Refractometry Division

UNIVERSAL-CYCLOPS STEEL CORPORATION

Technical Report

Bridgeville, Pennsylvania
INFAB PROCESSING OF TZM SHEET

Contract AF33(657)-8495

Fourth Interim Technical Engineering Report
15 January 1963 - 15 April 1963

Phase IV Report

Prepared By
F. R. Cortes

UNIVERSAL-CYCLOPS STEEL CORPORATION
REFRACTOMET DIVISION
BRIDGEVILLE, PENNSYLVANIA

BASIC INDUSTRY BRANCH
MANUFACTURING TECHNOLOGY LABORATORY
Directorate of Materials and Processes
Aeronautical Systems Division
United States Air Force
Wright-Patterson Air Force Base, Ohio
 Twenty-nine pieces of intermediate gage (mold out) TZM have been evaluated for soundness, contamination, recrystallization and tensile properties. Results show that mold out produced by rolling at 2400 and 2800°F are superior to mold out rolled at 2000 or 3200°F regardless of forging practice. Mold out properties are correlated with forging practice and results show that mold out produced from fine hot forged or partially hot forged sheet bar structures exhibit the heat combination of properties. This evaluation essentially completes the Phase IV program.
INFAB PROCESSING OF TZR SHEET

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REFRACTORITE DIVISION
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This Interim Technical Progress Report covers work performed under Contract AF 33(657)-8495 from 15 January 1963 to 15 April 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with the Refractomet Division of Universal-Cyclops Steel Corporation, Bridgeville, Pennsylvania was initiated under ASC Aeronautical System Division, Project 7-786, "InFab Processing of TZM Sheet." It was administered under the direction of Mr. Hugh L. Black, Project Engineer, Basic Industry Branch, Manufacturing Technology Laboratory, Wright-Patterson Air Force Base, Ohio. F. R. Cortes of the Development Group, Refractomet Division, Universal-Cyclops Steel Corporation was the engineer in charge.

Since the nature of this work is of interest to so many fields of endeavor, your comments are solicited as to the potential utilization of the material produced under this contract. In this manner, it is felt that a full realization of the resultant material produced will be accomplished.

PUBLICATION REVIEW

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TABLE OF CONTENTS

I Introduction 1

II Phase IV Intermediate Breakdown and Evaluation
   A. Continuation of Mold Out Evaluation 1
      1. Mold Out Contamination 2
      2. Structure and Recrystallization of Mold Out 5
      3. Tensile Properties of Mold Out 18

III Summary and Conclusions 23

IV Phase V Program Outline 26
LIST OF TABLES

<p>| | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>Iron and Interstitial Analyses of .125&quot; Mold Out</td>
<td>3</td>
</tr>
<tr>
<td>II</td>
<td>Recrystallization Temperatures and Grain Size of Nominal 0.125&quot; Mold Out</td>
<td>6</td>
</tr>
<tr>
<td>III</td>
<td>Room Temperature Ultimate Strength of .125 Mold Out</td>
<td>21</td>
</tr>
<tr>
<td>IV</td>
<td>Room Temperature Tensile Ductility .125 Mold Out</td>
<td>22</td>
</tr>
<tr>
<td>V</td>
<td>Quality Ratings for Phase III Sheet Bar and Phase IV Mold Out</td>
<td>24</td>
</tr>
</tbody>
</table>
# LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>As Rolled Mold Out Structures</td>
<td>8</td>
</tr>
<tr>
<td>2.</td>
<td>As-Rolled Structures of Mold Out Rolled at 2800°F</td>
<td>9</td>
</tr>
<tr>
<td>3.</td>
<td>Effect of Annealing Temperature on the Hardness of Nominal 0.125&quot; Mold Out Rolled at 2000°F</td>
<td>10</td>
</tr>
<tr>
<td>4.</td>
<td>Effect of Annealing Temperature on the Hardness of Nominal 0.125&quot; Mold Out Rolled at 2400°F</td>
<td>12</td>
</tr>
<tr>
<td>5.</td>
<td>Progress of Recrystallization in Mold Out T3 Produced From Hot Forged Sheet Bar by Rolling at 2400°F</td>
<td>13</td>
</tr>
<tr>
<td>6.</td>
<td>Effect of Annealing Temperatures on the Hardness of Nominal 0.125&quot; Mold Out Rolled at 2800°F</td>
<td>15</td>
</tr>
<tr>
<td>7.</td>
<td>Effect of Annealing Temperatures on the Hardness of Nominal 0.125&quot; Mold Out Rolled at 3200°F</td>
<td>16</td>
</tr>
<tr>
<td>8.</td>
<td>Effect of Rolling Temperature on Temperature for 50% Recrystallization of .125&quot; Mold Out</td>
<td>19</td>
</tr>
<tr>
<td>9.</td>
<td>Phase V Forging Outline</td>
<td>27</td>
</tr>
<tr>
<td>10.</td>
<td>Phase V Sheet Rolling Outline</td>
<td>28</td>
</tr>
</tbody>
</table>
I. Introduction

This program was designed to evaluate the potential of the InFab facility for the production of TZM alloy sheet. The evaluation consists of the following six phase program.

Phase I Literature Survey
Phase II Ingot Production and Evaluation
Phase III Production of Sheet Bar
Phase IV Intermediate Breakdown
Phase V Production of Evaluation Sheets
Phase VI Production of Sheets by Best Techniques

During this report period the Phase IV evaluation of intermediate gage was completed. This report covers the results of the Phase IV evaluation and the determination of the best sheet bar forging and mold out rolling practices for the Phase V program. The Phase V program was designed and billets were prepared for forging in InFab when operation resumes approximately May 1, 1963.

II. Phase IV Intermediate Breakdown and Evaluation

The previous interim report described sheet bar rolling procedures to nominal 0.125 mold out. Of the 34 sheet bar sections rolled, 29 pieces of mold out were produced for full evaluation.

A. Continuation of Mold Out Evaluation

Initial evaluation of 0.125" mold out was reported in the previous interim report. Included were the results of ultrasonic inspection, determination of
surface contamination and hardness surveys. The remainder of this evaluation was completed during this report period.

1. Mold Out Contamination

Visible contamination layers were reported previously along with chemical analyses to determine surface iron pickup during rolling. Wrought surface layers on recrystallized mold out (attributed to interstitial contaminants) appeared on all mold out rolled at 2800°F and on a few sections rolled at 2400°F but not on all mold out rolled at 2000°F. Mold out rolled at 3200°F, exhibited lightly worked structures, and showed no wrought contamination layers either as-rolled or recrystallized as the high rolling temperature offset any effects of contamination on recrystallization during rolling and the lightly worked surface prevented any delineation between surface and substrate after annealing. Chemical analyses revealed that surface iron pickup became a problem at rolling temperatures above 2800°F.

Interstitial analyses of mold out, during this report period (Table I) revealed that:
### TABLE I

<table>
<thead>
<tr>
<th>Mold Out Code</th>
<th>Rolling Temp.</th>
<th>Reheats</th>
<th>Gauge As Rolled</th>
<th>Chemistry of As-Rolled Cross Section</th>
<th>0-Lab 1</th>
<th>0-Lab 2</th>
<th>Surface Removed (per side)</th>
<th>Chemistry of Surface Milled Cross Section</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>3200</td>
<td>6</td>
<td>.138</td>
<td>Fe:  -  C:  0.010  N:  0.005</td>
<td>-</td>
<td>-</td>
<td>.025&quot;</td>
<td>Fe:  .0018  C:  .034  N:  .0014  O:  .0010</td>
</tr>
<tr>
<td>T9</td>
<td>3200</td>
<td>7</td>
<td>.151</td>
<td>Fe:  -  C:  0.034  N:  0.006</td>
<td>-</td>
<td>-</td>
<td>.025&quot;</td>
<td>Fe:  .0015  C:  .036  N:  .0009  O:  .0010</td>
</tr>
<tr>
<td>T17</td>
<td>3200</td>
<td>-</td>
<td>.162</td>
<td>Fe:  -  C:  0.055  N:  0.004</td>
<td>-</td>
<td>-</td>
<td>.025&quot;</td>
<td>Fe:  .0015  C:  .032  N:  .0014  O:  .0005</td>
</tr>
<tr>
<td>T10</td>
<td>2800</td>
<td>8</td>
<td>.131</td>
<td>Fe:  .0025  C:  0.034  N:  0.009</td>
<td>-</td>
<td>-</td>
<td>.020&quot;</td>
<td>Fe:  .0015  C:  .033  N:  .0012  O:  .0012</td>
</tr>
<tr>
<td>T14</td>
<td>2800</td>
<td>8</td>
<td>.129</td>
<td>Fe:  -  C:  0.050  N:  0.008</td>
<td>-</td>
<td>-</td>
<td>.020&quot;</td>
<td>Fe:  .0015  C:  .034  N:  .0008</td>
</tr>
<tr>
<td>T18</td>
<td>2800</td>
<td>-</td>
<td>.126</td>
<td>Fe:  .0025  C:  0.055  N:  0.016</td>
<td>-</td>
<td>-</td>
<td>.020&quot;</td>
<td>Fe:  .0015  C:  .034  N:  .0008</td>
</tr>
<tr>
<td>T11</td>
<td>2400</td>
<td>10</td>
<td>.129</td>
<td>Fe:  .0025  C:  0.030  N:  0.006</td>
<td>-</td>
<td>-</td>
<td>.015&quot;</td>
<td>Fe:  .0015  C:  .032  N:  .0014  O:  .0005</td>
</tr>
<tr>
<td>T19</td>
<td>2400</td>
<td>8</td>
<td>.131</td>
<td>Fe:  .0025  C:  0.032  N:  0.0118</td>
<td>-</td>
<td>-</td>
<td>.010&quot;</td>
<td>Fe:  .0015  C:  .033  N:  .0012  O:  .0012</td>
</tr>
<tr>
<td>T12</td>
<td>2000</td>
<td>8</td>
<td>.133</td>
<td>Fe:  &lt;.0015  C:  .031  N:  .0006</td>
<td>-</td>
<td>-</td>
<td>.010&quot;</td>
<td>Fe:  .0015  C:  .034  N:  .0008</td>
</tr>
<tr>
<td>T20</td>
<td>2000</td>
<td>8</td>
<td>.131</td>
<td>Fe:  -  C:  .034  N:  .045</td>
<td>-</td>
<td>-</td>
<td>.010&quot;</td>
<td>Fe:  .0015  C:  .034  N:  .0008</td>
</tr>
</tbody>
</table>
1. Nitrogen contamination did not occur as nitrogen contents did not differ in as-rolled and surface milled mold out.

2. Carbon contamination occurred only at the very highest rolling temperature, 3200°F. Mold out rolled at 3200°F showed carbon levels of .055% or almost double those for mold out rolled at the lower temperatures or for surface milled samples.

3. Oxygen contamination occurred in all cases but there was no correlation with rolling temperatures.

The graphite suscepter of the induction heating furnace is the known source of the carbon contamination. During this report period the induction furnace was replaced with a tungsten element resistance heated furnace which should prevent carbon contamination during future sheet rolling operations.

The source of the oxygen contamination at temperatures below 3200°F has not as yet been accurately determined but it is also believed to be a function of the induction furnace temperature though this was not necessarily indicated by analyses of the mold out. The difficulty in obtaining reproducible oxygen
analyses between laboratories is readily apparent from Table I. Initial analyses (Lab-1) of as-rolled mold out cross sections showed oxygen levels of about 50 ppm regardless of the rolling temperature. After removing from .010 to .025" per side by milling and pickling the oxygen contents dropped to about 10 ppm. A recheck of the analyses (Lab 2) on as-rolled mold out showed oxygen levels to be from 50 to 300% greater than results obtained from Lab 1. In addition results from Lab 2 give some indication of increasing oxygen contamination with rolling temperature up to 2800°F. The lowest oxygen levels reported by both laboratories were for mold-out rolled at 3200°F. Since carbon contamination was at its peak at 3200°F it may have been a factor contributing to the lower level of oxygen contamination.

2. Recrystallization and Structures of .125" Mold Out

The effects of rolling temperature and sheet bar forging practice on mold out structures, recrystallization temperatures and recrystallized grain size are summarized in Table II. As rolled mold out structures can
<table>
<thead>
<tr>
<th>Sheet Bar</th>
<th>Mold Out</th>
<th>Furnace or Rolling Temperature °F</th>
<th>As-Rolled Hold Out Structure</th>
<th>As Rolled Hardness</th>
<th>1 Hr. ReXl Temp. Est. From Micros 50%</th>
<th>100% ReXl Temp. Est. From Micros</th>
<th>ReXl Grain Size</th>
<th>(ASTM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extruded &amp; Forged</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1098A1 T1</td>
<td>3200</td>
<td>Hot Rolled to Lightly Wo.</td>
<td>276</td>
<td>-</td>
<td>*</td>
<td>&gt;3000°F</td>
<td>4.5</td>
<td></td>
</tr>
<tr>
<td>T3</td>
<td>2400</td>
<td>Fibered</td>
<td>376</td>
<td>2750°F</td>
<td>*</td>
<td>2900°F</td>
<td>6.5</td>
<td></td>
</tr>
<tr>
<td>1098A2 T5</td>
<td>3200</td>
<td>Hot Rolled to Lightly Wo.</td>
<td>276</td>
<td>-</td>
<td>*</td>
<td>&gt;3000</td>
<td>Non Uniform</td>
<td></td>
</tr>
<tr>
<td>T6</td>
<td>2800</td>
<td>Fibered</td>
<td>327</td>
<td>2650</td>
<td>2850</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T8</td>
<td>2000</td>
<td>Fibered</td>
<td>405</td>
<td>2650</td>
<td>2850</td>
<td>7.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1098A3 T9</td>
<td>3200</td>
<td>Hot Rolled to Lightly Wo.</td>
<td>281</td>
<td>-</td>
<td>*</td>
<td>&gt;3000</td>
<td>Non Uniform</td>
<td></td>
</tr>
<tr>
<td>T10</td>
<td>2800</td>
<td>Worked, Fibered Centered Fibered</td>
<td>289</td>
<td>2850</td>
<td>*</td>
<td>2900</td>
<td>4.5</td>
<td></td>
</tr>
<tr>
<td>T11</td>
<td>2400</td>
<td>Fibered</td>
<td>348</td>
<td>2650</td>
<td>2900</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T12</td>
<td>2000</td>
<td>Fibered</td>
<td>373</td>
<td>2600</td>
<td>2850</td>
<td>7.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1098B1 T13</td>
<td>3200</td>
<td>Hot Rolled to Lightly Wo. Fibered + Trace ReXl</td>
<td>262</td>
<td>-</td>
<td>*</td>
<td>&gt;3000</td>
<td>3-6</td>
<td></td>
</tr>
<tr>
<td>T14</td>
<td>2800</td>
<td>Fibered</td>
<td>333</td>
<td>2725</td>
<td>2850</td>
<td>6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T15</td>
<td>2400</td>
<td>Fibered</td>
<td>373</td>
<td>2725</td>
<td>*</td>
<td>2800</td>
<td>6.5</td>
<td></td>
</tr>
<tr>
<td>1098B2 T17</td>
<td>3200</td>
<td>Worked, Not Fibered</td>
<td>257</td>
<td>2800</td>
<td>2950</td>
<td>3-6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T18</td>
<td>2800</td>
<td>Worked, Fibered Centered</td>
<td>289</td>
<td>2750</td>
<td>2900</td>
<td>Non Uniform</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T19</td>
<td>2400</td>
<td>Heavily Fibered</td>
<td>333</td>
<td>2600</td>
<td>2900</td>
<td>3-6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T20</td>
<td>2000</td>
<td>Heavily Fibered</td>
<td>360</td>
<td>2550</td>
<td>2700</td>
<td>6.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1098B3 T21</td>
<td>3200</td>
<td>Worked</td>
<td>297</td>
<td>2800</td>
<td>2950</td>
<td>3-6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T22</td>
<td>2800</td>
<td>Worked, Fibered Centered Fibered</td>
<td>312</td>
<td>2650</td>
<td>2725</td>
<td>4.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T23</td>
<td>2400</td>
<td>Fibered</td>
<td>330</td>
<td>2500</td>
<td>2650</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T24</td>
<td>2000</td>
<td>Heavily Fibered</td>
<td>354</td>
<td>2500</td>
<td>2800</td>
<td>7.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1098B4 T25</td>
<td>3200</td>
<td>Worked</td>
<td>302</td>
<td>2625</td>
<td>*</td>
<td>2725</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>T26</td>
<td>2800</td>
<td>Worked, Fibered Centered Fibered</td>
<td>279</td>
<td>2650</td>
<td>*</td>
<td>2900</td>
<td>Non Uniform</td>
<td></td>
</tr>
<tr>
<td>T27</td>
<td>2400</td>
<td>Fibered</td>
<td>336</td>
<td>2500</td>
<td>2600</td>
<td>7.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T28</td>
<td>2000</td>
<td>Heavily Fibered</td>
<td>357</td>
<td>2475</td>
<td>2575</td>
<td>7.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cast and Forged</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1099A T34</td>
<td>2800</td>
<td>Fibered + Trace ReXl</td>
<td>333</td>
<td>2725</td>
<td>2825</td>
<td>6.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T35</td>
<td>2400</td>
<td>Heavily Fibered</td>
<td>360</td>
<td>2600</td>
<td>2800</td>
<td>7.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1099C T38</td>
<td>2800</td>
<td>Heavily Worked Fibered</td>
<td>299</td>
<td>2750</td>
<td>*</td>
<td>2900</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>T39</td>
<td>2400</td>
<td>Fibered</td>
<td>351</td>
<td>2625</td>
<td>2800</td>
<td>6.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T40</td>
<td>2000</td>
<td>Heavily Fibered</td>
<td>370</td>
<td>2375</td>
<td>2750</td>
<td>7.5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
be grouped with rolling temperature into three classes as follows:

<table>
<thead>
<tr>
<th>Rolling Temp °F</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>2000 &amp; 2400</td>
<td>Fibered</td>
</tr>
<tr>
<td>2800</td>
<td>Worked &amp; Fibered Center</td>
</tr>
<tr>
<td>3200</td>
<td>Hot Rolled to Lightly Worked</td>
</tr>
</tbody>
</table>

Structural variations were greatest in mold out rolled at 2800° as this temperature falls between the hot and cold working range for TZM. Figures 1 and 2 show typical structures for each group as well as some variations between mold out rolled at 2800°F.

The annealing response of TZM mold out rolled at 2000°F is shown in Figure 3. Hardnesses dropped to recrystallized values of 200-210 after a 1 hour anneal at 2600°F. Only six pieces of mold out were rolled successfully at 2000°F as 3 sections from hot forged sheet bars were not fabricable. Of the six produced mold out T8, from a hot forged sheet bar, maintained the highest annealed hardness and required 2850°F for 100% recrystallization.

Though hardnesses dropped rapidly upon annealing at 2600°F the microstructures revealed
that temperatures of 2700 to 2850°F were required for 100% recrystallization as shown in Table II except for mold out T24 and T28. These latter two, having been produced from heavily hot-cold forged sheet bars 1098Bc and 1098B4, were fully recrystallized at 2600°F. A fine recrystallized grain size of about ASTM 7.5 was produced in all mold out sections rolled at 2000°F indicating, as expected, finer grain size with lowest prior rolling temperature.

Except for sections T3 and T15, Figure 4 shows that hardness of mold out rolled at 2400°F also dropped close to recrystallized values after a 2600°F anneal. Sections T3 and T15 were rolled from primarily hot forged sheet bars and maintained worked hardness levels of 275 and 250 QPH up to 2700°F with microstructural evidence of 100% recrystallization at 2900 and 2800°F respectively. Progress of recrystallization in mold out T3 is shown in Figure 5.

The lowest recrystallization temperatures for mold out rolled at 2400°F occurred in sections T23 and T27 which were rolled from heavily cold forged sheet bar 1098B3 and 1098B4. Recrystallized grain sizes
**FIGURE 4**

EFFECT OF ANNEALING TEMPERATURE ON THE HARDNESS
OF NOMINAL 0.125" MOLDOUT ROLLED AT 2400°F

MOLD OUT  SHEET BAR FORGING TEMP.

- **T3** FROM 1098 A1 3810 - 3350
- **T11**  1098 A3 3810 - 2350
- **T15**  1098 B1 3200 - 2950
- **T19**  1098 B2 3120 - 2300
- **T23**  1098 B3 2600 - 2310
- **T27**  1098 B4 2320 - 1950
- **T35**  1099 A 3870 - 2400
- **T39**  1099 C 3850 - 2770

MOLD OUT ROLLED AT 2400°F
FIGURE 5
PROGRESS OF RECRYSTALLIZATION IN MOLD OUT T3 PRODUCED FROM HOT FORGED SHEET BAR BY ROLLING AT 2400°F
for mold out rolled at 2400°F averaged about ASTM 6.9 and ranged from 6.5 to 7.5. Thus increasing the rolling temperature from 2000 to 2400°F resulted in only a slight coarsening of recrystallized grain size amounting to about one half an ASTM number.

Mold out rolled at 2800 and 3200°F developed as-rolled hardnesses ranging from 255 to 332 in comparison to hardness ranging from 330 to 400 for mold out rolled at 2000 and 2400°F. As a result softening upon annealing appeared more gradual, Figures 6 and 7, for mold out rolled at the higher temperatures. Annealing temperatures from 2750°F to 2800°F were necessary to produce recrystallized hardness levels in mold out rolled at 2800°F except for sections T18, T22 and T26. These sections were produced from hot cold forged sheet bar and softened at about 2650 to 2700°F. Softening temperatures for mold out rolled at 3200 were the same as for mold out rolled at 2800°F, about 2750 to 2800°F. The microstructures indicated that recrystallization was complete at temperatures ranging from 2725 to 2900°F for mold out rolled at 2800 and at temperatures
FIGURE 6
EFFECT OF ANNEALING TEMPERATURE ON THE HARDNESS OF NOMINAL 0.125" MOLDOUT ROLLED AT 2800°F
FIGURE 7
EFFECT OF ANNEALING TEMPERATURE ON THE HARDNESS OF NOMINAL 0.125" MOLDOUT ROLLED AT 3200°F

RTA-0313
from 2725 to >3000°F for mold out rolled at 3200°F. The very high (>3000°) recrystallization temperatures for mold out rolled at 3200°F resulted from the small amount of work retained at the high rolling temperature.

Recrystallized grain size of mold out rolled at 2800°F ranged from ASTM 4.5 to 7 with an average about 5.75 while mold out rolled at 3200 exhibited coarse, non uniform structures averaging about ASTM 4.5.

Mold out sections which exhibited the outstanding hardness stability with annealing temperatures are as follows:

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<td>T1</td>
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<td>2800</td>
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<td>3850°F to 3200 min.</td>
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<td>3200°F to 2950 min.</td>
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<td>1099C*</td>
<td>3850°F to 2770 min.</td>
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</table>

* Forged directly from ingot
Significant observations are:

1. Outstanding hardness stability is exhibited by mold out rolled at either 2400 or 2800°F.

2. Sheet bars from which each mold out was rolled were primarily hot forged.

The supporting microstructural evidence for (50%) recrystallization is summarized in Figure 8. Mold out from cold forged sheet bar exhibit the lowest recrystallization temperatures while mold out from hot forged sheet bar showing good hardness stability (circled code nos.) exhibit the highest recrystallization temperatures. Mold out produced from directly forged ingots exhibit only average recrystallization temperatures.

3. **Tensile Properties of .125" Mold Out**

Room temperature tensile properties were obtained in the following conditions:

- As-Rolled - Longitudinal Direction Only
- As Rolled and Conditioned Surface - Longitudinal and Transverse
- Stress Relieved and Conditioned Surface - Longitudinal and Transverse
- Recrystallized and Conditioned Surface - Longitudinal and Transverse
FIGURE 8
EFFECT OF ROLLING TEMPERATURE ON TEMPERATURE FOR 50% RECRYSTALLIZATION OF 0.125" MOLDOUT
Ultimate strength and elongation for each of the above conditions is summarized in Tables III and IV. Comparing longitudinal tensile properties of as-rolled mold out with as-rolled and conditioned mold out reveals the detrimental surface contamination effect. In particular, mold out rolled at 2400 and above exhibited no as-rolled tensile ductility, Table IV, in the longitudinal direction. (Transverse properties were not determined as-rolled.) However surface conditioning by belt grinding produced tensile elongations of 5 to 15% in the longitudinal direction and 2 to 10% transverse. Surface conditioning plus a stress relief further increased tensile ductility of most of the mold out sections. However, mold out sections T1 and T5 hot rolled at 3200°F from hot forged sheet bar showed no improvement in elongation with either the surface conditioning or stress relief treatment. Recrystallization anneals greatly improved tensile elongation in almost every case.

A technique for rating mold out ductility was devised giving equal weight to as-rolled and stress relieved elongations and one half weight
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<tr>
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<th>Mold Out</th>
<th>Rolling Temp.</th>
<th>Annealing Temp.</th>
<th>Longitudinal Elongation</th>
<th>Transverse Elongation</th>
<th>Overall % El. Rating</th>
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**TABLE IV**

TENSILE DUCTILITY OF .125" MOLD OUT AT ROOM TEMPERATURE

<table>
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<tr>
<th>Sheet Bar</th>
<th>Mold Out</th>
<th>Rolling Temp</th>
<th>Annealing Temp</th>
<th>Longitudinal Elongation</th>
<th>Transverse Elongation</th>
<th>Overall % El. Rating</th>
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to recrystallized values. The resultant elongation rating is also listed in Table IV. On the basis of this rating, sheet bar 1098B2, forged between 3200 and 2300°F, produced the most uniformly ductile mold out. Of further significance is the fact that the best ductility rating for mold out from any one sheet bar occurred for the most part in sections rolled either at 2400 or 2800°F. Mold out rolled at 3200 showed the poorest overall ductility.

III Summary and Conclusions

The results obtained from the Phase III Sheet Bar Evaluation, reported in the last interim report, and the Phase IV Mold Out Evaluation of this report are summarized in Table V using an arbitrary rating system for the various properties. Sheet bar exhibiting the best quality rating of (18) (1098B1), was entirely hot forged at the low end of the hot work range for TZM which prevented excess grain growth. Sheet bars 1098A3 and 1098B2 exhibiting the next best quality ratings (15 and 16) were initially hot forged and then finish forged in the hot cold work range. Sheet bars produced by forging primarily at very high temperatures 3850°F, were low rated on the basis of grain size, carbide distribution and rollability while sheet bar forged at low temperatures 2000 to 2700 were low rated primarily because of low recrystallization temperatures
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which would undoubtedly be transmitted to finished sheet. Sheet bar produced by direct ingot forging were also low rated due to coarse non uniform carbides and structures. In addition sheet bar forged directly from ingot contained more surface contamination (both iron and interstitial) as many more forging blows and reheats were required. Though a rating was not applied for degree of contamination it is a factor in the choice of forging or rolling variables for the Phase V program.

Although the grain size of hot forged sheet bar was more coarse than the recrystallized grain size of hot-cold forged sheet bar, this difference had little effect on the grain size of recrystallized mold out (Table V). The recrystallized mold out grain size was instead more dependent upon rolling temperatures. However, the sheet bar forging practice did have considerable influence on other mold out properties such as recrystallization temperature, carbide distribution and tensile ductility. On the basis of these properties the three sheet bar rated as highest quality were also equal or superior to other sheet bar practices in mold out form, sub totals 2 and 2A of Table V.

Individual mold out sections with stand out ratings were T9 and 10, T14 and 15, and T17, 18, 19 and 20 all rolled from the three top quality sheet bars. The latter four in particular, all rolled from sheet bar 1098B2 (forged at 3200 to 2300°F) exhibited the most consistently good quality ratings for any one
group of mold out sections. The mold out section exhibiting the very highest quality rating, T14, was rolled at 2800°F from sheet bar entirely hot forged in the 3200°F range.

A cursory examination of Table V for outstanding individual property ratings as indicated by check marks reveals that in almost every case the standout values for recrystallization, elongation, etc., occurred in mold out rolled either at 2400 or 2800°F. Of particular note, was the fact that tensile ductility of mold out improved as rolling temperatures decreased to 2400°F but a further decrease to 2000°F did not result in any further ductility improvement.

IV Phase V Program

The overall objective of this investigation is to develop an improved TZM alloy sheet product through the use of the InFab facility. Primarily this means the evaluation of higher processing temperatures than those available for present commercial TZM practice. The proposed Phase V Program design resulting from the Phase III and Phase IV evaluation is shown in Figures 9 and 10. A total of 35 variables to .040" sheet are incorporated. Evaluation of reductions is limited to two and there is no evaluation of intermediate anneals as both parameters have been fully evaluated on previous programs, the results of which have been considered in this program design. Forging and rolling variables incorporated

(1) Navy Sheet Rolling Program Contract NOas 59-6142-C
EXTRUSION BILLET (4 PCS.)

4 1/16" DIA. X 20" L.G. — 95 LBS

(1 BILLET)

FLAT DIE FORGING

(2 BILLET)

FORGE AT 3200°F
2300° MIN.

1 SHEET BAR ROLLED
80% TO .040 SHEET

3 SHEET BARS ROLLED
90% TO .040 SHEET

FORGE AT 3850°F
2300° MIN.

PHASE X

FORGING VARIABLES

FIGURE 9
**PROCESS FROM NAVY SHEET ROLLING PROGRAM**

1. ROLL TO .400" M.O. AT 2400°F
2. R.E.X. AT 3000°F & CONDITION
3. CUT 3 PCS.

**1ST MOLD OUT**

1. ROLL TO .400" M.O. AT 2800°F
2. R.E.X. AT 3000°F & CONDITION
3. CUT 2 PCS.

**2ND MOLD OUT**

1. ROLL TO .140" FINAL MOLD OUT AT 2800°F
2. CONDITION & STRESS RELIEVE AT 2200°F
3. STR. REL. AT ___
4. TO BE DETERMINED___

**CROSS ROLL TO .040" AT 1600°F**

1. CROSS ROLL TO .040" AT 1800°F
2. CROSS ROLL TO .040" AT 1800°F
3. CROSS ROLL TO .040" AT 1800°F

**FINISH SHEET APPROX. 10-12" WIDE X 18-24" L.G. X 0.040"**

**PHASE II SHEET EVALUATION**

**FIGURE 10**

**PHASE II SHEET ROLLING VARIABLES**

RTB-0038
from Phase III and IV are as follows:

Sheet Bar - Three best practices from Phase III

1. 3250 start, 2950°F min. (2 pcs)
2. 3250 start, 2300°F Min. (1 pcs)
3. 3850 start, 2300°F Min. (1 pcs)

Mold Out - Two best Phase IV practices

1. 2400°F
2. 2800°F

Recrystallized mold out will then be rolled to final mold out at temperatures ranging from 2000 to 2800°F. Stress relief temperatures will be determined and final sheet produced at two temperatures 1400 and 1800°F. In addition one section from each sheet bar will be rolled to sheet according to the process developed on the Navy's Molybdenum Sheet Rolling Program. The .040" sheet produced from the 3 forging practices will contain a total of 90% reduction from recrystallized mold out. An additional reduction will be evaluated however by rolling a fourth sheet bar (from the 2nd forging practice Figure 9) to the same temperature schedule in Figure 9 but to a total of 80% reduction in .040" sheet.
<table>
<thead>
<tr>
<th>Company Name</th>
<th>Addressee</th>
<th>Location</th>
</tr>
</thead>
<tbody>
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