# BERYLLIUM, ITS POTENTIALS AND PROBLEMS

| | |
|---|---|
| Chairman | Mr. I. Perlmutter |
| Speaker | Lt S. S. Christopher |
| Panel Members | Mr. H. Black<br>Mr. R. Geyton |

# BERYLLIUM RESEARCH AND DEVELOPMENT

Lt S.S. Christopher

Directorate of Materials and Processes, ASD

## Introduction

In reviewing the technology of beryllium that has been generated over the past three years, since the last Air Force Materials Symposium, we find that there has been significant advancement in the four areas of: (1) brittleness (2) joining (3) fabrication of wrought products, and (4) evaluation of the structural integrity of beryllium.

Before we discuss in detail the areas mentioned above, it is important that we review briefly the properties of beryllium that make it attractive to the Air Force as a structural material. The modulus to density ratio of beryllium surpasses that of any other metallic material and this combined with its heat capacity, thermal conductivity, dimensional stability, and relatively high melting point make the metal a necessity for future space systems. No other material has this unique combination of properties to offer design engineers.

Along with the many advantageous properties of beryllium, we find that there are many factors preventing the widespread utilization of the metal. These can be readily summarized as follows: (1) brittleness; (2) joining; (3) preferred orientation in wrought products; (4) casting; and (5) toxicity. It might be well to mention that although the toxicity problem is real, techniques and procedures have been established to the extent that almost any metallurgical process can be applied to beryllium without any harmful effects on personnel.

In view of the great potential of the metal, numerous programs have been initiated by the Air Force, Navy, and the Atomic Energy Commission. These organizations, although having different end items in mind, find that the problems associated with beryllium are basic to all three.

Listed below are specific problems investigated in Air Force beryllium programs, which will be discussed. The problems will be reviewed also as they affect the research and development programs by the other Government agencies mentioned.

| BRITTLENESS | JOINING | PROCESSES AND WROUGHT PRODUCTS | STRUCTURAL INTEGRITY |
|---|---|---|---|
| Distillation | Fusion Welding | Rolling | Box Beam |
| Iodide Decomposition | Resistance Butt Welding | Forging | Thermantic Panels |
| Aging and Strain Aging | Ultrasonic Welding | Extrusion | Design Handbook |
| Dislocations | Resistance Spot Welding | Casting | |
| Splat Cooling | Brazing | Roll Forming | |
| Oxide Distribution | | | |
| Surface Effects | | | |
| Increasing Yield Strength | | | |

## Brittleness

Brittleness is the predominant factor restricting extended use of beryllium as a structural material. Although many theories have been postulated as to the probable cause of this brittleness, none have been substantiated. However, progress is being made through Government sponsored research and development, coupled with a genuine industrial interest in sustaining an attack on this and related problems.

One of the postulations advanced is that impurities play the major role in causing brittleness in beryllium. Investigations of the past year seem to support this theory. Purification studies have been initiated and three different approaches are being employed: zone refining, distillation, and iodide decomposition.

Zone refining studies under Navy sponsorship at the Franklin Institute (1) have produced beryllium single crystals with ductilities never before attainable. The mechanical property data obtained on these crystals are shown in table 1.

Polycrystalline beryllium being produced by distillation has given purity never before attainable in the metal.* Table 2 is an example of the purity obtained by this technique as compared with Pechiney beryllium. A complete mechanical property evalution on polycrystalline distilled beryllium will be completed in the near future.

Although beryllium iodide decomposition has not been successful in producing quantities of beryllium sufficient for X-ray or chemical analysis, much has been learned concerning the preparation of pure beryllium iodide, reaction materials, and kinetics of the decomposition. A series of experiments are now under way using a static system in a beryllium reaction chamber. The variables being studied are the temperature gradient between the reaction bulb and the hot filament, concentration of $I_2$, and the geometry of the reaction chamber.

Other programs under investigation which tend to support the purification theory are aging and yield point phenomena and dislocation studies in beryllium. Beryllium, solution treated and aged at various temperatures, shows a wide variance in the tensile properties and also the occurrence of a strong yield point. Strain aging studies have given a rough value for the activation energy for return of the yield point and this value is around 50 kilo cal/gm mole. This value is high and it would be expected that a substitutional element was causing this phenomenon.

Wilsdorf (2) strengthens the work of Gelles (2,3) above through actual observation of impurity clusters by electron transmission microscropy. These clusters observed in annealed Pechiney beryllium seem to preferentially precipitate along subgrain boundaries and dislocations (figure 1). This investigation is the first reported direct observation of these precipitates.

Electron transmission techniques (2) are being applied to study the distribution of oxide in hot pressed and wrought beryllium. Replica techniques have been developed and surface-oxidized vacuum melted Pechiney flake is being employed as the starting material to study the oxide films. No conclusive results on this program are available.

---

* No analysis is available as yet on Franklin Institute zone refined bars.

## Other Brittleness Investigations

Surface defects such as cracks and twinning are being evaluated according to the role they play in crack initiation and propagation in beryllium sheet. Metallographic examination of hot pressed sheet in the as-received condition has shown cracks to depths of 0.005 inch and twins to a depth of 0.01 inch. Surface and annealing treatments are being exploited in an attempt to remove the surface imperfections.

Although very little alloy development on beryllium is being supported by the Air Force, a program (2) was initiated to increase the yield strength of beryllium sheet using two approaches: by varying conditions of fabrication and by alloying with copper. The results of this investigation are shown in tables 3 and 4. In comparing this data, we find that copper is a potent strengthening agent, a one percent addition, increases the yield strength from 20 to 50 percent.

## Joining

Another problem area in the development of beryllium as a structural material is joining. Fabrication of structural components involves the joining of beryllium to other materials as well as to itself.

Fusion welding (2) of beryllium has resulted in crack free welds in sections under 1/4-inch thick using Metal-arc Inert Gas (MIG) and Tungsten-arc Inert Gas (TIG) processes. Mechanical properties of the welds produced are lower than the properties of the parent metal and techniques such as roll planishing to impart strength to the welds are now being attempted with some success. The primary problems in fusion welding are: (1) poor penetration due to the high heat conductivity of beryllium; (2) excessive grain growth in the weld and heat affected area; (3) development of appropriate filler material; and (4) fixturing.

Resistance butt welding studies are being performed on beryllium 5/8-inch diameter rod. Although welds have been made, sufficient data are not available to evaluate this process.

Ultrasonic welding (2) of beryllium sheet ranging in thickness from 0.010 to 0.013 inch is being attempted. Beryllium to beryllium welds have been made (figure 2), and it should be noted that the grain size in the weld area is not greatly affected by this technique. The limiting factor in this process seems to be the power requirements of the equipment available. To permit joining at a somewhat reduced power setting, interleaf materials are being selected and utilized. The interleaf materials under investigation are molybdenum, zirconium, titanium, gold, and aluminum. A complete analysis of this technique as applied to beryllium is not yet available but the process shows definite promise.

Resistance spot welding (2), now under investigation, consists of making single spot welds on 1 x 1 inch coupons of beryllium at various welding currents while holding all other variables constant. This procedure is repeated for each group of welds made except that one of the variables is changed for each group. This program is in its infancy and no data is now available.

To date, the most reliable method of joining beryllium is considered to be brazing. Silver braze alloys have been developed which give a satisfactory bond with approximately 60 percent of the base metal strength at room temperature.

## Fabrication

The fabrication of beryllium into reliable wrought forms such as sheet, forgings, and extrusions has been actively pursued by the Air Force. Fabrication and processing techniques are of the utmost importance in the adaptation of any material to structural usage. Since the primary utilization of a wrought product is in the form of sheet, let us discuss the current state of the art in beryllium sheet rolling (4).

Techniques have been developed for rolling sheet in sizes 24 in. x 60 in. x .020 in. to .060 in. Blended beryllium powder QMV vacuum hot pressed minus 200 mesh is considered the optimum starting material. The hot pressed block is jacketed in mild steel and bidirectionally rolled between 1400 and 1500°F. Bidirectional or cross-rolled beryllium has mechanical properties in the plane of the sheet within 10 percent of each other, generally the highest strength values are obtained in the final or predominant rolling direction. Table 5 is a statistical analysis of the yield strength and elongation of the sheet produced under the various phases of the rolling program. However, the problem now emerges of the ever-present problem of poor, essentially nonexistent, ductility in the short transverse (thickness) direction of the beryllium sheet material. Although the elongation in the plane of the sheet is sufficient for most structural components, utilization is highly limited due to the inability of the sheet material to withstand biaxial stresses. This is attributed to crystallographic orientation, and one must be familiar with the active modes of deformation in beryllium to understand the effect of orientation on anisotropy.

It would be of considerable value at this point to discuss briefly the attempts made to solve the preferred orientation problem. The objectives of these investigations were to obtain a randomly oriented material and evaluate the effect of random orientation on mechanical properties. However, our need is not only one for which a process or technique must prove successful, but one that could be conveniently adapted to large production capability. The first technique tried can be termed "hot-upsetting", wherein an effect comparable to infinite-directional rolling could conceivably be attained (5). Sheet was produced by pure compression applied along the longitudinal axis of a cylindrical beryllium billet machined out of a hot-pressed block. Crystallographic orientation in sheet produced thusly has the basal plane (0001) essentially parallel to the plane of the sheet and the secondary prism plane $(11\bar{2}0)$ perpendicular but random with respect to any given direction within the plane of the sheet. The short transverse or third dimensional ductility increases from 0.017 through 0.226 percent (in a bidirectionally rolled) to 1.5 through 2.0 percent (in hot-upset) sheet. The strength and elongation in the plane of the hot-upset sheet are lower, as would be expected.

The second approach to the preferred orientation problem was to obtain random orientation through heat treatment (6). Bidirectionally rolled sheet was annealed at various temperatures ranging from 950° to 1200°C for periods of 0.1 to 10 hours at each temperature. The effects of these various annealing cycles on texture and on third dimensional ductility were measured. X-ray analysis was used to establish pole figures to determine basal plane orientation in the annealed sheet. These figures reveal a basal plane shift of approximately 10 degrees from the plane of the sheet. This drop in basal plane pole population perpendicular to the sheet means that the generally parallel basal plane orientation has been disrupted or has become somewhat randomized. The effect of the high temperature annealing on the basal plane (0001) orientation of bidirectionally rolled sheet is shown in figure 3.

The accompanying increase in third dimensional ductility through high temperature annealing compares favorably with that realized by hot-upsetting. Biaxial ductilities obtained through annealing vary between 1.0 and 1.77 percent with the optimum annealing cycle indication of 1050°C for 0.1 hour.

From this discussion it immediately becomes apparent that although both hot-upsetting and high temperature annealing seem to alleviate the preferred orientation problem, the latter is more conventional and appealing. We can foresee the difficulty which arises with respect to sheet size limitations in the hot-upsetting technique. However, the information obtained through this investigation is extremely valuable. Investigation in a new AMC sheet rolling program will utilize the above information, and provisions are being made for the adaptation of the annealing technique developed. A thorough and comprehensive evaluation of this added feature is programmed.

## Evaluation of Structural Integrity

To determine the structural integrity of the bidirectionally rolled sheet produced, many programs have been designed to fabricate and test structural components. These programs have been initiated on theoretical formability, mechanical and physical property evaluation, crack propagation studies, design criteria, coatings, high energy forming processes, etc. Bidirectionally rolled sheet has been supplied to many aircraft companies and research organizations for evaluation under these programs. An example (7) of some of the aforementioned forming and testing that is being accomplished is shown in figure 4.

Results reported from these investigations are as yet very limited and conclusions cannot be drawn. One of the most comprehensive investigations on structural evaluation is in process at the Martin Company (8). The objectives are: to establish a standard measure of merit for characterizing so-called structural beryllium and to determine experimentally the relative superiority of beryllium, in the structural sense. An evaluation is being conducted utilizing hot-upset, hot-pressed, and hot-pressed bidirectionally rolled sheet. Mechanical property evaluation will involve bend tests, notched and smooth tensile tests, bulge tests, compression, and box beam structure testing. Specimens of varying widths and gauge thickness will be used in the bend ductility testing.

Although the evaluation is not complete, considerable progress has been made. Box beams have been fabricated from all three sheet materials and tested by constant moment bending until fracture. The beam and fracture data are shown in figure 5. The designations are: bidirectionally rolled, hot-pressed and hot-upset. Until all the evaluations are complete and the data compiled and correlated with tests and materials, we cannot draw any concrete conclusions concerning the structural merit of beryllium sheet.

## Future Recommendations in Beryllium Research and Development

Even with the brief review presented herein, we can readily see that many areas of beryllium research and development necessitate further investigative effort. In addition to problems discussed, attention must also be given to the compilation of design data and, ultimately, testing of structural components for actual use in specified systems. Although, the most promising developments concerned with ductility are centered around purification, further evaluation for conclusive proof that this is the answer and that reproducibility is assured must be accomplished. Results on the single crystal work must be confirmed and the feasibility of zone refining must be substantiated by similar results on polycrystalline material. Once these are resolved and the cause of impurities is isolated, concentrated effort may be expended on developing techniques for producing the degree of purity needed on a commercial scale.

Proven success in the above programs would precipitate further efforts such as study of deformation modes and characteristics, recrystallization and grain growth behavior, alloying characteristics, further development of joining methods, and evaluation of physical and mechanical properties of the high purity material.

If the above course is pursued, another aspect for consideration is the possibility of limited improvement or negative results through purification of beryllium. This would, in effect, dictate the need for a critical review of the basic research effort, and the initiation of new programs designed to answer questions of a more developmental nature. These programs would include studies on the effect of BeO content and distribution on the mechanical properties, aging studies to determine the cause of certain phenomena under certain conditions of time, temperature, and work, and dislocation studies because of the very nature of the data they produce. Results from all these investigations will have to be evaluated and correlated with the specific properties each imparts on the material.

The future of beryllium research and development hinges on a relatively few highly oriented studies currently under way. Yet, great strides have been made, and even more encouraging is the fact that each day more and more people connected with the research and development effort in beryllium are becoming more consistent in their reasoning and in their approaches to the existing problems.

## REFERENCES

1. Herman, M., Spangler, G. E., and Hein, E., "The Preparation of High Purity Beryllium and the Study of Its Flow and Fracture Properties," Final Report on Contract NOas 59, 6242-c, June 30, 1959 to June 30, 1960.

2. "Beryllium Research and Development Program," Nuclear Metals, Inc., Contract AF 33(616)-7065.

3. Gelles, S. H., Pickett, J. J., and Wolff, A., "Some Recent Advances in Beryllium Metallurgy," Nuclear Metals, Inc., TJ-31, August 31, 1960.

4. Wikle, K.G., Armstrong, J. W., and Perrin, H. N., "Development of Beryllium Sheet Rolled Flat to Gauge," Final Report, Contract AF 33(600)-35829, March 1960.

5. Yans, F. M., "Third Dimensional Ductility and Crack Propagation in Beryllium Sheet," Final Report, Contract AF(30-1)-1565.

6. Yans, F. M., Wolff, A. K., and Kaufman, A. R., "Development of Randomly Oriented Wrought Beryllium Sheet," WADD Technical Report 60-403, October 17, 1960.

7. Aeronca Manufacturing Corporation, "Development of Beryllium Composite Structures," Contract AF 33(616)-7050.

8. Giemza, C. J., "Relation of Mechanical Behavior of Beryllium Sheet to Fabrication Variables," Final Report, Contract AF 33(600)-40648, March 1961.

## TABLE I

### MECHANICAL PROPERTIES OF ZONE REFINED SINGLE CRYSTALS PRODUCED BY THE FRANKLIN INSTITUTE

| NO. OF PASSES | ORIENTATION | REDUCTION IN AREA | GLIDE STRAIN | ELONGATION IN 3/8" | CRITICAL RESOLVED SHEAR STRESS |
|---|---|---|---|---|---|
| 5 | Basal Plane 20° to Tension Axis | 20% | 60% | 25% | 1100 psi |
| 8 | Basal Plane 45° to Tensile Axis | 45% | 156% | 56% | 520 psi |

## TABLE 2

### CHEMICAL ANALYSIS OF BERYLLIUM

|  | ELEMENT, ppm |  |  |  |  |
|---|---|---|---|---|---|
|  | Fe | Ni | Cr | Mn | C |
| PECHINEY FLAKE | 140 | 45 | 6 | 15 |  |
| VACUUM MELTED FLAKE | 275 | 100 | 10 | 20 | 20 |
| SUPER PURE PECHINEY FLAKE | 45 | 8 | 35 | 5 |  |
| DISTILLATION NO. 5 |  |  |  |  |  |
| BOTTOM | <5 | <2 | <2 | <5 | 30 |
| MIDDLE | <5 | <2 | <2 | <5 |  |
| DISTILLATION NO. 6 |  |  |  |  |  |
| BOTTOM | 4.0 | 1 | 3 | <5 | 35 |
| MIDDLE | 2.5 | 2 | 2 | <5 | <10 |
| TOP | 3.5 | 5 | <1 | 10 |  |
| DISTILLATION NO. 7 |  |  |  |  |  |
| BOTTOM | 1.5 | 1 | <1 | 5 |  |
| MIDDLE | 3.0 | 1 | 1 | 5 |  |
| TOP | 3.0 | 4 |  | 15 |  |

## TABLE 3

### MECHANICAL PROPERTIES OF UNALLOYED BERYLLIUM SHEET UNDER VARIOUS CONDITIONS OF FABRICATION

| ROLLING TEMP (°F) | ROLLING REDUCTION | FINISH PASSES | 0.2% OFFSET YIELD STRENGTH (psi) | ULTIMATE TENSILE STRENGTH (psi) | PERCENT ELONGATION |
|---|---|---|---|---|---|
| 1800 | 1.5 : 1 | NONE | 30,200 | 49,100 | 3.6 |
| 1800 | 3 : 1 | NONE | 35,100 | 61,600 | 10.9 |
| 1800 | 6 : 1 | NONE | 42,000 | 67,300 | 8.0 |
| 1600 | 1.5 : 1 | NONE | 38,600 | 59,800 | 5.8 |
| 1600 | 3 : 1 | NONE | 41,000 | 71,600 | 16.1 |
| 1600 | 6 : 1 | NONE | 37,700 | 65,300 | 16.6 |
| 1400 | 1.5 : 1 | NONE | 42,800 | 63,600 | 6.3 |
| 1950 | 3 : 1 | NONE | 32,300 | 61,800 | 11.3 |
| 1950 | 3 : 1 | 5% at 1400°F | 30,400 | 55,300 | 6.9 |

## TABLE 4

### MECHANICAL PROPERTIES OF Be-1 w/o Cu ALLOY SHEET UNDER VARIOUS CONDITIONS OF FABRICATION

| ROLLING TEMP (°F) | ROLLING REDUCTION | FINISH PASSES | 0.2% OFFSET YIELD STRENGTH (psi) | ULTIMATE TENSILE STRENGTH (psi) | PERCENT ELONGATION |
|---|---|---|---|---|---|
| 1800 | 1.5 : 1 | NONE | 44,000* | 59,000* | 4.6* |
| 1800 | 3 : 1 | NONE | 42,300 | 66,400 | 8.2* |
| 1800 | 6 : 1 | NONE | 51,300 | 72,200 | 10.2 |
| 1600 | 1.5 : 1 | NONE | 54,500 | 63,000 | 2.5 |
| 1600 | 3 : 1 | NONE | 52,500 | 76,600 | 10.1 |
| 1600 | 6 : 1 | NONE | 57,000* | 81,300* | 17.2* |
| 1400 | 1.5 : 1 | NONE | 59,800 | 69,700 | 2.9 |
| 1950 | 3 : 1 | NONE | 41,900* | 70,900* | 11.2* |
| 1950 | 3 : 1 | 5% at 1400°F | 43,600 | 70,600* | 10.2* |

* AVERAGE OF TWO TESTS

## TABLE 5

### SYNOPSIS OF STATISTICAL ANALYSES OF YIELD STRENGTH AND ELONGATION

| MATERIAL | TESTING DIRECTION | YIELD STRENGTH | | | | |
|---|---|---|---|---|---|---|
| | | HIGH | LOW | AVERAGE | LOWEST * PROBABLE | STANDARD DEVIATION |
| PHASE II | | | | | | |
| LYB - 1081 - 86 | LONG. | 70,300 | 58,400 | 64,900 | 55,900 | 3,000 |
| SINGLE SHEET | TRANS. | 74,700 | 58,400 | 64,200 | 54,600 | 3,200 |
| PHASE III | | | | | | |
| LYS - 1112 | LONG. | 77,000 | 50,900 | 64,500 | 47,700 | 5,600 |
| 46 SHEETS | TRANS. | 80,700 | 52,500 | 65,300 | 48,200 | 5,700 |
| PHASE IV | | | | | | |
| BLEND MATERIAL | LONG. | 58,800 | 37,400 | 48,300 | 34,800 | 4,500 |
| 68 SHEETS | TRANS. | 58,300 | 37,800 | 46,000 | 33,100 | 4,300 |

| | | ELONGATION IN PERCENT | | | |
|---|---|---|---|---|---|
| | | HIGH | LOW | AVERAGE | STANDARD DEVIATION |
| PHASE II | | | | | |
| LYB - 1081 - 86 | LONG. | 19.0 | 5.0 | 11.7 | 4.4 |
| SINGLE SHEET | TRANS. | 27.3 | 5.5 | 15.4 | 5.9 |
| PHASE III | | | | | |
| LYS - 1112 | LONG. | 44.0 | 4.0 | 17.2 | 8.1 |
| 46 SHEETS | TRANS. | 34. | 2.0 | 16.0 | 6.3 |
| PHASE IV | | | | | |
| BLEND MATERIAL | LONG. | 19.5 | 3.5 | 8.8 | 3.5 |
| 68 SHEETS | TRANS. | 22.0 | 2.5 | 8.8 | 4.5 |

* BASED ON THREE (3) STANDARD DEVIATIONS TO INCLUDE 99.73 PERCENT OF ALL POSSIBLE VALUES.

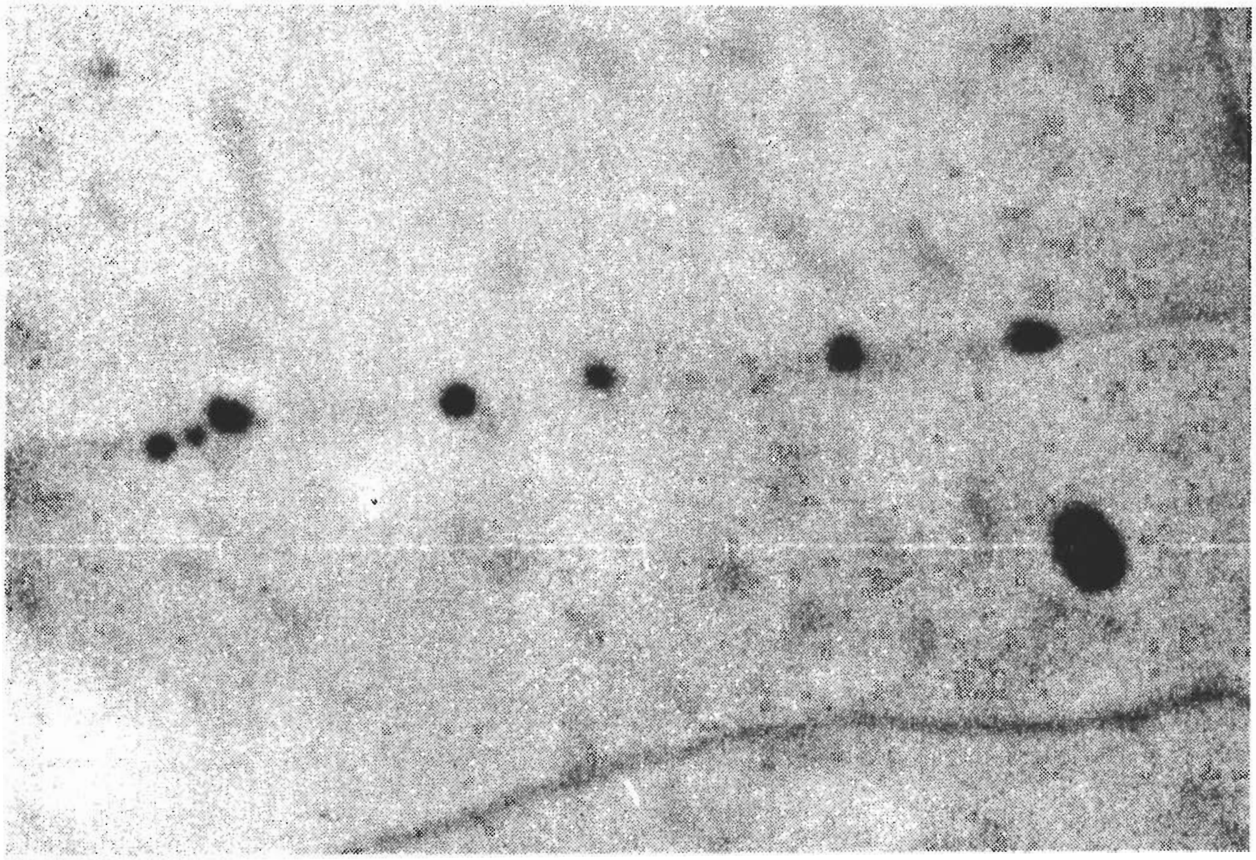


Figure 1.

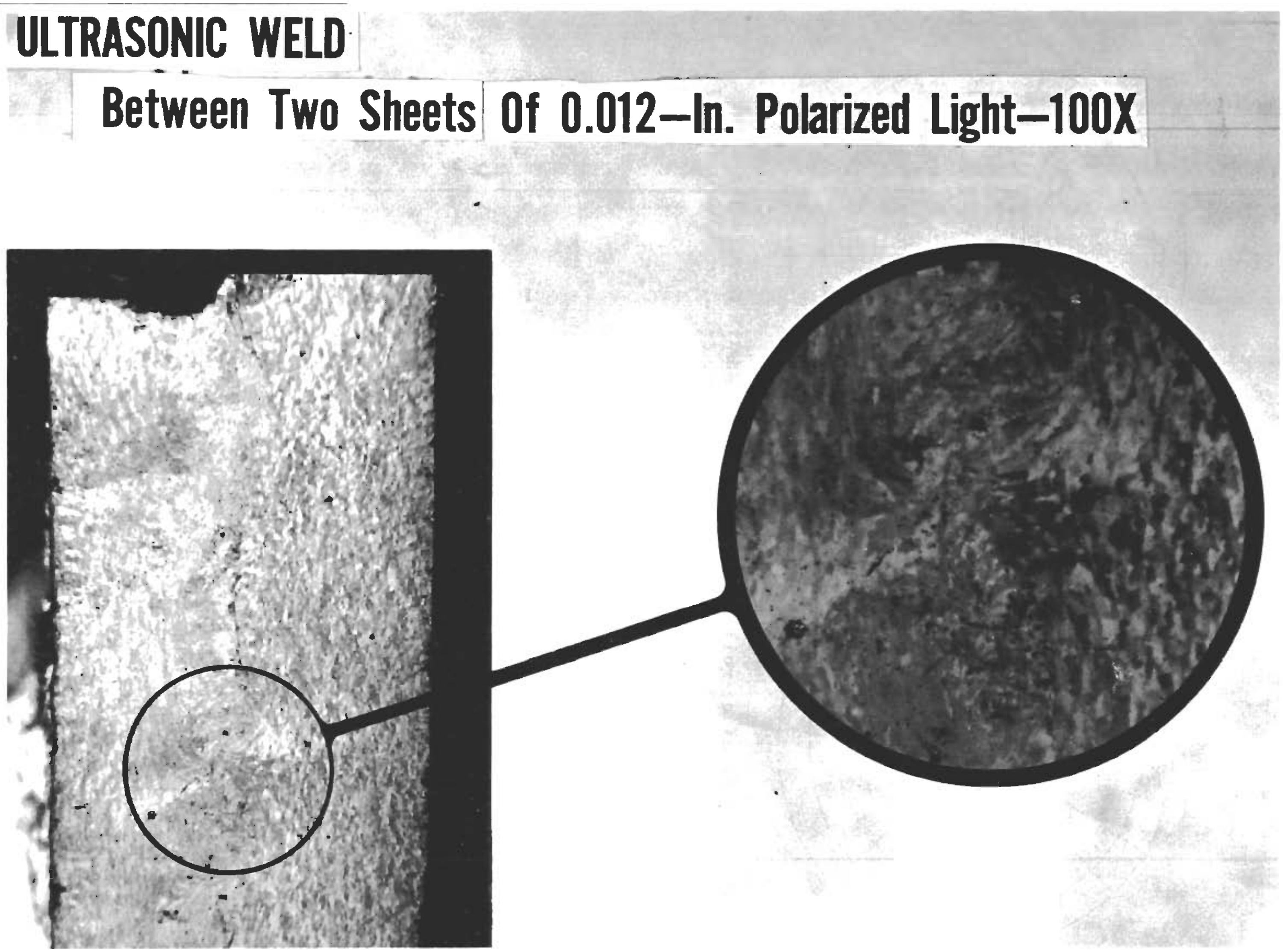
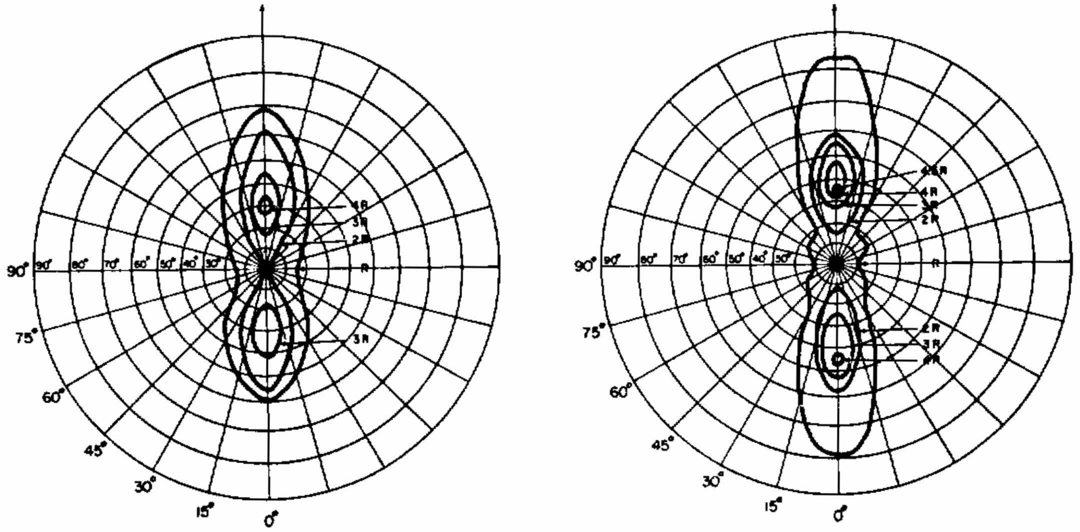
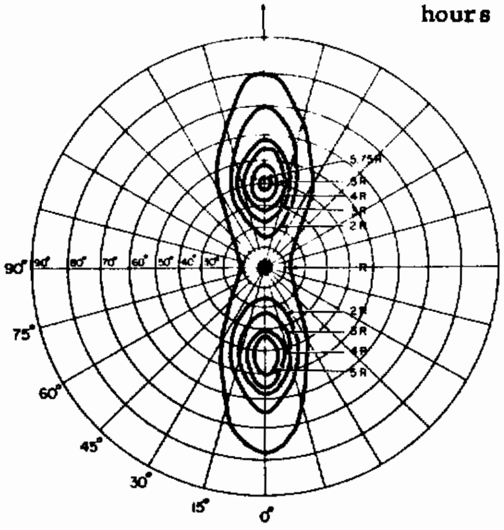

ULTRASONIC WELD
Between Two Sheets Of 0.012—In. Polarized Light—100X

Figure 2.



(a) (0001) texture of as-rolled sheet

(b) (0001) texture for sheet annealed at 1050°C for 10 hours



(c) (0001) texture for sheet annealed at 1100°C for 22 hours

Figure 3.

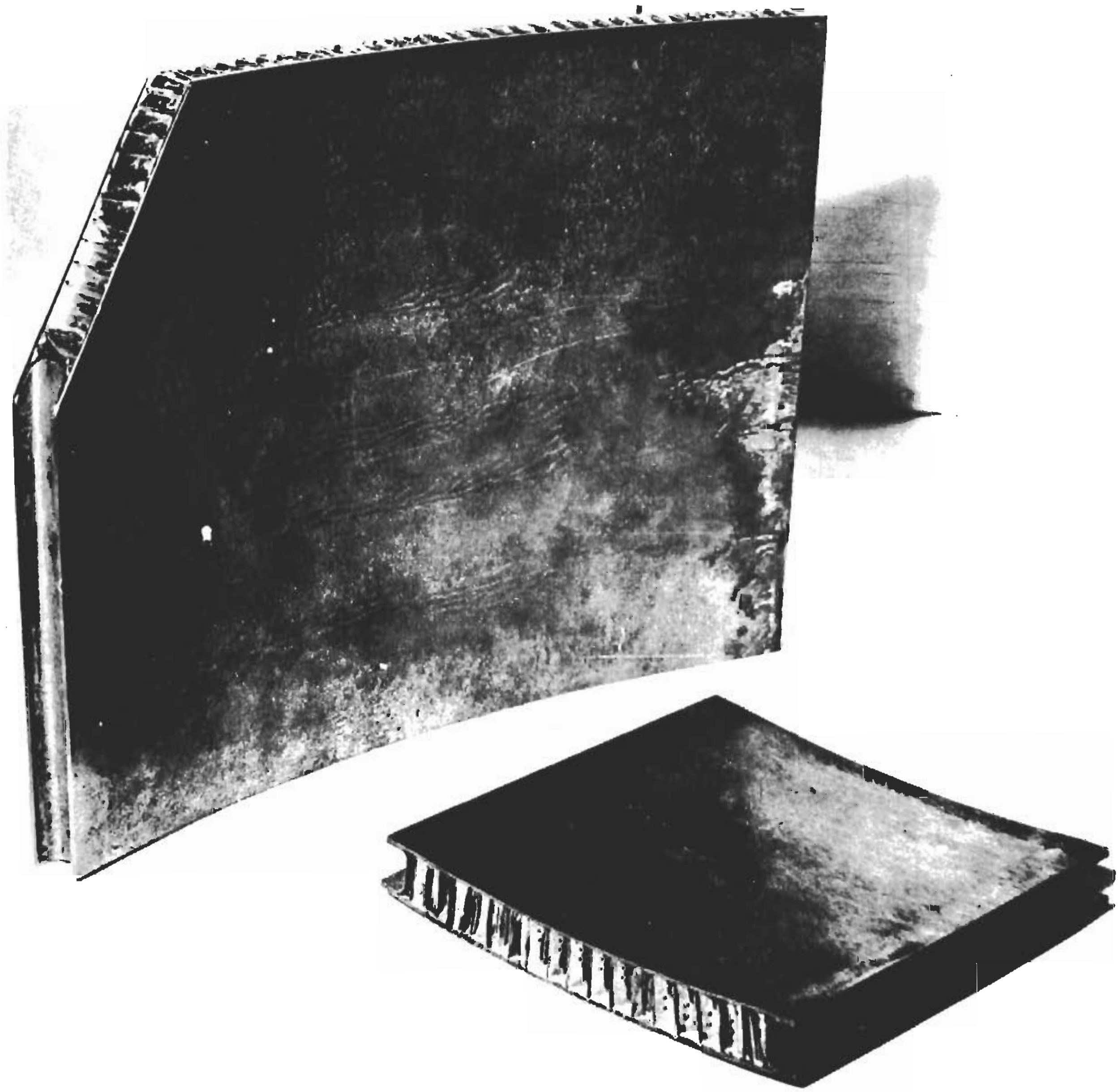


Figure 4.

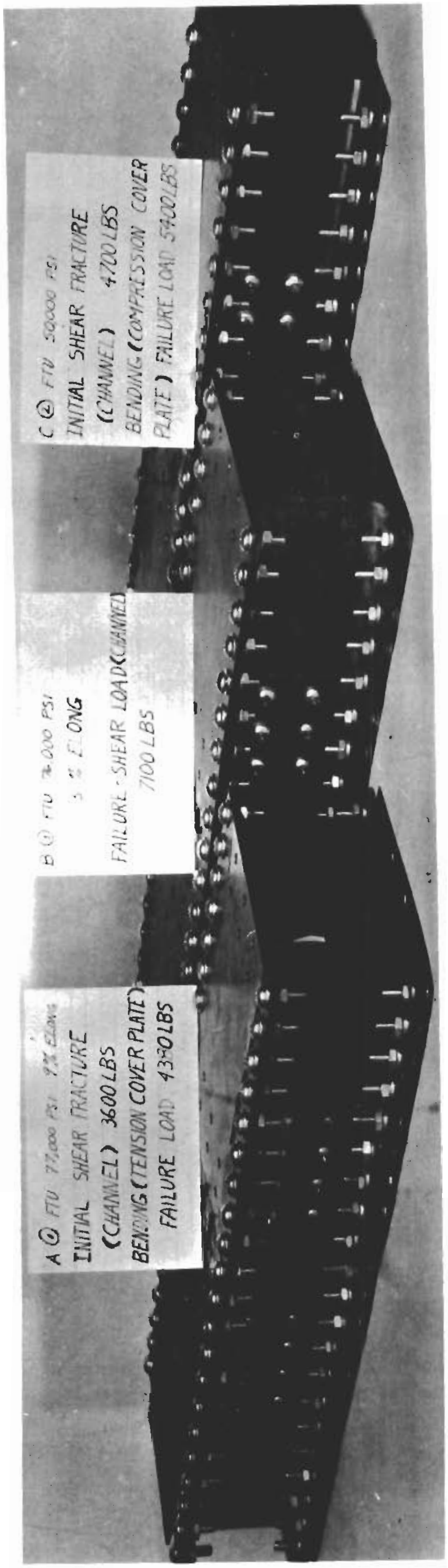

A ① FTU 77,000 PSI 9% ELONG
INITIAL SHEAR FRACTURE
(CHANNEL) 3600 LBS
BENDING (TENSION COVER PLATE)
FAILURE LOAD 4390 LBS
B ① FTU 76,000 PSI
3% ELONG
FAILURE - SHEAR LOAD (CHANNEL)
7100 LBS
C ② FTU 50,000 PSI
INITIAL SHEAR FRACTURE
(CHANNEL) 4700 LBS
BENDING (COMPRESSION COVER
PLATE) FAILURE LOAD 5400 LBS

Figure 5.