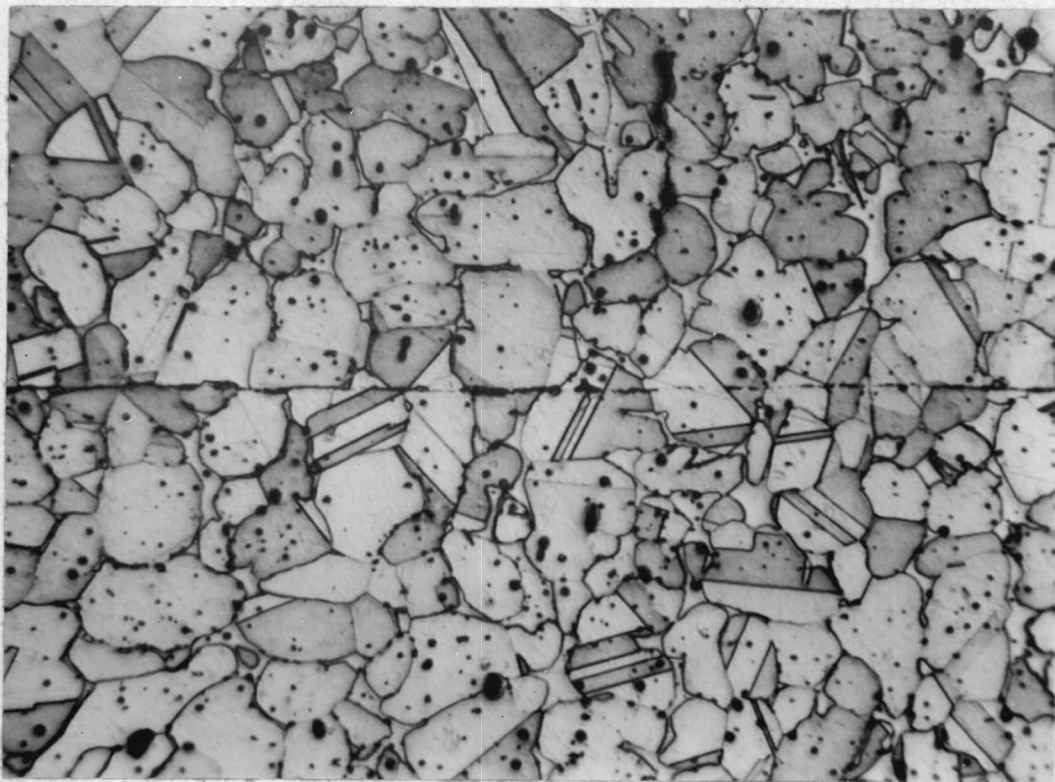


Figure 18 Specimen 18

support the occurrence of recrystallization during bonding due to the application of sufficient hot work.

The photomicrograph of specimen 18 also shows a large amount of trans-interface grain growth. As verified by the tensile tests this increase in grain growth across the interface is accompanied by a gain in the tensile strength of the bond. Numerous small grains are present to suggest the possibility of partial recrystallization during bonding due to hot work imposed on the specimen during loading. The oxides present within the bridging grains

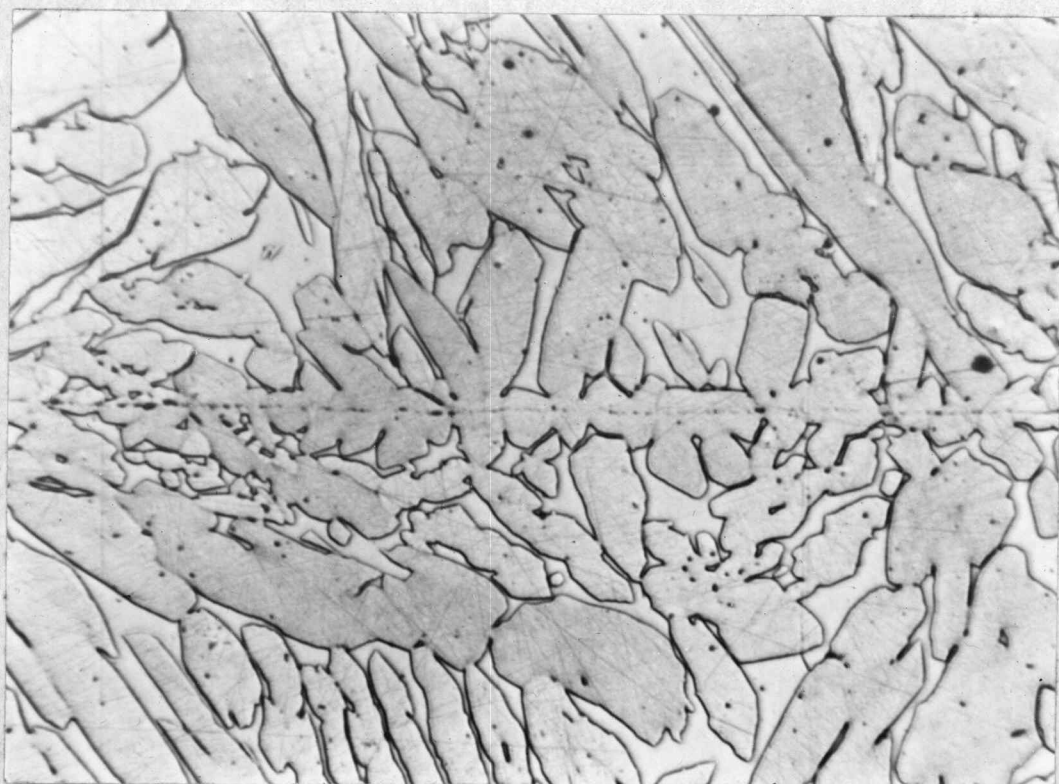


Figure 19 Specimen 11

are well dispersed as in preceding specimens.

The photomicrograph of specimen 11 shows the interface to be bridged almost continuously by trans-interface grain growth. The oxides are not completely dispersed, however. The deformation during the loading of this specimen for bonding was measured as approximately 3 percent. The grain size is quite large in comparison with that of preceding specimens and this would tend to indicate that grain growth occurred in the absence of recrystallization. However, it might also indicate that

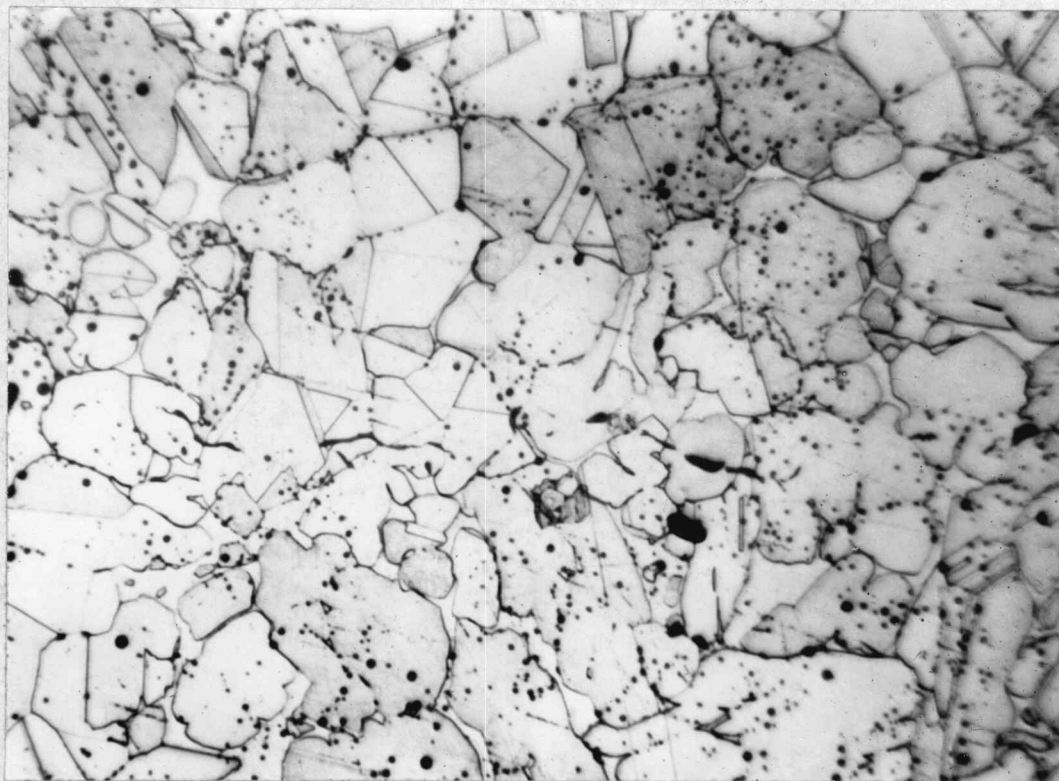


Figure 20 Specimen 31

the conditions during bonding were satisfactory for producing a critical deformation which resulted in a large grain size. A semi-dendritic structure does exist in some portions of the metallographic specimen. Due to the presence of lead in the brass this material might well fail to conform to the basic rules of recrystallization, cold work and hot work. If a specimen were hot worked a sufficient amount at a temperature above the normal recrystallization temperature of the material, recrystallization would be spontaneous. From the photomicrograph it

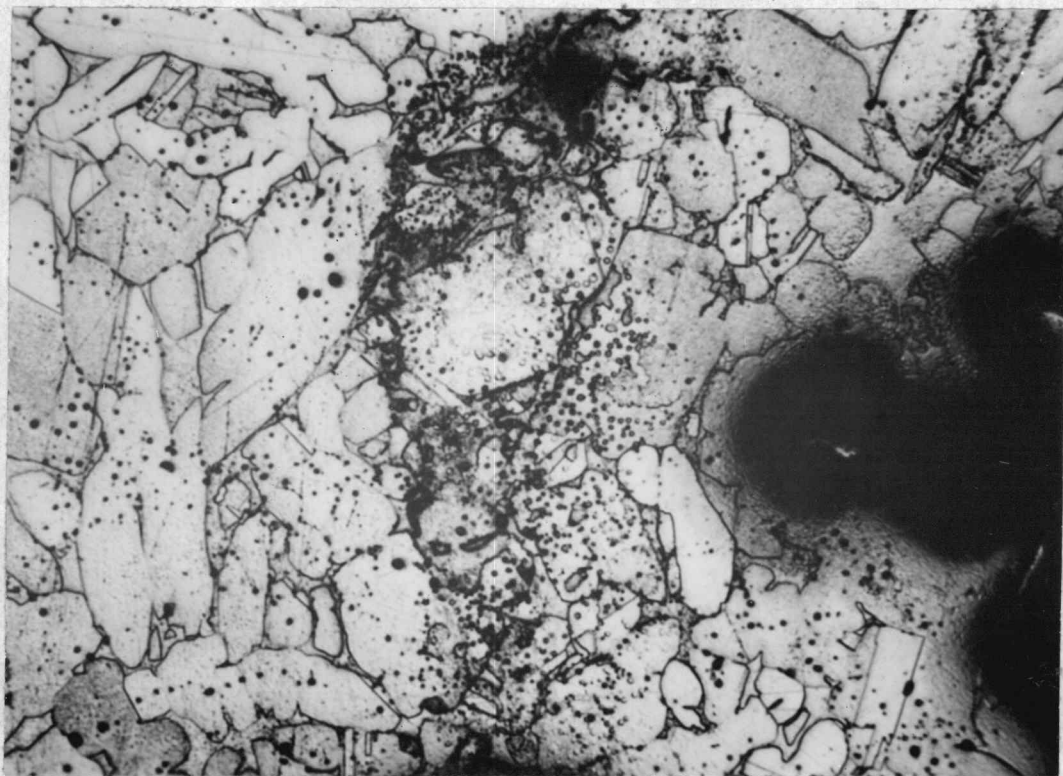


Figure 21 Specimen 31

is evident that if recrystallization did occur during bonding, it was fully completed.

From the photomicrograph of specimen 31 (Figure 20), it is evident that the interface is completely eliminated and that the grains across the interface are indistinguishable from those of the parent metal. The segregation and subsequent vaporization of the lead, as described under physical testing of the specimens, was responsible for the low strength characteristic of this specimen. It is the writer's belief that the solid phase bond between the

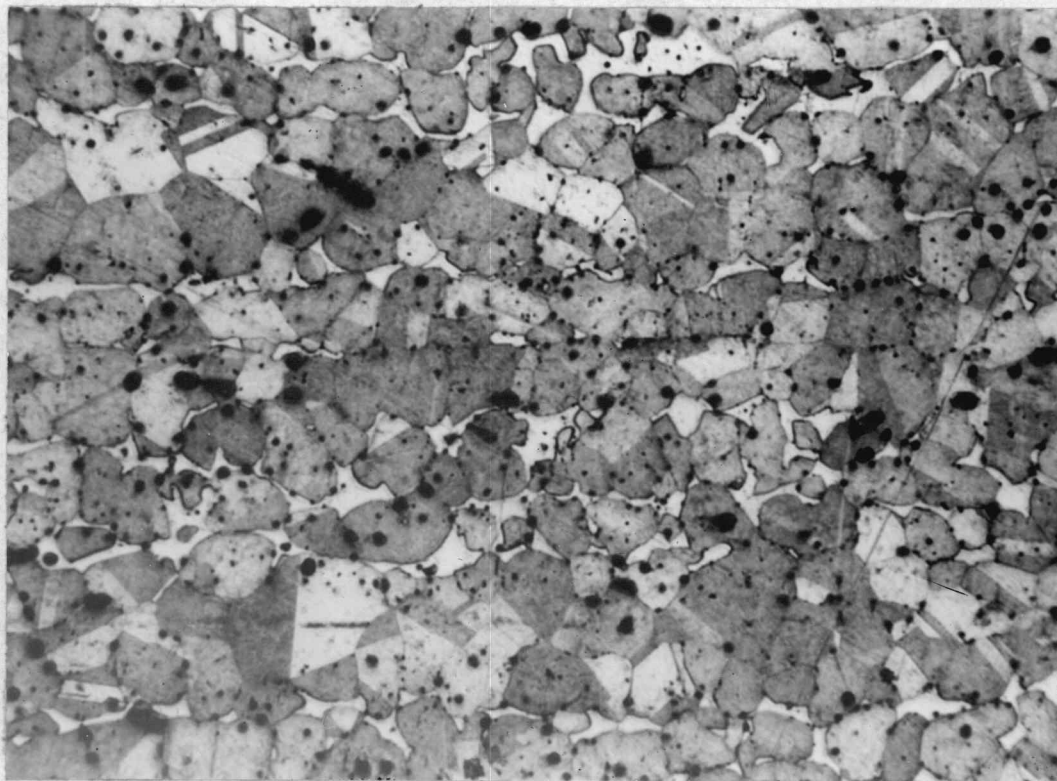


Figure 22 Specimen 28

brass specimens, neglecting the gas holes, is equivalent in strength to that of the parent metal. Due to the loss of area and the stress concentrations set up by the gas holes, the tensile test of this specimen did not verify this belief.

Figure 21 shows another portion of specimen 31. From this photomicrograph the lead segregation and its subsequent vaporization are obvious.

Figure 22 shows a photomicrograph of specimen 28. This specimen was used, as described under physical

testing, to determine the strength of the parent metal when treated exactly the same as specimen 11. This specimen was not bonded but a slight amount of lead segregation did occur.

SUMMARY AND CONCLUSIONS

The mechanism of solid phase bonding seems to be a process of atom diffusion across the interface. It seems logical to expect the laws of diffusion to apply to this atom transfer, however, the necessary information for checking such a hypothesis is not available even in the latest references.

The experiments conducted in this thesis tend to support the basic diffusion theory whereby the diffusion coefficient is believed to increase rapidly when the melting point temperature is approached. An increased diffusion coefficient would undoubtedly result in a more perfect solid phase bond.

The experiments also indicated that any transformation plays an important role in trans-interface crystal growth. This phenomenon also increases the diffusion coefficient and can be expected to provide trans-interface crystal growth at temperatures often far below that required when no transformation takes place.

The dispersion of interface oxide films can be completed by heating to a sufficient temperature and then maintaining this temperature for a prescribed period of time. In these experiments complete diffusion of the films was observed in the specimen which was bonded at a temperature of 870 C. It is commonly agreed that any upsetting action brought about by the loading of the interfaces will tend to break up the oxide films and to further expedite their complete dispersion. In these experiments the specimens were designed to minimize any upsetting of the interfaces and in this manner to provide a more direct method of evaluating the importance of an oxide film at elevated temperatures. The vacuum employed in these experiments was evidently just sufficient to prevent the formation of an oxide film which would not have been completely dispersed under any of the combinations of conditions employed. It seems quite certain that if a diffusion pump or an inert atmosphere had been employed, the bonds obtained with similar experiments would have been perfect over a wider range of conditions. However, in the opinion of the writer the elimination of the use of a vacuum and of a specimen container would also have resulted in the development of various experimental difficulties.

These experiments showed that temperature was the predominant factor in solid phase bonding. The pressure

applied for bonding and the time interval required for bonding are factors of lesser importance. No attempt was made in these experiments to evaluate the effect of upsetting on a solid phase bond. Deformations were measured for operational reasons and also to afford data in recrystallization studies.

These experiments also indicate that if sufficient pressure is applied to bring the interfaces close enough together to permit adequate diffusion across the interface, a greater pressure will only result in severe deformations which are not necessary for obtaining perfect bonds. The maximum pressures applicable in solid phase bonding are limited due to the loss of strength of the material being bonded at elevated temperatures. With decreasing pressure the maximum temperature that can be employed is limited only by the possible evolution of a liquid phase.

These experiments showed that the time required for trans-interface grain growth is very short in comparison to that required for the complete dispersion of the oxide film. The various photomicrographs contained in this thesis verify this hypothesis. Several photomicrographs display structures showing a bridging grain completely formed but still containing oxide inclusions. The time required for dispersing the oxide films at the interfaces is obviously dependent upon both time and temperature.

In the opinion of the writer the most important question remaining to be answered conclusively, by a large amount of similar experimental work, is the determination of the actual process of diffusion during solid phase bonding. Do the grains on each side of the interface, even though their crystal lattices are somewhat distorted, undergo a slight change resulting in a combining effect or is there a general upheaval of the existing grain structure whereby the diffusion coefficient is increased many fold in comparison to that in the aforementioned change? Until this question is answered, the true mechanism of solid phase bonding can only be postulated.

The equipment constructed for the experimental work proved very satisfactory and no major difficulties were encountered. However, certain idiosyncrasies were apparent.

The presence of a specimen container, deemed necessary to the experiments, did influence, at least slightly, the actual loads received by some of the specimens which were bonded. No method of elimination or complete correction could be devised. However, all the possible remedies that could be formulated by the writer were employed to diminish its ultimate effect on the data obtained.

A control circuit applied to the furnace, enabling closer control of the temperatures involved, might have proven beneficial although another portion of the available time for this thesis would have been exhausted in the construction of such additional equipment.



ADVANCE BOND

W. L. BROWN Paper

BIBLIOGRAPHY

1. American Society for Testing Materials. Standard Methods of Chemical Analysis of Brasses. Designation E 36-45. American Society for Testing Materials, 1946. p 185-186.
2. American Society for Testing Materials. Standard Specifications for Free Cutting Brass Rod and Bar for use in Screw Machines. Designation B16-46. Part I-B, 1946. p 101.
3. American Society for Testing Materials. Standard Methods of Tensile Testing of Metallic Materials. Designation E8-46. Part I-B, 1946. p 296.
4. Durst, George. A Few Observations on Solid-Phase Bonding. Metal Progress, Vol 51, Jan 1947. p 97-101.
5. Heyer, R. H. Engineering Physical Metallurgy. D. Van Nostrand Co, 1939. p 105-122.
6. Kinzel, A. B. Adams Lecture - Solid-Phase Welding. The Welding Journal, Vol 23, Dec 1944. p 1124-1143.
7. Mehl, R. F. Recrystallization. American Society for Metals (Metals Handbook), 1948 Edition. p 259-263.
8. Smith, Cyril. History of Powder Metallurgy.
9. Wilkins, R. A. and Bunn, E. S. Copper and Copper Base Alloys. McGraw-Hill Book Co, 1943. p 334.