Wah Chang Corporation

# REDUCTION AND CONSOLIDATION OF SUPERIOR QUALITY MOLYBDENUM ALLOYS

Prepared Under Navy, Bureau of Aeronautics
Contract NOas 60-6046-c
Interim Report No. WCA 1001-8
1 November, 1961 to 15 February, 1962

WAH CHANG CORPORATION
ALBANY DIVISION
P. O. Box 366
Albany, Oregon



PROJECT NO. WCC-1001

This report prepared and submitted by

WAH CHANG CORPORATION
ALBANY DIVISION

Socrates S. Christopher

Socrates S. Christopher
Research Project Metallurgist

James Wong

James Wong
Director, Metallurgical
Research and Development

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## ABSTRACT

Two arc melted and one electron-beam melted extrusion billets of calcium reduced molybdenum were extruded to sheet bars at the Directorate of Materials and Processes, Wright-Patterson Air Force Base. These extrusions were processed into sheet and evaluated. The electron-beam melted molybdenum had a recrystallization temperature of 1500°F for one hour compared to 2600°F for the arc melted material. Mechanical properties of the two materials tested were comparable to commercial arc melted molybdenum.

Heat treatment studies on hydrogen reduced electron-beam melted molybdenum showed the material to be brittle after complete recrystallization.

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## INTRODUCTION

The past quarterly report contained base line data on primary breakdown by extrusion and rolling of hydrogen-reduced molybdenum which had been consumably arc and electron-beam melted. The effort was aimed at determining the metallurgical difference in the two materials and to evaluate the extrusion process as a means of obtaining a more suitable material for subsequent fabrication into sheet.

Three hydrogen-reduced electron-beam melted molybdenum billets were extruded at 1600°F, 1800°F, and 2400°F at extrusion ratios of 4:1, 6:1, and 7.5:1, respectively. These billets were compared with hydrogen-reduced molybdenum which was arc melted and extruded at 2400°F and a 5.6:1 extrusion ratio. All of the billets were extruded into sheet bars varying from 0.5- inch to 0.9-inch thick by 1.5-inch wide. Hardness measurements and metallographic examinations were made on each extrusion. In all cases, the electron-beam melted material showed areas where recrystallization occurred during extrusion. At 1800°F with an extrusion ratio of 6:1, the electron-beam melted material showed a completely recrystallized hot worked structure. In comparison, the arc melted material extruded at 2400°F, 5.6:1 ratio showed a partially recrystallized duplex structure.

Both the electron-beam and arc melted molybdenum sheet bars were rolled to .030-inch thick sheets and evaluated. Recrystallization temperatures were determined together with tensile and bend properties. Although the strength properties were similar at room temperature, the electron-beam melted material showed improved ductility. The recrystallization behavior of the two materials varied greatly. The electron-beam melted material showed small areas of recrystallized grains appearing after one hour at temperatures as low as 1000°F; after one hour at 1400°F, this material was completely recrystallized while, by comparison, less than five per cent recrystallization occurred at 1800°F for the arc melted molybdenum. The recrystallization temperature at 1400°F for one hour is the lowest reported value for molybdenum to date.

Studies were carried out on electron-beam-melted material to determine the effects of various heat treatments on room temperature properties. Specimens were treated for one hour at 1000°F, 1400°F, and 1800°F. The strength properties of the molybdenum tested at 1400°F and 1800°F remained at the same level. The 1400°F treated specimens, however, had elongation values greater than 30 per cent as compared to less than five per cent for the 1800°F treated material.

From the results to date and consultation with the Navy Bureau of Weapons, a program was prepared to fully evaluate the properties of the five materials being investigated under this program. These include:

1. Arc-Melted Calcium-Reduced Molybdenum
2. Electron-Beam-Melted Calcium-Reduced Molybdenum
3. Arc-Melted Hydrogen-Reduced Molybdenum
4. Electron-Beam-Melted Hydrogen-Reduced Molybdenum
5. Inert-Handled Molybdenum (powder product)

The extrusion process was selected for primary breakdown of the ingots into sheet bars. Billets were extruded at 2000°F at a 7.5:1 extrusion ratio. These extrusions were evaluated metallographically and by hardness measurements.

Rolling schedules for the two materials were selected based on past investigations and the data obtained from the present studies. Sheet was rolled to .030-inch gage and tested in the as-rolled, stress relieved and recrystallized conditions. Tensile and bend transition tests were used to evaluate the properties of the sheet. Recrystallization behavior was determined on all the sheet produced.

This quarterly report describes the research carried out on three calcium-reduced molybdenum ingots and the results of heat treating studies on sheet from Extrusion Number 525 made from hydrogen-reduced electron-beam-melted molybdenum.

## EXTRUSION

Three billets were extruded at the Aeronautical Systems Division, Directorate of Materials & Processes Facility at Wright-Patterson Air Force Base, Ohio. The starting materials in all cases were calcium-reduced molybdenum. Two of the billets had been consumably arc melted and the third had been melted in an electron-beam furnace. These ingots were machined into extrusion billets weighing approximately fifteen pounds each. The billet extrusion numbers and extrusion parameters are listed below:

1. Extrusion Number 555, Electron-Beam Melted
   Extrustion Ratio 7.5:1 at 2000°F

2. Extrusion Number 556, Arc-Melted
   Extrusion Ratio 7.5:1 at 2000°F

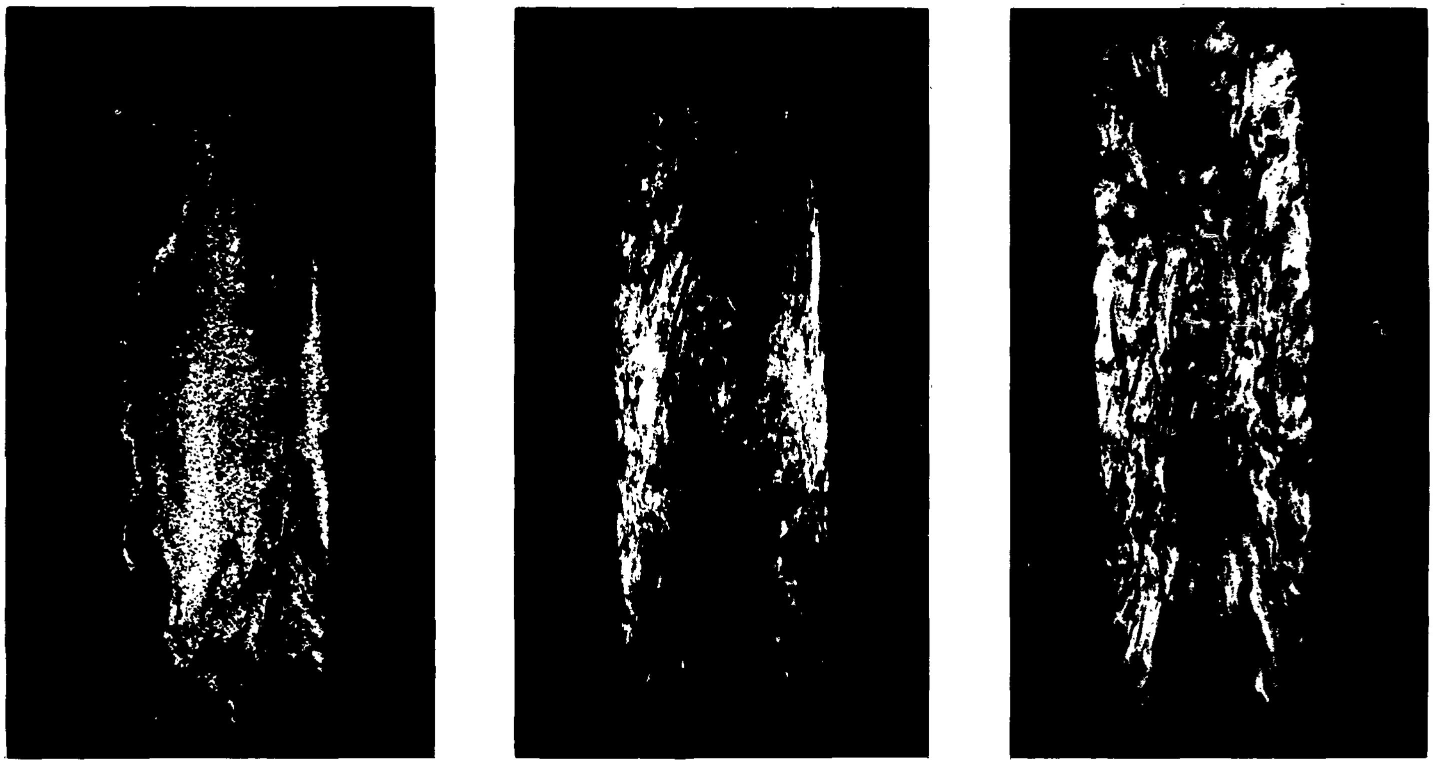
3. Extrusion Number 557, Arc-Melted
   Extrusion Ratio 7.5:1 at 2400°F

The conditions for Extrusions 555 and 556 were the same in order to evaluate the relative properties resulting from the two methods of melting. Extrusion 557 was carried out at a higher temperature to obtain more data on the effects of higher extrusion temperature on microstructure and hardness. All the billets were extruded into sheet bars, approximately 1/2-inch by 1-1/2 inch by 40 inches with yields in excess of 90 per cent. The as-extruded surfaces were free from any observable defects.

The extrusions were evaluated metallographically and by hardness measurements. Metallographic specimens were taken from the front, middle, and back portions of the extrusions and both center and edge micrographs were taken on each section. The front and back transverse sections were taken six inches from the nose and rear of the extrusion to obtain representative microstructures. Transverse macrostructures from the middle portion of the three extrusions are shown in Figure 1.

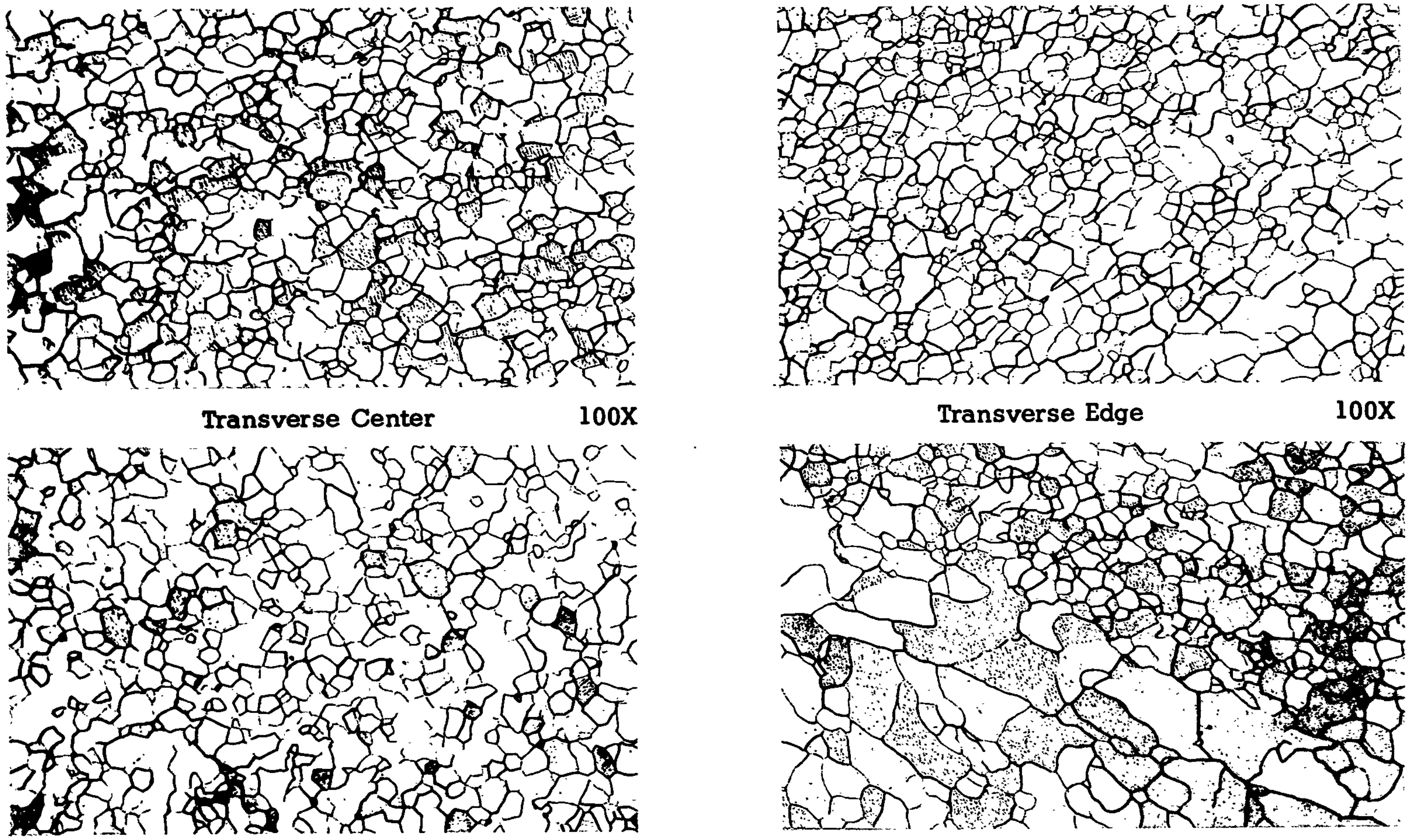
The microstructures of all the extrusions were consistent throughout the length and across the width. A large variation in structure was observed between the arc-melted and electron-beam-melted molybdenum.

The electron-beam-melted molybdenum exhibited a completely hot worked microstructure. The grain size of the recrystallized microstructures varied considerably due to certain areas where coalescence occurred and other areas which did not have sufficient time for grain growth. This microstructure is typical of hot extruded electron-beam-melted material and is attributed to the large grain size in the electron-beam ingot. The fully recrystallized microstructure of the calcium reduced electron-beam-melted molybdenum is comparable to hydrogen-reduced and electron-beam melted material extruded above 1800°F. The hot worked microstructure of Extrusion Number 555 is shown in Figure 2.



555 556 557

**Figure 1. Transverse Macrostructures of the Middle Portions of Extrusions 555, 556 and 557**



Transverse Center 100X

Transverse Edge 100X

Longitudinal Center 100X

Longitudinal Edge 100X

**Figure 2. Extrusion No. 555 Calcium Reduced-Electron Beam Melted**
**Extrusion Ratio 7.5:1 Extrusion Temperature 2000°F**
**Microstructures Showing Variation Between the Center and Edge of the Back Section**

Extrusions 556 and 557 were arc-melted and extruded at 2000°F and 2400°F, respectively. Both showed a highly cold worked structure composed of large elongated grains. Extrusion Number 557 was carried out at a higher temperature in an attempt to obtain a uniform hot work structure comparable to that obtained in Extrusion Number 555. The microstructures of Extrusions Number 556 and 557 are shown in Figures 3 and 4.

Hardness values were obtained on the three extrusions. The electron-beam-melted material had an average DPH hardness of 176 compared to 243 for Extrusion Number 556 and 224 for Extrusion Number 557. These hardness values correlate very well with the microstructure observed and reflect the difference between starting material and extrusion temperature.

## ROLLING

Sheet bars .5-inch by 1-inch by 4-inch were machined from extrusions 555 and 556. The rolling schedules selected for the two materials, Figure 5, were based on the results derived from past studies on the program. An attempt was made to work the material in a manner such that sheets with a similar fabrication history and sufficient in size for evaluation purposes would be produced. It was intended that initial breakdown of the sheet bars would be carried out above the recrystallization temperature of the material. Based on past results, 1800°F and 2400°F were selected for Extrusions Number 555 and Number 556, respectively. Also from past results, it was assumed that the recrystallization temperature for the arc melted material would be between 2000°F and 2200°F and the electron-beam material below 1600°F. The specimens were then rolled in one direction to 0.100-inch and conditioned by sandblasting and pickling. After conditioning, the specimens were reheated to 1000°F and 1800°F, respectively, and cross-rolled to 0.030-inch sheet. Again, the temperatures were selected to impart cold work to the sheet for subsequent recrystallization studies. The sheets were then conditioned and cut into specimens for evaluation

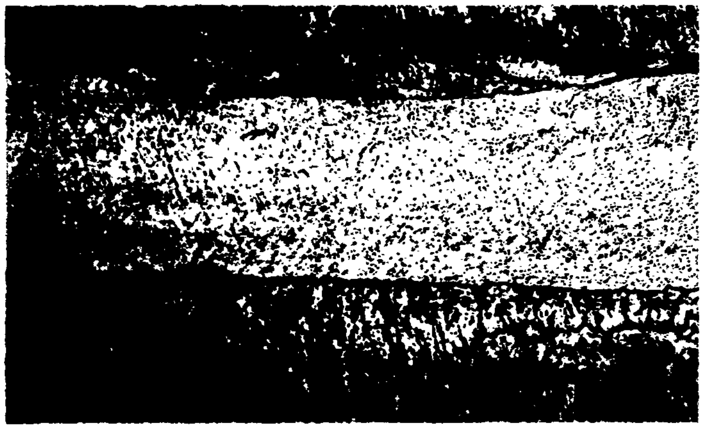
## EVALUATION OF .030-INCH SHEET ROLLED FROM EXTRUSIONS NUMBER 555 AND NUMBER 556

### A. Recrystallization

Recrystallization studies were carried out utilizing hardness measurements and metallographic techniques. Small sheet specimens approximately 1/2-inch square were heat treated for one hour at temperatures ranging from 800°F to 1600°F for the electron-beam

Transverse Center 100X

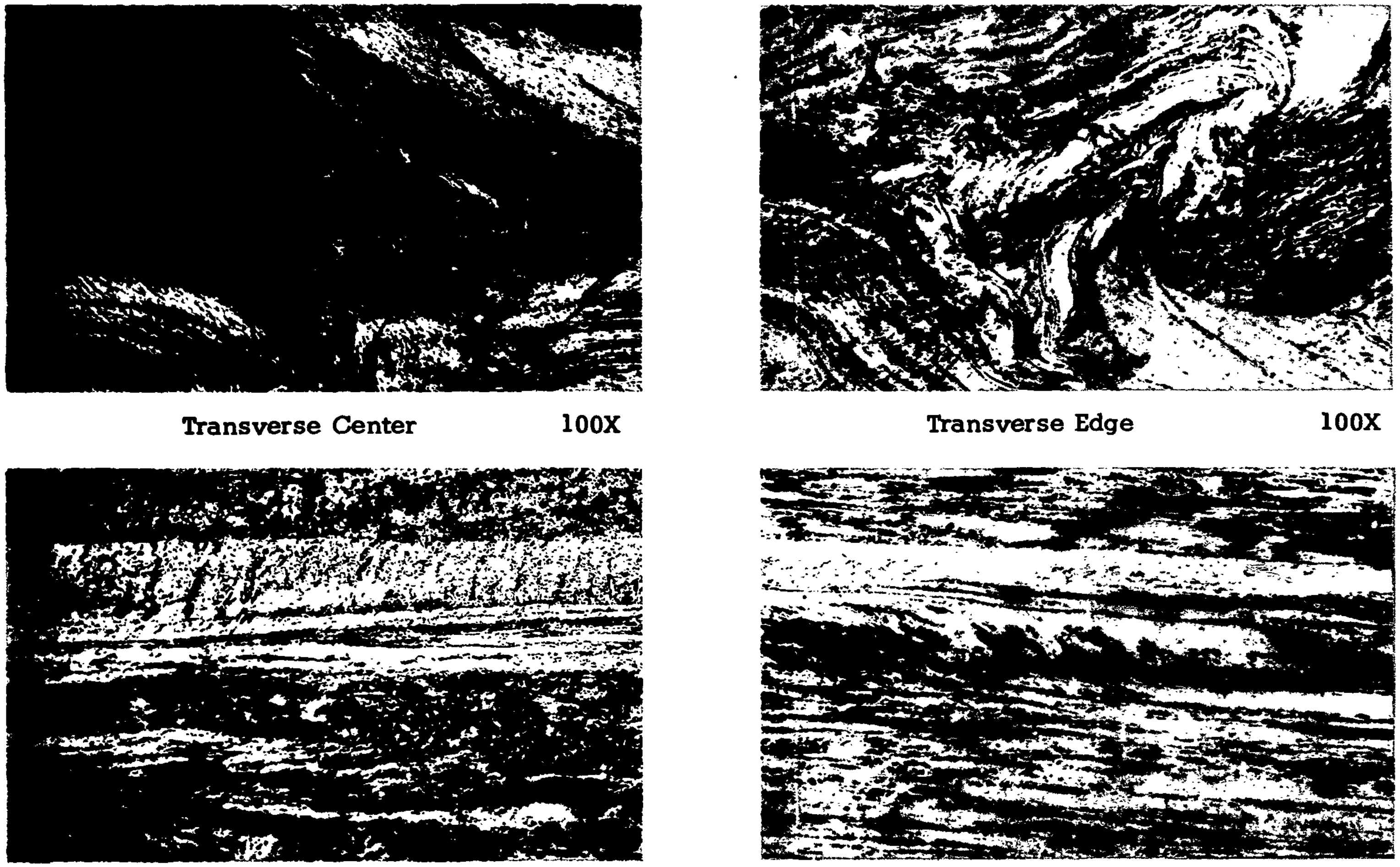
Transverse Edge 100X



Longitudinal Center 100X

Longitudinal Edge 100X

**Figure 3. Extrusion No. 556 Calcium Reduced Arc Melted Extrusion Ratio–7.5:1 Extrusion Temperature 2000°F Microstructures Showing Variation Between Center and Edge of the Back Section**



Transverse Center 100X

Transverse Edge 100X

Longitudinal Center 100X

Longitudinal Edge 100X

Figure 4. Extrusion No. 557 Calcium Reduced-Arc Melted
Extrusion Ratio 7.5:1 Extrusion Temperature 2400°F
Microstructures Showing Variation Between Center and Edge of Back Section

| Extrusion 555 | Extrusion 556 |
|---|---|
| Preheat 20 Min. @ 1800°F | Preheat 20 Min. @ 2400°F |
| Reheat 5 Min. Between Passes | Reheat 5 Min. Between Passes |
| 20% Red/pass ↓ | 20% Red/pass ↓ |
| Roll to .100 Inch | Roll to .100 Inch |
| Sandblast and Pickle | Sandblast and Pickle |
| Roll @ 1000°F | Roll @ 1800°F |
| 10% Red/pass ↓ | 20% Red/pass ↓ |
| Final Thickness .030 Inch | Final Thickness .030 Inch |
| Sandblast and Pickle | Sandblast and Pickle |

Figure 5. Rolling Schedules for Extrusions 555 and 556

melted material and 1800°F to 2800°F for the arc-melted molybdenum. The specimens were metallographically prepared and DPH hardness traverses were made across the specimens. The hardness versus heat treating temperature curves for both materials are shown in Figure 6. The low recrystallization temperature as shown by the hardness drop is substantiated by the photomicrographs shown in Figures 7 and 8. The electron-beam-melted material had small equiaxed grains appearing as low as 1200°F after one hour and recrystallization was complete at 1600°F. The 100 per cent recrystallization temperature for this material is considered to be 1500°F for one hour. The hardness drop in the temperature of 1200°F to 1600°F was from 265 to 184 DPH. The arc melted molybdenum had a 100 per cent recrystallization temperature of 2600°F for one hour with some recrystallization occurring at the highly worked surface of the sheet as low as 1800°F. The hardness dropped from 257 to 178 DPH in the temperature range of 1800°F to 2600°F, respectively.

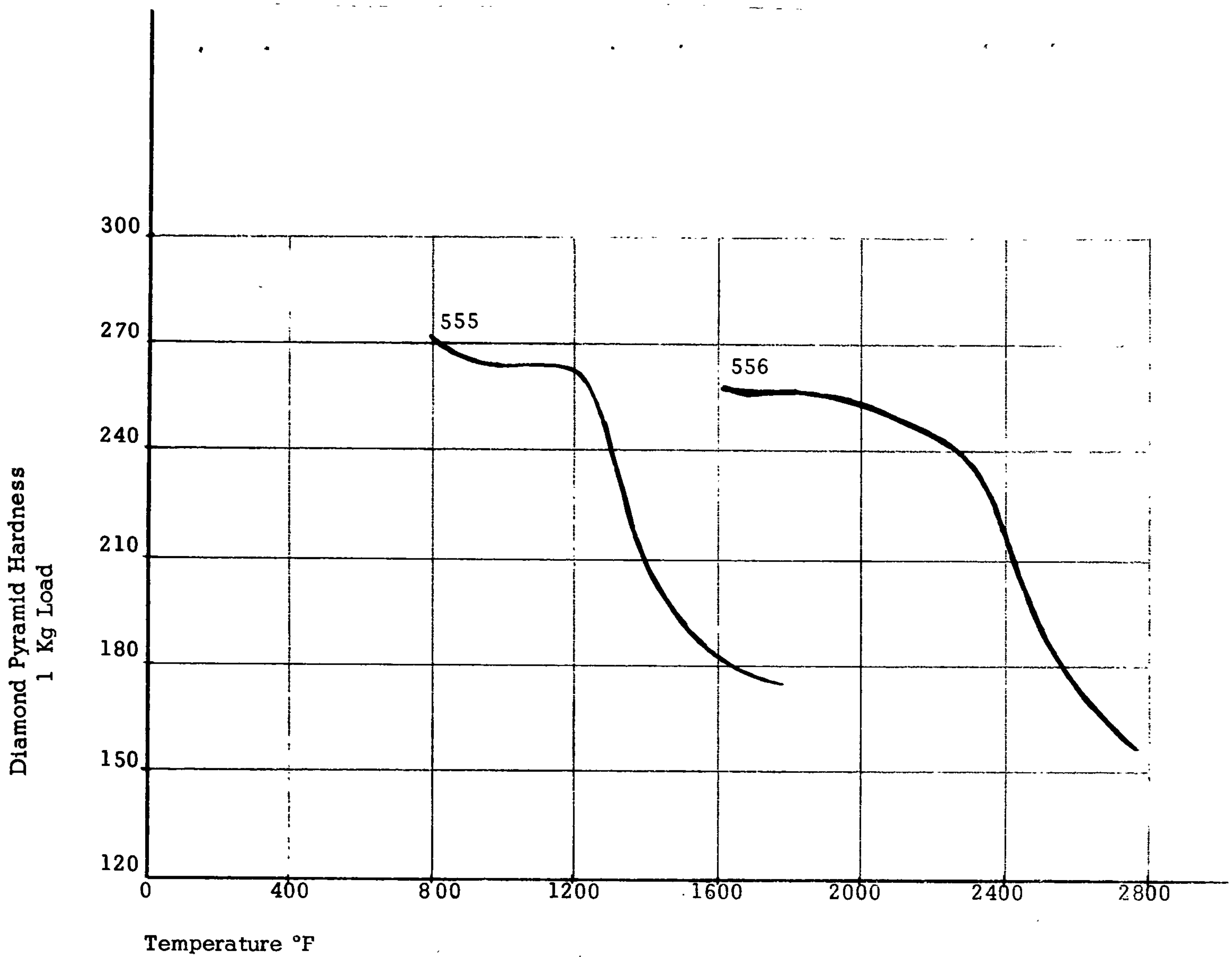
## MECHANICAL TESTING

Sheets from Extrusions Number 555 and Number 556 were tested in tension in the following three conditions: (1) as-rolled, (2) stress relieved, and (3) recrystallized. Both longitudinal and transverse directions in the sheet were tested. The sheet was tested at a strain rate of .005 in/in/min to .6 per cent offset and then increased to .05/in/in/min to failure. A three inch tensile specimen with a one inch gage length was employed in all cases.

Sheet 555 was stress-relieved one hour at 1200°F and recrystallization was done above 1500°F for one hour. The 1200°F stress relieving temperature was selected based on the microstructures shown in Figure 7, which shows recrystallized grains appearing in the center of the sheet. The tensile strength properties of the sheet were of the same magnitude in the longitudinal and transverse directions with the transverse direction having slightly lower elongation values. An upper and lower yield point effect was observed in one of the specimens in the recrystallized condition. This yield point behavior is attributed to interstitial impurities interacting with dislocations according to the mechanism proposed by Cottrell*. The tensile properties of the sheet are given in Table 1.

Sheet from Extrusion Number 556 was conditioned by stress relieving at 1800°F for one hour and recrystallization was carried out at 2600°F for one hour. The tensile strength of the sheet was comparable to that of sheet 555, but elongation values were lower in most cases.

* Cottrell, A.H., Dislocations and Plastic Flow in Crystals, Oxford Press, Clarendon, England, 1953




.030 Inch Sheet From Extrusions No. 555 & 556
Final Rolling Temperature 1000°F & 1800°F, Respectively
Heat Treated One Hour at Temperature

Figure 6. Room Temperature Hardness vs. 1 Hour Heat Treatment

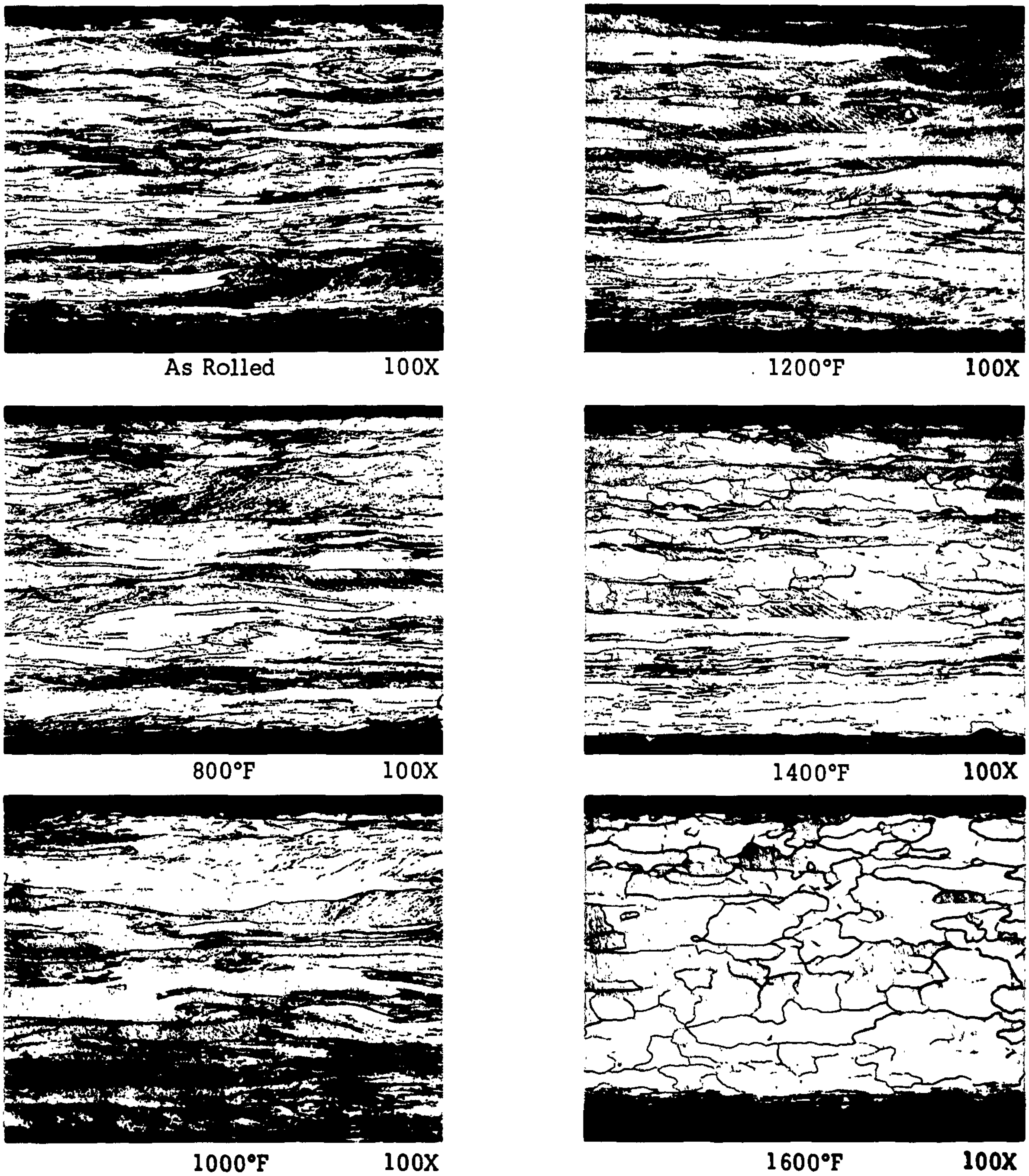



Figure 7. .030-Inch Sheet From Extrusion No. 555
Final Rolling Temperature 1000°F
Heat Treated One Hour at Temperature

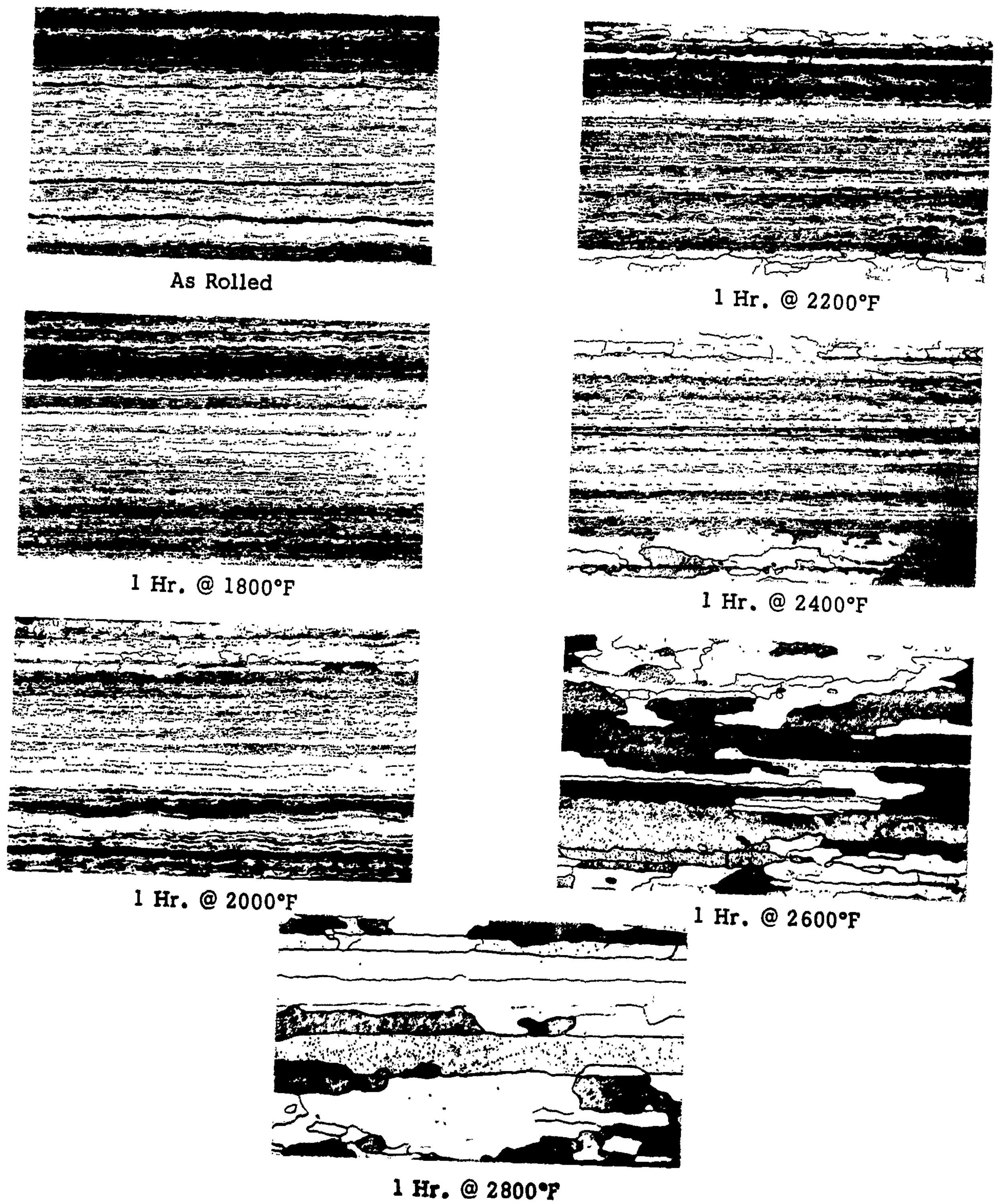



Figure 8. .030-Inch Sheet From Extrusion No. 556
Final Rolling Temperature 1800°F
Heat Treated One Hour at Temperature

TABLE 1

Tensile Properties of .030-inch Sheet
from
Extrusion No. 555

| Condition | Direction | UTS | YS | % Elongation 1" |
|---|---|---|---|---|
| As Rolled | Long. | 112,000 | 101,000 | 10 |
| As Rolled | Long. | 119,000 | 101,000 | 13 |
| Stress Relvd. | Long. | 108,000 | 93,400 | 13 |
| Stress Relvd. | Long. | 108,000 | 92,100 | 14 |
| Recrystallized | Long. | 75,700 | upper 47,000<br>lower 45,000 | 27 |
| Recrystallized | Long. | 76,700 | 45,900 | 28 |
| As Rolled | Trans. | 119,000 | 101,000 | 6 |
| As Rolled | Trans. | 119,000 | 103,000 | 7 |
| Stress Relvd. | Trans. | 115,000 | 98,000 | 11 |
| Stress Relvd. | Trans. | 114,000 | 96,200 | 10 |
| Recrystallized | Trans. | 74,600 | 43,600 | 24 |
| Recrystallized | Trans. | 73,700 | 43,000 | 22 |

Stress Relieved 1 hour @ 1200°F
Recrystallized 1 hour @ 1500°F

The tensile properties of sheet 556 showed a larger variation between the longitudinal and transverse directions of testing; the transverse direction in the as-rolled and stress-relieved condition had higher strength values with lower elongation. The strength properties of the material tested in the recrystallized condition were of the same level in both directions, but again the transverse direction exhibited lower elongation values. In all the tests conducted on specimens in the recrystallized condition, a strong yield point behavior was apparent. The variation between the upper and lower yield point was from 4,000 to 17,000 psi. The yield point behavior is attributed to a higher interstitial content in the arc melted material. The tensile properties of the .030-inch sheet from Extrusion Number 556 are given in Table 2. Figure 9 is a comparison of the typical load-strain curves for the two sheet materials.

## BEND PROPERTIES

The bend properties of the two sheet materials were evaluated in the as-rolled and recrystallized conditions. The specimens in each condition were tested at room temperature (72°F), 32°F and -100°F. Both directions in the sheet were tested. Initial attempts to test according to MAB specifications resulted in brittle failure at room temperature in all heat treated conditions. Micro examination of the surface of the sheet showed a contaminated surface layer which was subsequently removed by pickling. Testing of the pickled material showed little improvement. The ram speed was then decreased from 10 in/min to 1 in/min. The combination of slower ram speed and removal of .002 in of surface by pickling resulted in a testing procedure which would allow a relative comparison of the bend properties of the two materials.

The sheet from Extrusion Number 555 exhibited extreme anisotropy in bend properties. In the as-rolled condition, brittle fracture occurred at all temperatures in the transverse direction. No failure occurred in the longitudinal specimen tested at room temperature and 32°F. At -100°F both longitudinal and transverse directions were completely brittle. Specimens tested in the recrystallized condition were completely brittle at all temperatures. The anisotropy exhibited by the bend properties is associated with the sensitivity of the transverse direction to the stress conditions prevalent in the bend test. The ductile to brittle transition temperature in bending would be initially apparent in the transverse direction. The sensitivity of the transverse direction is associated with structural and crystallographic anisotropy developed during rolling.

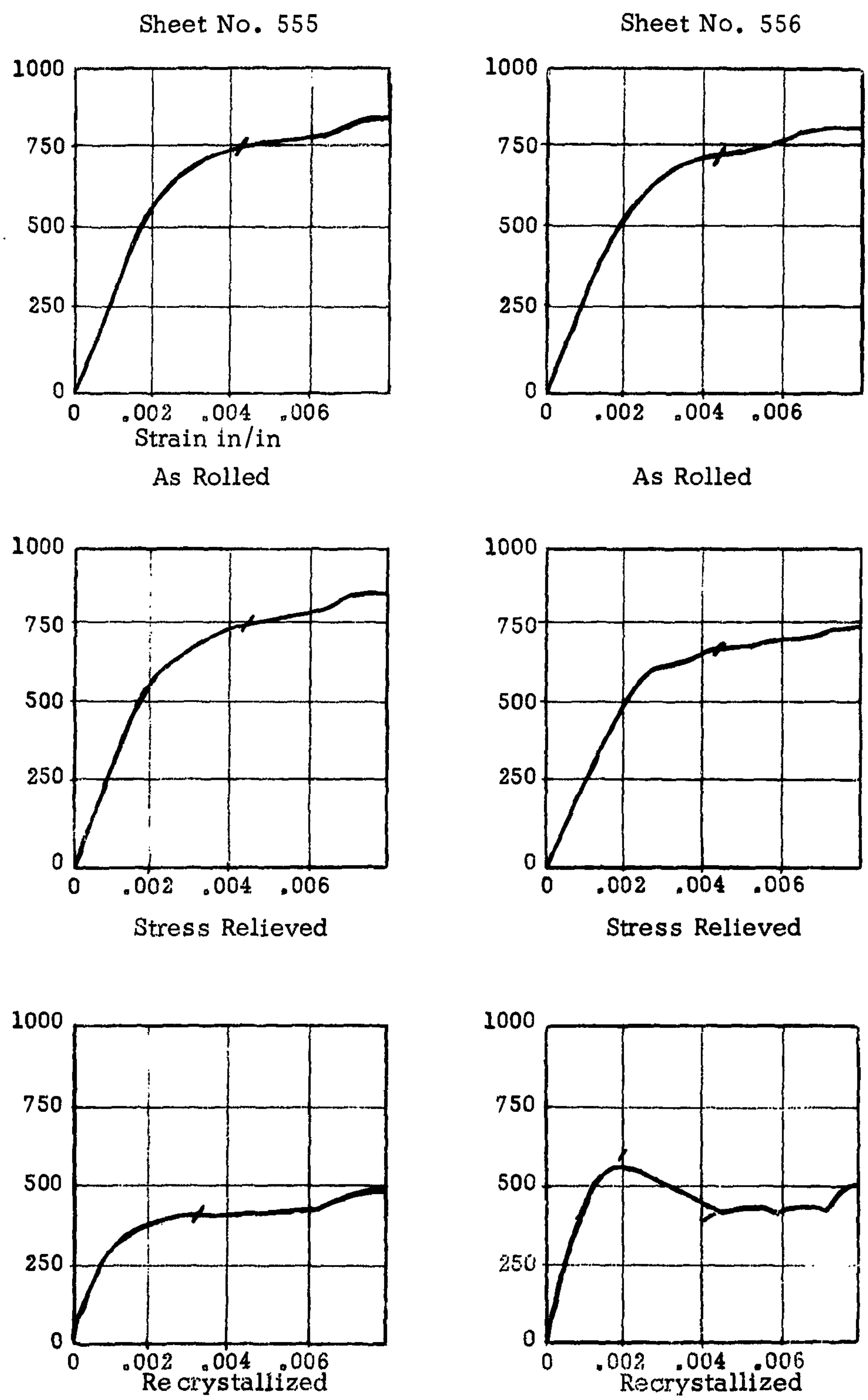



Figure 9. Load Strain Curves for .030 Inch Sheet

TABLE 2

Tensile Properties of .030-inch Sheet
from
Extrusion No. 556

| Condition | Direction | UTS | YS | % Elongation 1" |
|---|---|---|---|---|
| As Rolled | Long. | 108,200 | 94,100 | 12 |
| As Rolled | Long. | 111,000 | 99,600 | 13 |
| Stress Relvd. | Long. | 106,800 | 94,200 | 13 |
| Stress Relvd. | Long. | 106,000 | 86,500 | 13 |
| Recrystallized | Long. | 72,100 | upper 56,300<br>lower 51,800 | 18 |
| Recrystallized | Long. | 70,800 | upper 67,000<br>lower 50,600 | 16 |
| As Rolled | Trans. | 119,500 | 99,600 | 3 |
| As Rolled | Trans. | 122,000 | 105,000 | 4 |
| Stress Relvd. | Trans. | 110,000 | 99,600 | 6 |
| Stress Relvd. | Trans. | 111,000 | 98,300 | 6 |
| Recrystallized | Trans. | 69,200 | upper 60,600<br>lower 54,200 | 11 |
| Recrystallized | Trans. | 64,500 | upper 59,200<br>lower 50,100 | 12 |

Stress Relieved 1 hour @ 1800°F
Recrystallized 1 hour @ 2600°F

In the as-rolled condition, sheet 556 exhibited both longitudinal and transverse ductility in bending as low as 32°F. At -100°F both longitudinal specimens and one transverse specimen showed ductile behavior with no fracture. The other transverse specimen failed in a brittle manner with lamination extending the width of the bend.

The recrystallized bend properties of sheet 556 demonstrated ductile behavior in both directions at room temperature, and transverse brittleness at 32°F. Both longitudinal and transverse specimens were brittle at -100°F.

## HEAT TREATING of EXTRUSION 525

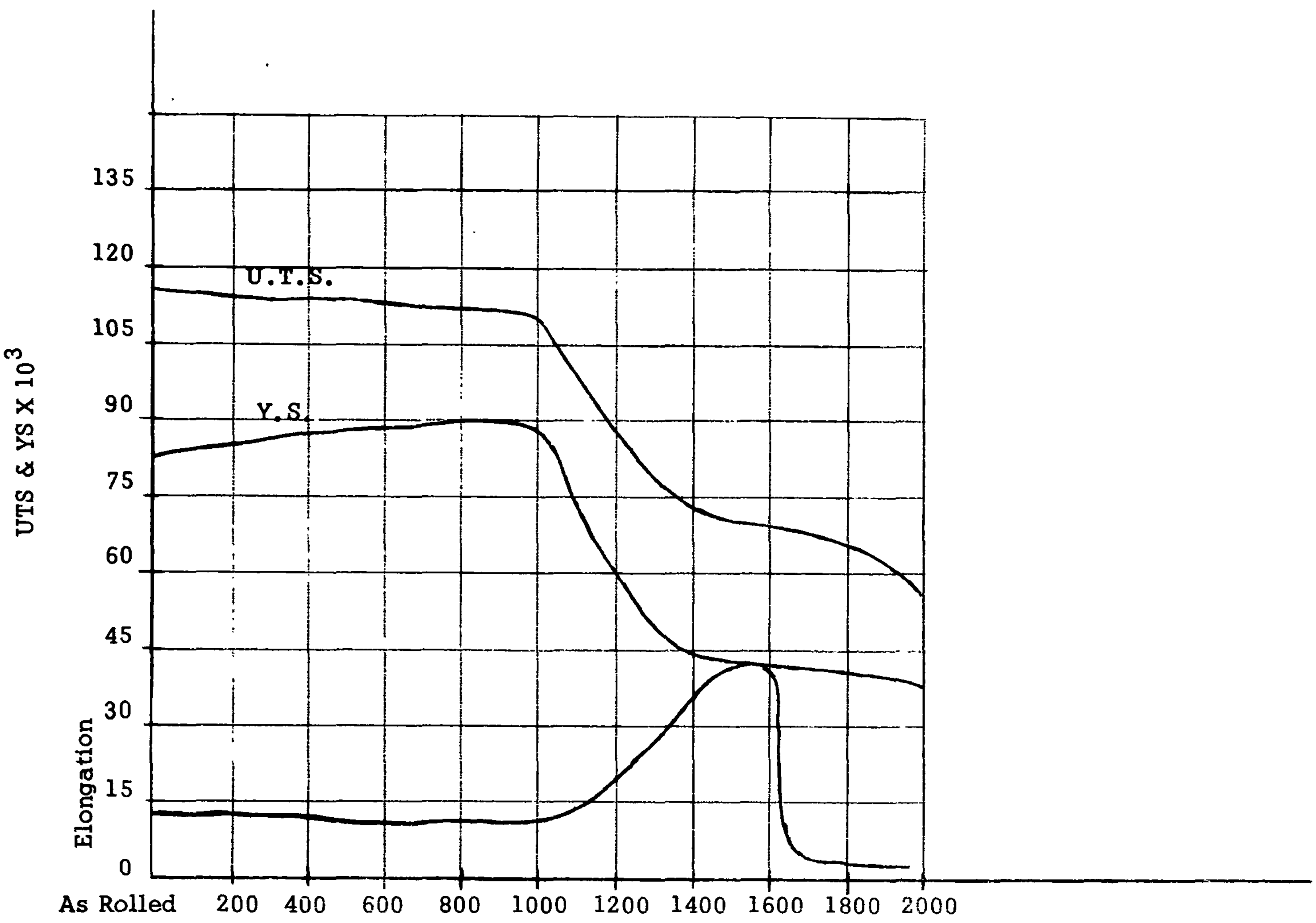
In the last progress report, sheet from Extrusion Number 525* was heat treated for one hour at 1000°F, 1400°F, and 1800°F, and tested in tension. The results of these tests showed a large drop in ductility occurring after the 1800°F treatment. In order to study the nature of this precipitous decrease, specimens in both the longitudinal and transverse directions were heat treated for 1 hour at 1600°F, 1650°F, 1700°F, and 2000°F and tested at room temperature. The results of these studies show that brittleness in both directions of the sheet occurs between 1600°F and 1650°F. The large decrease in ductility from greater than 40 per cent to less than 10 per cent is attributed to complete recrystallization of the sheet. The tensile properties versus heat treating temperature for the longitudinal specimens are given in Figure 10.

## FUTURE PROGRAM

Two billets of hydrogen-reduced molybdenum, one arc-melted, and the other electron-beam melted, have been extruded at 2000°F and a 7.5:1 extrusion ratio. These billets will be evaluated in the same manner as Extrusions Number 555 and Number 556 and a comparison will be made of the properties.

Sheet bars of inert handled molybdenum have been canned in one-quarter inch mild steel and the compacts evacuated. These bars will be rolled using conventional rolling procedures for pure molybdenum. The sheet bars will be rolled to .030-inch sheet and evaluated for hardness, recrystallization temperature, and mechanical properties.

* Hydrogen-Reduced, Electron-Beam Melted, Extrusion Ratio 6:1 at 1800°F




Extrusion 525

Figure 10. Room Temperature Tensile Properties vs. One Hour Heat Treatment

In previous investigations conducted under this program single crystals of molybdenum approximately one inch square by five inches were press forged to 1/4-inch thick at 500°F. The forged slab was conditioned and rolled at 800°F, 20 per cent reduction per pass to 0.050-inch thick sheet. The sheet was then conditioned, cut into specimens 2-1/2-inch by 2-inch and rolled to 0.015 inch. Both pieces were badly laminated and could be easily split in half manually. Although laminations are usually attributed to grain boundary effects, the laminations in the single crystals are most likely to be crystallographic in nature. In order to further analyze the above results, two single crystals with known orientations will be rolled below their recrystallization temperature. The sheet produced will be evaluated for lamination, mechanical properties, and recrystallization temperature.

## CONCLUSIONS

Starting material, whether hydrogen-reduced or calcium-reduced after electron-beam melting seems to have little effect on the final properties of the material. Electron-beam melting with higher vacuum, slower melting rate, and better control during melting appears to be the primary factor for the higher purity of the molybdenum as compared to normal arc-melted material. The higher purity of the molybdenum following electron-beam melting is evident by its extremely low recrystallization temperature. The higher recrystallization temperature determined on the arc-melted material is attributed to a lack of purification during the melting process. The high oxygen content of the calcium-reduced molybdenum starting material is not adequately removed during arc melting.

In this investigation of calcium-reduced electron-beam and arc-melted molybdenum, the higher purity of the electron-beam material was readily apparent. Electron-beam and arc-melted molybdenum exhibited one hour recrystallization temperatures of 1500°F and 2600°F, respectively. Further evidence of the high interstitial level in the arc-melted material was observed by the strong yield point behavior of all the recrystallized specimens. This yield point behavior although observed in one of the electron-beam melted materials tested in the recrystallized condition was not as definite as that found in the arc-melted.

Mechanical properties of the two materials showed variations which in most cases could be attributed to the differences in the metallurgical history or structure such as; amount of cold work, grain size, and fibering. Although the specimens were fabricated under precise controls, some variations are unavoidable.

The tensile strength properties of the electron-beam melted material were of the same order of magnitude in both directions of testing. The arc-melted molybdenum showed a large variation between the longitudinal and transverse directions of testing. This variation was most noticeable in the elongation values. This directionality in the arc-melted material is probably due to the large prior as extruded cast grain size and higher percentage of cold work in the sheet.

Bend testing of the materials both showed a higher ductile to brittle transition temperature in the transverse than in the longitudinal direction. From the results obtained, the arc-melted material had a ductile brittle transition temperature somewhere below 32°F. At -100°F for this material, the transverse direction became brittle. The transverse direction in the electron-beam melted material failed at 72°F and 32°F and both directions failed at -100°F. In the recrystallized condition and at all temperatures, both directions in the electron-beam material were brittle. Arc-melted molybdenum tested in the recrystallized condition showed ductile behavior at room temperature and longitudinal ductility at 32°F. Specimens tested at -100°F were brittle in both directions.

The low transition temperature of the arc-melted material is attributed to a greater percentage warm working in the specimens when tested in the as-rolled condition. The relatively low transition temperature of the recrystallized arc-melted specimens can probably be ascribed to the less than 100 per cent recrystallization of the specimens.

Room temperature tensile tests on sheet 525 (hydrogen-reduced and electron-beam melted) were conducted after heat treating. The results of these tests point out the brittleness of this material in the fully recrystallized condition. This behavior is comparable to commercial powder and arc-melted molybdenum tested in the recrystallized condition.

Based on results to date the calcium-reduced arc-melted material appears to result in a lower purity than commercially processed arc-melted molybdenum.

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Aerojet General Corporation
P. O. Box 1947
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ATTN: Alan V. Levy, Head
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Los Angeles 36, California
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Detroit 3, Michigan

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Lockheed Missiles and Space Division
3251 Hanover Street
Palo Alto, California
ATTN: Mr. Roger Perkins
Metallurgy and Ceramics - 53-36

Lockheed Missile and Space Division
3251 Hanover Street
Palo Alto, California
ATTN: Dr. T. E. Tietz

Los Alamos Scientific Laboratory
University of California
P. O. Box 1663
Los Alamos, New Mexico
ATTN: Dr. R. D. Baker

Marquardt Aircraft Company
16555 Saticoy Street
P. O. Box 2013 - South Annex
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National Aeronautics and Space Administration
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Cleveland 35, Ohio
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70 Memorial Drive
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ATTN: Mr. J. H. Gardner

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ATTN: Mr. L. M. Raring, Chief
Metallurgical and Chemical Laboratories

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1512 H Street, N. W.
Washington 25, D. C. (4)

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Missile Systems Division
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Metallurgist

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ATTN: Mr. E. A. Simkovich
Advanced Systems and Research

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Birmingham, Alabama
ATTN: Mr. E. J. Wheelahan

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ATTN: Dr. R. C. Nelson

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Richmond, California
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Staff Research and Development

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Niagara Falls, New York
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Technology Department

Universal-Cyclops Steel Corporation
Bridgeville, Pennsylvania
ATTN: Mr. P. C. Rossin

Office of Ordnance Research
Box CM, Duke Station
Durham, North Carolina

Naval Research Laboratory
Navy Department
Washington 25, D. C.
ATTN: Code 423

Bureau of Ships
Navy Department
Washington 25, D. C.
ATTN: Codes 347 and 1500

Naval Air Material Center
Philadelphia 12, Pennsylvania
ATTN: Aeronautical Materials Laboratory

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Navy Department
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Navy Department
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Naval Research Laboratory
Washington 25, D. C.

Branch Office
Inspector of Naval Material
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208 S. W. Fifth Avenue
Portland 4, Oregon

Watertown Arsenal Laboratory
Watertown, Massachusetts
ATTN: Mr. S. V. Arnold

Westinghouse Electric Company
Metals Plant
Blairsville, Pennsylvania
ATTN: Mr. A. E. LeMarche

Westinghouse Electric Company
Lamp Division
Bloomfield, New Jersey
ATTN: Dr. R. H. Atkinson

Wright-Patterson Air Force Base
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ATTN: WCLTIPH

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Ohio
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