MECHANICAL PROPERTIES OF OXIDE DISPERSION STRENGTHENED MOLYBDENUM ALLOYS

by

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Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science

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Contents

LIST OF FIGURES ......................................................... iii

LIST OF TABLES ......................................................... vi

ACKNOWLEDGEMENTS .................................................... vii

1 INTRODUCTION ....................................................... 1
  1.1 Development of ODS Molybdenum Alloys ................. 3
  1.2 Chemistry and Processing ......................................... 5
  1.3 Microstructure and Recrystallization ......................... 6
  1.4 Mechanical Properties ............................................ 8
    1.4.1 Low Temperature ............................................. 9
    1.4.2 High Temperature ........................................... 10
  1.5 Applications of High Temperature Molybdenum Alloys .... 11

2 RESEARCH OBJECTIVES ............................................. 12

3 PROCEDURE ......................................................... 14
  3.1 Source Material .................................................. 14
  3.2 Heat Treatment ................................................... 15
  3.3 Mechanical Testing ............................................... 16
    3.3.1 Hardness ...................................................... 17
3.3.2 Bend Testing ................................................. 17
3.3.3 Bending Analysis ........................................... 19
3.3.4 Tensile Testing ............................................. 23

3.4 Microstructure Analysis ....................................... 23
  3.4.1 Optical Metallography .................................... 24
  3.4.2 Scanning Electron Microscopy with EDX Analysis ...... 26
  3.4.3 Transmission Electron Microscopy ....................... 27

4 RESULTS AND ANALYSIS ....................................... 28
  4.1 Mechanical Testing ......................................... 28
    4.1.1 Qualitative Bend Results ............................ 28
    4.1.2 Quantitative Bend Results ......................... 30
    4.1.3 Hardness ............................................. 36
    4.1.4 Tensile Testing ...................................... 37
  4.2 Microstructure Analysis .................................... 42
    4.2.1 Optical Metallography ............................... 42
    4.2.2 Scanning Electron Microscopy with EDX ........... 47
    4.2.3 Transmission Electron Microscopy ................ 52

5 CONCLUSIONS .................................................. 55

REFERENCES ..................................................... 57
## List of Tables

<table>
<thead>
<tr>
<th>Table</th>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Tensile Properties of Molybdenum Alloys at 1000°C</td>
<td>10</td>
</tr>
<tr>
<td>3.2</td>
<td>Heat Treatment</td>
<td>16</td>
</tr>
<tr>
<td>3.3</td>
<td>Bend Radii Employed</td>
<td>17</td>
</tr>
<tr>
<td>4.4</td>
<td>Grain Size</td>
<td>44</td>
</tr>
</tbody>
</table>
# List of Figures

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-1</td>
<td>Creep Properties</td>
<td>11</td>
</tr>
<tr>
<td>3-2</td>
<td>Test Coupon Geometry</td>
<td>15</td>
</tr>
<tr>
<td>3-3</td>
<td>Test Coupon Orientations</td>
<td>15</td>
</tr>
<tr>
<td>3-4</td>
<td>Bend Test Experimental Matrix</td>
<td>19</td>
</tr>
<tr>
<td>3-5</td>
<td>Bend Test Setup</td>
<td>20</td>
</tr>
<tr>
<td>3-6</td>
<td>Bend Test Sequence</td>
<td>20</td>
</tr>
<tr>
<td>3-7</td>
<td>Bend Quality Definition</td>
<td>21</td>
</tr>
<tr>
<td>3-8</td>
<td>Bend Angle</td>
<td>23</td>
</tr>
<tr>
<td>3-9</td>
<td>Cross Section used for Grain Size Measurement</td>
<td>25</td>
</tr>
<tr>
<td>3-10</td>
<td>Grain Size Measurement Grid</td>
<td>25</td>
</tr>
<tr>
<td>3-11</td>
<td>Four Principle Test Directions Observed</td>
<td>26</td>
</tr>
<tr>
<td>4-12</td>
<td>Hardness Measurements</td>
<td>29</td>
</tr>
<tr>
<td>4-13</td>
<td>Chart Comparing Bend Angle for all Experimental Conditions</td>
<td>31</td>
</tr>
<tr>
<td>4-14</td>
<td>Bend Angle for Longitudinal Samples</td>
<td>31</td>
</tr>
<tr>
<td>4-15</td>
<td>Bend Angle for Transverse Samples</td>
<td>32</td>
</tr>
<tr>
<td>4-16</td>
<td>Key for Quantitative Bend Charts</td>
<td>32</td>
</tr>
<tr>
<td>4-17</td>
<td>Chart Comparing Yield Strength for all Experimental Conditions</td>
<td>34</td>
</tr>
<tr>
<td>4-18</td>
<td>Yield Strength for Longitudinal Samples</td>
<td>34</td>
</tr>
<tr>
<td>4-19</td>
<td>Yield Strength for Transverse Samples</td>
<td>35</td>
</tr>
<tr>
<td>4-20</td>
<td>Chart Comparing Modulus for all Experimental Conditions</td>
<td>35</td>
</tr>
<tr>
<td>Section</td>
<td>Title</td>
<td>Page</td>
</tr>
<tr>
<td>---------</td>
<td>------------------------------------------------------------</td>
<td>------</td>
</tr>
<tr>
<td>4-21</td>
<td>Modulus for Longitudinal Samples</td>
<td>36</td>
</tr>
<tr>
<td>4-22</td>
<td>Modulus for Transverse Samples</td>
<td>37</td>
</tr>
<tr>
<td>4-23</td>
<td>Hardness Measurements</td>
<td>38</td>
</tr>
<tr>
<td>4-24</td>
<td>Tensile Yield Strength</td>
<td>39</td>
</tr>
<tr>
<td>4-25</td>
<td>Tensile Ultimate Tensile Strength</td>
<td>40</td>
</tr>
<tr>
<td>4-26</td>
<td>Tensile Elongation</td>
<td>41</td>
</tr>
<tr>
<td>4-27</td>
<td>Representative Optical Micrographs</td>
<td>43</td>
</tr>
<tr>
<td>4-28</td>
<td>Average Grain Size</td>
<td>45</td>
</tr>
<tr>
<td>4-29</td>
<td>Grain Aspect Ratio</td>
<td>45</td>
</tr>
<tr>
<td>4-30</td>
<td>Incomplete Transformation after Low Temperature Heat Treatment</td>
<td>47</td>
</tr>
<tr>
<td>4-31</td>
<td>Abnormal Near-Surface Structure</td>
<td>48</td>
</tr>
<tr>
<td>4-32</td>
<td>Fracture Surface Showing Sample Thickness</td>
<td>49</td>
</tr>
<tr>
<td>4-33</td>
<td>High Magnification View of Selected Areas of Fracture Surface</td>
<td>49</td>
</tr>
<tr>
<td>4-34</td>
<td>Elemental Maps Showing Lanthanum Distribution</td>
<td>50</td>
</tr>
<tr>
<td>4-35</td>
<td>High Magnification View of Oxide Particles</td>
<td>51</td>
</tr>
<tr>
<td>4-36</td>
<td>TEM Microstructure for AR Condition</td>
<td>53</td>
</tr>
<tr>
<td>4-37</td>
<td>TEM Microstructure for 1800-2 Condition</td>
<td>53</td>
</tr>
<tr>
<td>4-38</td>
<td>Diffraction Pattern from Molybdenum Matrix</td>
<td>54</td>
</tr>
</tbody>
</table>
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Mechanical Properties of Oxide Dispersion Strengthened Molybdenum Alloys

by

David L. Poerschke

Abstract

Lanthanum oxide dispersion strengthened (ODS) molybdenum has excellent high temperature properties but low ductility.

The effect of heat treatment on the mechanical properties and formability of ODS-Mo sheet has been investigated in the temperature from 1500°C to 1800°C. Mechanical tests included three point bending, high temperature tensile testing, and hardness testing. Optical metallography, SEM, and TEM were used to evaluate the microstructure for each heat treatment condition.

The 1500°C heat treatment temperature led to cracking due to incomplete transformation to a stress relieved structure. Samples treated at 1800°C produced 90° bends but the bend surfaces of samples treated for long time at this temperature were not smooth. Heat treated samples had lower YS and UTS than the as-rolled material.

Grain growth along the rolling direction was faster than along transverse directions. This behavior is explained by the banding of oxide particles along the rolling direction.
Chapter 1

INTRODUCTION

Molybdenum is a body centered cubic refractory metal, characterized by a high melting point and good mechanical properties at high temperatures. These properties, coupled with low thermal expansion, high thermal conductivity, creep resistance, and relative ease of manufacture make it an ideal material for many aerospace, nuclear, electronic, and industrial applications [1][2].

Molybdenum is usually prepared through arc casting or powder metallurgy methods due to the high melting point. These processes generally yield material with low mechanical properties. To improve properties, the metal is processed through hot and cold working into bar, plate, sheet, and foil products. These processing methods are often accompanied by one or more stress relief anneal cycles [3]. In addition to increasing the yield and tensile strength of the material, this deformation processing also increases the ductility. The yield strength of as cast molybdenum has been shown to increase by approximately 125% through rolling with additional increase in strength after a heat treatment below the recrystallization temperature [4].

The primary mechanism for improved properties resulting from deformation processing is the reduction in grain size. The strength increases with the inverse of the square root of grain size through the Hall-Petch relationship[3].
While preserved at low and moderately elevated temperatures, this added strength is lost as the metal recrystallizes. An increase in grain size and embrittling due to the accumulation of impurities at the grain boundaries are detrimental to mechanical properties. Recrystallization occurs between \(900^\circ C\) and \(1250^\circ C\) depending on the amount of cold work in the material. Highly worked metal undergoes recrystallization at lower temperatures than moderately and lightly worked metals \([2][4]\). The loss of strength with recrystallization limits the application of molybdenum in structural components to environments below the recrystallization temperature.

To produce materials for structural applications above the molybdenum recrystallization temperature, alloys have been developed to remain stable at higher temperatures. Examples include solid solutions with titanium, known as Mo-Ti, titanium and zirconium, called TZM alloys, and rhenium, denoted Mo-XRe where X is the weight percentage of Re in the alloy. Alloys containing titanium and zirconium are strengthened by the formation of Ti and Zr carbides \([1]\). TZM exhibits higher overall strength than Mo-Ti alloys, but the strength of both falls drastically at temperatures of approximately \(1400^\circ C\), indicating the onset of recrystallization \([3]\). Mo-Re alloys have improved mechanical properties at high temperatures. However, the primary motivation for Re addition, especially at concentrations below 20 wt\%, is increased ductility at low temperatures \([5]\).

The strength of molybdenum alloys could be further improved through precipitation hardening, similar to techniques used in structural alloys of aluminum, magnesium, and titanium as well as some steels. Precipitation hardening is a heat treatment process in which a second phase is precipitated in the matrix. These small, finely dispersed precipitates retard dislocation motion, increasing material strength. While effective at low temperatures, the precipitated second phase dissolves back into the matrix as the temperature is
increased, eliminating the strengthening effect. For this reason application of precipitation strengthening is limited in refractory metals because most types of precipitates would dissolve in high temperature applications.

Mueller, et al[6], enumerate three criteria for the successful dispersion strengthening of molybdenum alloys for high temperature applications including the need for:

1. A dispersed strengthening phase that is stable at high temperatures,

2. Low solubility in the matrix phase, and

3. Low diffusivity in the matrix phase.

These requirements for an inert, dispersed phase capable of withstanding very high temperatures point logically to metal oxides. Many research efforts have been devoted to the development of oxide dispersion strengthened (ODS) molybdenum alloys.

1.1 Development of ODS Molybdenum Alloys

Aluminum potassium silicate (AKS) is often used as a dopant in molybdenum and tungsten wires to improve creep resistance [6][7]. These oxides are not actually dispersion strengtheners as their melting points are significantly lower than that of pure molybdenum metal. In these applications the strengthening effect is a result of thin potassium layer left along the grain boundaries after the aluminum and silicon evaporate from the alloy. Stable oxides of rare earth metals have melting temperatures closest to molybdenum and thus are a logical choice for dispersion strengthening [8].

Early rare earth oxide containing refractory alloys were produced using a dry, mechanically mixed powder metallurgy process wherein oxide particles
approximately 2µm in diameter were blended with molybdenum powder before
cold isostatic pressing and sintering[9]. While these alloys met the criteria
described above for successful dispersion strengthening, creep performance was
less than commercial molybdenum alloys[10]. The key drawback to these alloys
was the relatively large size of the dispersed particles.

Bianco and Buckman identified the need for finer particles and investigated
the impact of doping molybdenum alloys with CeO₂, La₂O₃, Y₂O₃, and ThO₂.
The effects of processing and composition were studied to determine their ef-
fect on the microstructure and mechanical properties. Wet and dry doping
processes were used with two and four volume percent additions of each ox-
ide. Results from these tests showed that oxide dispersion strengthened alloys
are suitable for applications at higher temperatures than possible with pure
molybdenum. Using the wet doping process, much smaller particle sizes were
achieved with the wet doped lanthana particles an order of magnitude smaller
than the previously described particles. [10].

Endo and colleagues have studied the mechanical and microstructure be-
behavior of molybdenum alloys doped with the rare-earth oxides Y₂O₃, La₂O₃,
Nd₂O₃, Sm₂O₃, and Gd₂O₃. Results from testing of thin wires showed a re-
crystallization temperature between 1100°C and 1200°C for undoped wire and
1500°C to 1600°C for similarly processed wire doped with the above oxides. In
addition to increasing the recrystallization temperature, presence of the dis-
persed oxide phase also changes the shape of recrystallized grains. In undoped
molybdenum, the fibrous microstructure yields to fine equiaxed grains during
recrystallization. In La₂O₃, Sm₂O₃, and Nd₂O₃ the grains grow during re-
crystallization but maintain a high aspect ratio with large interlocking grains.
The recrystallized Gd₂O₃ and Y₂O₃ microstructure has both small and large
equiaxed grains. The key difference between the two groups of oxide doped
alloys is the size and distribution of the oxide particles. Observed Gd₂O₃ and
Y₂O₃ particles were larger than La₂O₃, Sm₂O₃, and Nd₂O₃ particles so they are less effective barriers to grain boundary movement than a similar volume fraction of smaller particles. Creep behavior was determined using a high temperature sag test. Results from this testing showed that La₂O₃ provided the highest strength and creep resistance [8].

Buckman continued research on oxide dispersion strengthening with comparisons between La₂O₃, Y₂O₃, and ZrO₂, finding that La₂O₃ doping produced the highest UTS and significantly higher creep rupture properties than the other doping oxides [6].

The lanthanum oxide dispersion strengthened molybdenum (ODS Mo-La) has improved strength and reduced creep rate at high temperatures. However, ductility is reduced at low temperatures with brittle behavior below the ductile to brittle transition temperature (DBTT). The alloy also exhibits an increased recrystallization temperature, requiring higher temperature for the stress relieving anneals to improve low temperature ductility for forming.

1.2 Chemistry and Processing

Effort has been invested in developing a technique for producing fine oxide particle dispersion that is uniformly distributed through the bulk of the material. These efforts first identified rare earth oxides as promising choices. The field was narrowed based on desirable mechanical properties mechanical properties to leaving La₂O₃ as the most suitable candidate. Lanthanum doped ODS alloys are currently produced commercially through a powder metallurgy process. Wet and dry doping of lanthanum at concentrations of 2 and 4 volume percent were investigated. Wet doping of 2 volume percent was found to produce the highest mechanical properties [5][6][8]. A lanthanum nitrate salt is sprayed onto molybdenum oxide powder. This powder is reduced in
a hydrogen atmosphere where the salt is pyrolized, forming a fine, dispersed oxide phase[11].

1.3 Microstructure and Recrystallization

Deformation processing of this alloy is used to maximize mechanical properties. Rolling and drawing are used to cold work the metal to final strain hardening of as much as 99%. The resulting microstructure is very fine, composed of high aspect ratio pancake shaped grains. The oxide is present as dispersed particles less than 0.5\(\mu\)m in diameter in the matrix and as stringers or ribbons along the longitudinal grain boundaries[12]. This size and particle distribution is thought to be a result of deformation of the oxide particles during thermomechanical processing. As sintered, particles are on the order of 2-3\(\mu\)m. During rolling, these particles are elongated, forming stringers. Additional deformation coupled with thermal treatment breaks some of these stringers into the smaller particles observed in sheet product[10].

Transmission electron microscopy has been used to characterize the microstructure of this and other molybdenum alloys. Wright and colleagues have shown that when subjected to high deformation, dislocation cells develop within grains. Shifted spots in the diffraction patterns indicate slight misorientations between these subgrains[13]. TEM analysis of the ODS alloys described in [10] has focused on the shape of oxide particles and the interface between these particles and the molybdenum matrix. The results indicate that mechanical processing of alloys containing hard, non-deformable particles such as yttria causes microvoid formation at the interface while the processing is accommodated by particle deformation in softer oxides such as ceria and lanthana.

The strength of metals decreases slowly with increasing anneal temperature
until the recrystallization temperature is reached, marked by a sharp decrease in strength and significant change in grain morphology. Authors report two key differences exist between recrystallization of pure molybdenum and the ODS Mo-La alloy. Arc cast and powder metallurgy molybdenum products have a recrystallization temperature of around 1200°C and the recrystallized grains are equiaxed and larger than the microstructure before annealing while the ODS recrystallization temperature is higher and grains maintain high aspect ratio after heat treatment[4][8][14].

The recrystallization of molybdenum alloys has been studied over a range of temperatures and initial texture. Fujii and Wantabe report that some doped alloys are susceptible to Neilsen-type [15] secondary recrystallization with this abnormal grain grown starting near the surface of the material and progressing along the surface and through the thickness of the material[16][17].

Oxide particles influence both the nucleation of strain free grains and the growth of these grains during recrystallization. During nucleation, fine particles with small inter-particle spacing are most effective at pinning dislocations and grain boundaries. If particles are large enough the high strain fields around the particles can actually serve as nucleation sites for new grains due to the initially distressed state of the crystal structure. Once nucleation has occurred and grain growth begins all particles can impede grain boundary movement, but again fine, evenly dispersed particles are most effective[10]. In the current alloy a combination of these effects may be at play; regions with large particles or stringers may undergo recrystallization at temperatures far lower than those regions with a fine dispersion.

The accepted recrystallization temperature of ODS Mo-La is around 1800°C and while the recrystallized grains are larger, they maintain a high aspect ratio ranging from 10:1 to 100:1. This recrystallization behavior is termed anisotropic recrystallization, with one preferred direction for grain growth.
This preferential growth can be caused by a barrier to grain growth in one or more directions or by release of strain energy in one direction. For this alloy, the former mechanism is believed act due to elongated stringers of oxide particles present along the rolling direction [10]. Even after recrystallization at 1800°C, shadows of the original grain boundaries still exist, pinned with by the oxide particles. Annealing at 2000°C was required to completely develop an elongated interlocking structure of fully recrystallized grains [18].

1.4 Mechanical Properties

The development of ODS Mo-La products has been driven by the desire to improve high temperature mechanical properties. Significant effort has been invested in evaluating the material performance in several conditions and over a range of temperatures. Early work focused on extruded rods and drawn wires of the ODS material with studies of high temperature tensile and creep behavior.

Only recently has there been an effort to understand the low temperature mechanical properties of sheet and plate products which can have significantly different microstructures given similar levels of mechanical work. Research in these temperature and geometry spaces has been limited to material in the as-rolled and annealed condition without additional heat treatment. While often unimportant for the final material applications, these properties are important during component fabrication.

In the following discussion mechanical property data from this published work is presented as background for the current research. For these purposes, low temperature is used to describe properties below 1000°C while high temperature denotes properties above 1000°C.
1.4.1 Low Temperature

Like many other body centered cubic metals, molybdenum undergoes a ductile to brittle transition. Depending on the condition of the material and test orientation with respect to the rolling direction this temperature can range from \(-150^\circ\) to \(200^\circ\) \[12\][10].

The work most relevant to the current investigation was performed on La-ODS sheet 0.250" thick produced in a similar manner to the as-received material used in this investigation. This material has been tested over a range of temperatures from \(-150^\circ\) to \(1000^\circ\) in a combination of tension, compact tension, and notched bending tests to determine the range of expected tensile properties, fracture toughness and mode, and DBTT. Tensile properties decrease steadily with increasing temperature over this range. Near room temperature, between the reported DBTT for longitudinal and transverse orientations, the yield strength is above 700MPa with the transverse orientation having slightly higher strength. As the temperature is increased over the DBTT for the transverse orientation the properties for both orientations were similar for each test temperature[19]. Based on compact tension results at temperatures greater than the DBTT toughness values of 68 to 205MPa√\(m\) and 53 to 139MPa√\(m\) for longitudinal and transverse orientations, respectively, have been recorded[20].

Much of the early work on this alloy involved use of drawn wires rather than rolled sheet or other wrought products. There was not a great understanding of the fracture mode acting in ODS molybdenum and how this mode compares to other molybdenum alloys. Cockeram and Chan have studied this behavior in depth using compact tension samples and ultrasonic evaluation between application of incrementally increasing loads to study the fracture behavior. They found that all the low carbon arc cast (LCAC), TZM, and ODS alloy sheet fractured with some degree of delamination along grain boundaries. The
Longitudinal
Yield Stress
(MPa)
Longitudinal
Modulus
(GPa)
Transverse
Yield Stress
(MPa)
Transverse
Modulus
(GPa)

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<th>TZM</th>
<th>ODS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>253</td>
<td>489</td>
<td>307</td>
</tr>
<tr>
<td></td>
<td>187</td>
<td>223</td>
<td>187</td>
</tr>
<tr>
<td></td>
<td>293</td>
<td>550</td>
<td>288</td>
</tr>
<tr>
<td></td>
<td>214</td>
<td>223</td>
<td>156</td>
</tr>
</tbody>
</table>

Table 1.1: Tensile properties of three molybdenum alloys at 1000°C.[1]

LCAC and TZM exhibiting some combination of grain boundary and cleavage fracture while the ODS material failed almost exclusively by grain boundary delamination. This difference is attributed to the thinner ODS grains compared to the other alloys. The ligaments formed through this process experience ductility resulting in improved toughness[21][12].

1.4.2 High Temperature

Analysis of the high temperature mechanical properties of lanthanum oxide doped ODS molybdenum has been performed by measuring tensile and creep behavior. Similar analysis has been performed on nominally pure LCAC, TZM, and Mo-Re alloys. Cockeram reports values for the first three alloys over a wide range of temperatures. Table 1.1 includes a summary of his results for tensile tests for these alloys at 1000°C, which is near the 0.4T_m cutoff after which creep behavior becomes more important [1]. Most notable in these values is the observation that the ODS material is the only alloy with higher yield strength in the longitudinal direction than the transverse.

Tests have shown that ODS material has much better creep-rupture properties than powder metallurgy or arc cast molybdenum, Mo-50Re, and powder metallurgy Re alloys. Mueller reports creep-rupture times for ODS Mo that
are four to five orders of magnitude over pure Mo alloys in a similar metallurgical condition for a range of temperatures from 1200°C to 1700°C[6]. Bianco reports similar improvements over arc cast molybdenum as well as noticeable improvements over other refractory alloys known for their high creep strength. These results are shown in Figure 1-1.

![Figure 1-1: Creep rupture properties for several refractory alloys.][14]

1.5 Applications of High Temperature Molybdenum Alloys

There are many practical applications for high temperature materials. The development of ODS molybdenum alloys has extended these applications by combining advantages of molybdenum including strength, cost, and ease of manufacture with higher working temperatures. Applications of these alloys include support structures and functional components of industrial process heating equipment, materials for the next generation of nuclear powder systems, jet and rocket engine components, and sintering boats for the production of nuclear fuel products.
Chapter 2

RESEARCH OBJECTIVES

Oxide dispersion strengthened molybdenum alloys provide improved high temperature mechanical properties and creep resistance compared to pure molybdenum and molybdenum-rhenium alloys. During the development of $\text{La}_2\text{O}_3$ dispersion strengthened molybdenum rolled product, emphasis was placed on maximizing high temperature creep strength. While good high temperature properties are important for the alloy’s final application, the low temperature mechanical properties must also be considered because fabrication often occurs in this temperature range.

In the as-rolled condition this alloy exhibits brittle properties at low and moderately elevated temperatures ($25^\circ\text{C}$ to $400^\circ\text{C}$) used during fabrication of products and components from rolled sheet. The stress relief annealing process used to improve ductility and formability of other molybdenum alloys is not effective in improving the ductility of this alloys for forming. It is thought that the alloy’s improved high temperature stability reduces the effect of the stress relief heat treatment.

The objective of this research was to develop a heat treatment process that improves the ductility of $\text{La}_2\text{O}_3$ dispersion strengthened rolled products. The objective is also to study the effect of time and temperature on the bending
properties and to understand the changes in mechanical properties by performing microstructure analysis.

The specific goal is to develop a process that will allow $90^\circ$ bends to be made in the material without cracks or appearance of orange-peel texturing on the outside surface of the bend.
Chapter 3

PROCEDURE

3.1 Source Material

The material used in this project was provided by H.C. Starck in Euclid, OH. Ingots of the alloy were produced using a wet-doped powder metallurgy process, yielding a uniform distribution of approximately two volume percent La$_2$O$_3$ particles in a molybdenum matrix. Ingots were straight rolled to a nominal thickness of 0.100” following standard rolling and annealing procedures described by Cockeram[19]. After rolling, material was subject to a stress relief anneal at 950°C for one hour.

Sample coupons of size 1” by 2” by 0.100”, illustrated in Figure 3-2, were cut from rolled sheets using electrical discharge machining (EDM). Samples were taken from areas away from the edges and ends of the sheets to best represent the bulk material. Coupons were cut to capture longitudinal and transverse grain structures for mechanical testing as defined in Figure 3-3.
3.2 Heat Treatment

The heat treatment matrix was designed to represent a range of time and temperature and was based on standard practice and published data presented in Section 1.3. Table 3.2 summarizes the time/temperature combinations used to prepare the alloy for testing.

The as-rolled condition, which has undergone the 1 hour, 950°C anneal described previously, served as the basis for all heat treatments. The lowest temperature, 1500°C, was selected to represent a temperature just above the recrystallization temperature for commercially pure molybdenum alloys. Since the ODS material is not sufficiently stress relived using the pure molybdenum heat treatment it is expected that this value will serve as a lower bound for the present material. The highest temperature, 1800°C, was selected because previous work by others suggests that in some conditions the recrystallization temperature for the alloy is near this value. This temperature also represents
the maximum temperature for many available heat treatment furnaces and thus a technical limit for commercial application of any heat treatment routine developed. The intermediate value, 1700°C, was selected based on data generated during the first round of tests where it was shown that an intermediate temperature between 1500°C and 1800°C would provide the optimal combination of improved ductility with minimal recrystallization.

Anneal times of two and six hours were selected to determine the time dependence of the annealing process. The CW cycle represents a preexisting process cycle at H.C Starck and was selected to provide long expose at high temperature. A limited amount of material was available in the GE condition. This heat treatment represents the combination of several thermal cycles and was considered an over-annealed condition due to significant grain growth.

All heat treatments were performed in a protective atmosphere to prevent oxidation.

### 3.3 Mechanical Testing

The mechanical properties of the material for each condition were evaluated by measuring hardness and evaluating the bending performance in a three
Table 3.3: Bend Radii Employed

<table>
<thead>
<tr>
<th>Pin Diameter</th>
<th>R/t</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.200”</td>
<td>1.000</td>
</tr>
<tr>
<td>0.175”</td>
<td>0.875</td>
</tr>
<tr>
<td>0.125”</td>
<td>0.625</td>
</tr>
</tbody>
</table>

point bend test. Tensile tests were conducted for a range of temperatures to determine the effects of heat treatment on the mechanical properties of the material.

### 3.3.1 Hardness

Hardness measurements were made on samples prepared for microstructure analysis, after polishing but before etching. Vickers microhardness measurements were taken at room temperature using 500gf and a 15 second hold time.

### 3.3.2 Bend Testing

The bend test procedure described here is based on ASTM E 290 guided bend test for material ductility[22]. Minor deviations were made to accommodate inclusion of pin radius as a variable in the experimental matrix. Three experimental variables, temperature, bend radius, and bend direction, were evaluated for each material condition with bend testing. The parameters for bend radius, determined by the diameter of the center pin, are summarized in Table 3.3. The three pin diameters chosen correspond to radius-thickness ratios (R/t) of 1.000, 0.875, and 0.625.

Samples were tested at 25°C, 300°C, and 400°C. Based on initial tests, it was known that in the as-received condition this material was brittle at room temperature. While ductile-brittle transition temperatures were reported in
the literature survey, this property depends on the condition of the material. There were no reported values for this condition, but extrapolation of available data indicated that ductile behavior could be expected at temperatures above 250°C. The 300°C test temperature was chosen to be close to the DBTT and provide a range of ductile and brittle behavior based on specific condition while the 400°C test temperature was expected to be above the DBTT for all conditions.

Samples were bent longitudinal and transverse to the rolling direction as described above. The longitudinal samples were expected to be less likely to crack than the transverse samples due to longer grains perpendicular to the bend. Based on this expectation, emphasis was placed on bending as many samples as possible in the transverse orientation for the GE condition due to limited quantity of this material available for testing.

Similar logic was applied in other cases. If early tests were successful in producing smooth bends to 90° for transverse oriented samples in a given condition, corresponding longitudinal samples were diverted for testing other parameter sets to minimize redundant information. The distribution of the available samples through to these parameter combinations is summarized in Figure 3-4. Each shaded box indicates that at least two samples were tested for the condition. In cases expected to be near the ductile-brittle transition as many as seven samples were tested to build a more complete understanding. Consideration was also given to creating several complete rows and columns so that the effect of each parameter could be isolated from other parameters during analysis.

Bend tests were performed using the MTS Model 810 computer-controlled servo-hydraulic testing machine. Each sample coupon was bent using a three-point bend fixture in the manner depicted in Figures 3-5 and 3-6. All tests were performed with a crosshead speed of 1mm/min. The resulting strain rate was
Figure 3-4: Shaded boxes indicate that at least two samples were tested for the parameter set.

... slow enough to neglect major contribution of strain rate dependent behavior. The span between the lower supports was 0.85”. Each sample was preheated to the desired test temperature, aligned in the heated fixture, and the test was started. The goal was to bend the sample to 90°. Testing was stopped before reaching this target in cases where cracking was observed during the test.

### 3.3.3 Bending Analysis

**Qualitative Results**

To evaluate progress toward meeting the ultimate objective of this work, forming smooth, 90° bends in the material, a system was developed to uniformly describe the quality of the bends formed during testing. Four broad groups, illustrated in Figure 3-7, were defined to group the bends. Each group was
Figure 3-5: Experimental setup used for performing bend tests of samples

Figure 3-6: Experimental setup used for performing bend tests of samples
Figure 3-7: Method used to classify outer bend surface as (1) fully cracked, (2) partially cracked, (3) orange peel, and (4) smooth

assigned a numeric value to assist in summarizing the results from multiple tests for each parameter set. The classifications include:

1. Fully cracked: one or more cracks span the entire width of the specimen.

2. Partially cracked: small cracks present but individual cracks do not span the width of the specimen.

3. Orange peel: no cracking but the outer surface of the bend has a rough appearance.

4. Smooth: no cracking or orange peel.

Quantitative Results

While the primary motivation for using this bend test configuration was to determine the effects of material condition and bending parameters on the quality of the formed piece, it is also possible to quantify data collected during the tests. Load displacement curves were generated for each bend test. Three-point bend tests are often used to evaluate brittle materials where fracture occurs and low angles and typically without plastic deformation. Based on standard methods applied in such cases, Equations 3.1 and 3.2 were used to
convert recorded load and displacement data into stress and strain. These values were then used to determine the modulus of elasticity and yield stress for each sample. It is understood that there is some plastic deformation under the pins even in the elastic regime and thus these results cannot be directly compared to the results of a tensile test. Regardless, these calculations provide a means to compare the results between tests for this test setup.

\[
\sigma_{flex} = \frac{3LS}{2wt^2} \tag{3.1}
\]

\[
\epsilon_{flex} = \frac{6DT}{S^2} \tag{3.2}
\]

\(\sigma_{flex} = \text{Flexural Stress}\)

\(\epsilon_{flex} = \text{Flexural Strain}\)

\(L = \text{Applied Load}\)

\(S = \text{Span Between Lower Supports}\)

\(w = \text{Sample Width}\)

\(t = \text{Sample Thickness}\)

\(D = \text{Deflection}\)

The final bend angle, illustrated in Figure 3-8, was measured for each sample. This measurement provides another perspective on the quality of the bend for a specific material and serves as a means to differentiate performance of cracked samples.
Figure 3-8: The bend angle measured for each specimen after unloading.

3.3.4 Tensile Testing

Although the heat treatment processes applied in this investigation are important to improve ductility for forming, the high temperature properties must be maintained for successful application. To address this concern, tensile tests were conducted for a range of temperatures and heat treatment conditions. These tests were performed at PMTI laboratories according to standard methods.

Material in AR, 1500-2, 1500-6, 1800-2, and 1800-6 conditions was tested at 25°C, 300°C, 450°C, 800°C, and 1000°C. Two specimens were tested for each temperature and condition combination.

3.4 Microstructure Analysis

Samples for microstructure analysis were prepared from material removed from the end of the bend specimens after heat treatment but before mechanical testing. This method was chosen to produce samples that accurately represent the condition of the material tested in the bend tests without concern that the observed structure was affected by the deformation. Three methods for analysis, optical metallography, scanning electron microscopy with XEDS chemical analysis, and transmission electron microscopy were selected to provide information about grain size and aspect ratio, oxide particle distribution,
and oxide particle morphology.

### 3.4.1 Optical Metallography

Optical metallography techniques were used to study the effect of heat treatment on the grain size and aspect ratio of the molybdenum matrix. It was expected that annealing would lead to grain growth and in the most intense heat treatments partial or complete recrystallization would occur, leading to an equiaxed grain structure compared to the rolled texture.

#### Sample Preparation

Grain size was determined for the cross sections of bending specimens as illustrated in Figure 3-9. Samples were prepared for analysis using standard mounting and polishing techniques. Specimens were then etched using a solution of 60% lactic acid, 30% nitric acid, and 10% hydrofluoric acid. Etching time ranged from 25 to 60 seconds depending on the heat treatment employed and orientation of the plane examined to create a fully exposed grain structure. After etching the sample surfaces were cleaned thoroughly with distilled water and cotton tipped swabs to remove the oxide layer formed on the surface. Micrographs were recorded for three regions of each prepared sample at 400x to capture the microstructure. For some specimens, images were also recorded at lower magnification to provide additional information about microstructure variations observed through the thickness of sheet.

#### Grain Size Measurement

Grain size measurement and calculation were performed using the linear intercept method described in ASTM E 112 §16[23]. A grid of three directed test lines in two orthogonal principle directions was applied to each micrograph,
illustrated in Figure 3-10. The same grid was used for all images to reduce bias in the measurement. In those cases where this grid did not produce at least fifty intercepts, additional equidistant lines were applied to the micrograph until this condition was met.

Since longitudinal and transverse specimens were sectioned in the same direction with respect to bend direction it was possible to integrate data from each heat treatment condition to observe four of the six principle test directions. This satisfies the condition that for non-equiaxed grains at least three principle test directions must be observed to calculate the grain size. Figure 3-11 summarizes the four directions, $L_n$, $L_t$, $T_n$, and $T_t$, analyzed. Each of these directions yielded $N$ intercepts per unit length. These values were averaged
Figure 3-11: Four principle test directions were observed for the two cross sections examined.

using Equation 3.3 to produce a mean intercept per unit length value. Because grain size was measured in two normal directions, an average, \( N_{LTn} \), was used in this calculation.

\[
\bar{N} = (N_{Ll} \cdot N_{Tt} \cdot N_{LTn})^{1/3} \tag{3.3}
\]

Anisotropy in grain size was represented by normalizing \( N_{Ll} \) and \( N_{Tt} \) by \( N_{Ln} \) and \( N_{Tn} \):

\[
\frac{N_{Ll}}{N_{Ln}} and \frac{N_{Lt}}{N_{Ln}} \tag{3.4}
\]

### 3.4.2 Scanning Electron Microscopy with EDX Analysis

Scanning electron microscopy was used to provide additional information about the material microstructure with the goal of determining the size and distribution of oxide particles within the molybdenum grains and along grain boundaries. For this analysis two conditions, AR and 1800-2, were selected to represent two ends of the heat treatment spectrum. Samples were sectioned to view cross-section and plan views. Specimens were prepared using the method
described in Section 3.4.1 except that the oxide layer formed during etching was not removed to prevent the loss of exposed lanthanum oxide particles. An FEI xT Nova Nanolab 200 was used to examine the microstructure of the material. XEDS elemental mapping was used to confirm the identity of oxide and molybdenum phases present in the material.

### 3.4.3 Transmission Electron Microscopy

TEM specimens were prepared from the same material used in SEM analysis. Thin wafers were cut using a diamond saw. These wafers were polished and cut into disks which were thinned by dimple grinding. Ion beam milling was used to perforate the disks, creating a thin foil for analysis. This method was selected to mimic the technique used in [10] to minimize the effect of external factors when comparing these results. Representative images were recorded for particles and matrix for both samples. Selected area diffraction patterns were used to verify the identity of the particles and matrix.
Chapter 4

RESULTS AND ANALYSIS

4.1 Mechanical Testing

4.1.1 Qualitative Bend Results

The results from observations made about the condition of the outside surface of each bend are presented in Figure 4-12. The general format of this chart will be used throughout this chapter to graphically represent the multiple sets of experimental conditions for easy identification of trends. The classifications used represent the average of all samples in each experimental condition based on the numerical classification system outlined in § 3.3.3. “Cracked” indicates that some degree of cracking was observed on all samples in the group, “orange peel/mixed” indicates that either rough orange peel finish was observed or a combination of smooth and cracked bends was observed. The former was typically the case for high temperature heat treatments while the latter was more common for lower temperature conditions. “Smooth” indicates that all tests in the parameter group produced smooth bends without cracks.

These results indicate that low temperature heat treatment and room temperature bending lead to cracking with a higher temperature needed to prevent
Figure 4-12: Average value for bend surface quality for each set of experimental conditions.
cracking in the transverse orientation. It is also observed that if the temperature and time of heat treatment are too high even though cracking does not occur the quality of the bends is reduced by the appearance of a rough orange peel finish due to large grains. To achieve the desired results a balance must be maintained between relieving enough cold work without causing excessive grain growth.

4.1.2 Quantitative Bend Results

The first of three quantitative measures used to evaluate material performance during bending was bend angle. Figure 4-13 represents this data in three broad categories. Conditions shown in red indicate that samples underwent little or no plastic deformation and cracked at low angles. Samples in yellow bent further before cracking, indicating ductility in at least part of the condition group. Samples reaching at least 80° were considered full bends that failed to reach 90° only because the test was inadvertently stopped prematurely. Processing conditions classified in this category are considered successful for material forming.

The bend angle results are also presented in Figures 4-14 and 4-15. In these figures data is presented as a function of temperature for each heat treatment and pin radius combination. While this data is labeled in the legend for each graph, Figure 4-16 provides a visual reference for understanding the experimental conditions represented in the graphs found in this section.

These results mirror the results of the qualitative analysis. Samples tested in the transverse orientation cracked before comparable longitudinal samples. In all but one case samples tested at room temperature cracked before reaching a full bend. All transverse samples in this group cracking at low angles. It is also noted that the bend angle for longitudinal samples tested at room temperature decreases with increasing heat treatment time and temperature.
Figure 4-13: Chart comparing bend angle for all experimental conditions.

Figure 4-14: The final average bend angle for longitudinal samples for each heat treatment and pin radius as a function of bending temperature.
Figure 4-15: The final average bend angle for transverse samples for each heat treatment and pin radius as a function of bending temperature.

Figure 4-16: This table visually represents the experimental conditions reported for all graphs in this section. Each colored bar corresponds to specific condition as a function of test temperature as represented in Figures 4-14, 4-15, 4-18, 4-19, and 4-21.
A similar but less defined trend is observed for the transverse orientation.

The 1500-2 heat treatment was effective in improving the bending properties of longitudinal samples but transverse specimens still cracked during bending. The 1500-6 and all higher temperature heat treatments allowed for full bends to be made, but there were isolated instances of cracking in several transverse specimens leading to lower averages. The one case of cracking in a heat treated longitudinal sample tested at elevated temperature is considered an anomaly and will be discussed in § 4.2.1.

Computed values for yield strength in bending are shown in Figures 4-17, 4-18, and 4-19. It is observed that the yield stress decreases with increasing heat treatment and test temperature with longitudinal samples having slightly higher values than the transverse samples.

The decrease in yield strength with increasing test temperature appears to occur primarily between room temperature and 300°C with little change between 300°C and 400°C. The change resulting from heat treatment does not appear to be influenced by the test temperature with the difference between any two treatment conditions remaining roughly constant across all test temperatures.

The final quantitative measure used to analyze the bend test results was the calculation of an elastic modulus from the bending load displacement curves. These results are shown in Figures 4-20, 4-21, and 4-22. It is observed that the modulus decreases with increased test temperature. In general specimens heat treated at higher temperatures had lower modulus. It is useful, however, to consider the pattern shown in Figure 4-20 in addition to the trends in Figures 4-21 and 4-22. Here it is clear that the lowest modulus values were actually observed in the samples heat treated for intermediate time and temperature and tested at elevated temperature.
### Figure 4-17: Chart comparing yield strength for all experimental conditions.

<table>
<thead>
<tr>
<th>Condition</th>
<th>25</th>
<th>300</th>
<th>400</th>
<th>25</th>
<th>300</th>
<th>400</th>
</tr>
</thead>
<tbody>
<tr>
<td>As rolled</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
</tr>
<tr>
<td>1500 - 2</td>
<td>0.200</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
</tr>
<tr>
<td>1500 - 6</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
</tr>
<tr>
<td>1700 - 2</td>
<td>0.200</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
</tr>
<tr>
<td>1800 - 2</td>
<td>0.200</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
</tr>
<tr>
<td>1800 - 6</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
</tr>
<tr>
<td>1800 - CW</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
</tr>
<tr>
<td>GE</td>
<td>0.200</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
<td>0.126</td>
<td>0.175</td>
</tr>
</tbody>
</table>

Legend:
- Yellow: Less than 400 MPa
- Orange: 400 MPa to 800 MPa
- Red: Greater than 800 MPa

### Figure 4-18: The final average strength for longitudinal samples for each heat treatment and pin radius as a function of bending temperature.

0.2% Offset Yield Stress (MPa) vs. Test Temperature (°C)
Figure 4-19: The average yield strength for transverse samples for each heat treatment and pin radius as a function of bending temperature.

Figure 4-20: Chart comparing calculated flexural modulus for all experimental conditions.
There are two possible explanations for this behavior. The first explanation assumes that all starting material had the same general behavior during heat treatment. In the AR and 1500°C conditions there are sufficient dislocations present to increase material stiffness. As heat treatment temperature is increased and some of these dislocations are relieved the material softens. The recrystallization present at higher temperature and time increases the stiffness slightly, especially in the transverse direction. The second explanation is based on the observation of differences in the grain growth for some 1500-2 and 1800-2 and all 1700-2 material tested. This difference, thought to result from the prior processing, could also impact the bending modulus.

### 4.1.3 Hardness

Figure 4-23 presents the results of hardness tests performed on samples from each heat treatment condition. The material softens with increasing thermal treatment, supporting the decrease in yield strength discussed above. It is also
Figure 4-22: The average elastic modulus for transverse samples for each heat treatment and pin radius as a function of bending temperature.

noted that the hardness is around 200HV for all 1800°C treatments lasting 6 hours and longer indicating that the structure may reach a stable condition after long exposure at a given temperature.

### 4.1.4 Tensile Testing

Results from tensile tests are reported in Figures 4-24, 4-25, and 4-26. Several trends are observed in the results for yield and ultimate tensile strength. Considering first the effect of heat treatment, at low temperature there are large differences between values for each heat treatment temperature. There is little difference observed when the heat treatment time was increased from two hours to six hours for both temperatures.

Considering the effect of test temperature, the strength of samples in all conditions decreased with increasing temperature. At higher test temperatures, in the application range for this alloy, the strength of the AR material drops faster than the heat treated samples. For this study this is a positive
result indicating that while the heat treatment is effective in improving the ductility of the material, the impact on possible application of the heat treated material may not be significant. While differences exist in specific values between the two test directions, similar trends are observed in both cases.

Analysis of the elongation data reveals several interesting trends that reinforce observations made by previous researchers and data collected in other parts of this project. As expected, the transverse AR material has limited, consistent ductility over the range of test temperatures. This trend is also observed in the 1500°C transverse samples with slightly higher but still constant ductility over the range of test temperatures.

In the longitudinal direction the AR material is most ductile at low temperatures with the ductility dropping as the test temperature is increased. The 1500°C samples are also most ductile at low temperatures but the drop is not
Figure 4-24: Tensile yield strength for each heat treatment condition as a function of test temperature.
Figure 4-25: Ultimate tensile strength for each heat treatment condition as a function of test temperature.
Figure 4-26: Percent elongation for each heat treatment condition as a function of test temperature.
as large as the AR condition.

The ductility in both directions of the 1800°C conditions is lowest for room temperature tests, increasing significantly with increased test temperature. This result supports the bend test results that showed cracking in these samples at room temperature but successful bending at higher temperatures.

4.2 Microstructure Analysis

4.2.1 Optical Metallography

Investigation of the microstructure through optical metallography yielded many of the expected results. The observed grain size increased with increasing heat treatment time and temperature. There were, however, several unexpected results and complications in the analysis.

First, etching the material in the AR condition did not produce optically identifiable grains due to small grain size. One transverse cross section sample prepared using an etchant with 15% HF, an increase of 5%, produced a measurable microstructure and was imaged using SEM for grain size measurement.

Second, one group of samples, including all of the 1700-2 heat treatment condition, came from a second lot of material due to a limited amount of material available in the first and primary lot tested. Many specimens in this group had abnormal microstructures, with varied grain size and aspect ratio through the thickness of the sample.

The results from the bulk of the samples analyzed had a typical microstructure, were systematically analyzed, and the results are presented in the next section. The special cases encountered are discussed in detail in the following section.
Figure 4-27: Representative (a) longitudinal and (b) transverse optical micrographs showing grains. Both samples received CW heat treatment. The rolling direction is labeled in blue.

**Typical Microstructure**

Representative microstructures for longitudinal and transverse cross sections are shown in Figure 4-27. The structure for the longitudinal cross section shows highly elongated grains along the rolling direction. The grains in the transverse cross section are elongated perpendicular to the rolling direction and parallel to the sheet surface, but with a lower aspect ratio than in the longitudinal direction.

Table 4.4 summarizes the results of grain size measurement. The mean normal intercept lengths for each heat treatment condition are also compared in Figure 4-28. It is observed that the grain size increases with heat treatment time and temperature such that the most heavily heat treated samples had grains roughly twice the size of the 1500-2 condition. The grains in the AR material were even smaller but due to differences in sample preparation and imaging technique they cannot be directly compared with the rest of the results. The longitudinal microstructure of the samples that experiences more severe heat treatment were so elongated that it was not possible to identify fifty grid-boundary intercepts for standard analysis so a smaller number of
Condition | $L_n(\mu m)$ | $L_l(\mu m)$ | $T_n(\mu m)$ | $T_l(\mu m)$ | $\bar{N}(\mu m)$ | $\frac{N_{Ll}}{N_{Ln}}$ | $\frac{N_{Lt}}{N_{Ln}}$
--- | --- | --- | --- | --- | --- | --- | ---
AR | | | 87 | 21 | | 4.1 |
1500-2 | 816 | 90 | 324 | 64 | 273 | 9.1 | 5.1
1500-6 | 1197 | 94 | 600 | 72 | 391 | 12.8 | 8.3
1800-2 | 1338* | 79 | 633 | 114 | 434 | 5.6 | 9.6
1800-6 | 1637* | 93 | 493 | 91 | 420 | 16.9 | 5.6
CW | 1897* | 75 | 567 | 109 | 462 | 25.2 | 5.2
GE | 2297* | 84 | | | | 27.4 |

* Due to length of grains a limited number of boundaries were observed.

Table 4.4: Summary of grain size measurements made for heat treated conditions

While an increase in grain size with heat treatment was expected, comparison of the grain aspect ratios for longitudinal and transverse orientations yields an interesting result, illustrated in Figure 4-29. The longitudinal aspect ratio increased by approximately a factor of three over the range of conditions compared while the transverse cross section aspect ratio remained relatively unchanged across all conditions. This indicates that while the grain growth is occurring in all directions, the growth is fastest along the rolling direction.

These observations can be rationalized by returning to an understanding of the role of the oxide particles in structure development. During the rolling process, particles are elongated along the rolling direction resulting in stringers of fine particles. This results in a very fine microstructure in the AR condition leading to difficulty when preparing samples for optical analysis. During heat treatment the particles preferentially limit grain growth along these stringers.
Figure 4-28: Average grain size for each heat treatment condition. Error bars represent the standard deviation in mean intercept length for each direction measured, propagated through the calculation of $N$.

Figure 4-29: Aspect ratio for grains along longitudinal and transverse cross sections.

perpendicular to the rolling direction resulting in fast longitudinal growth and slower, uniform growth in the transverse and normal directions.
Abnormal Microstructure

Two types of abnormal grain structure were observed in the specimens examined. The first is a non-uniform development of the typical annealed structure most pronounced in the low temperature, short duration of the 1500-2 heat treatment. A representative photomicrograph illustrating this structure is shown in Figure 4-30. Here the top half of the micrograph shows large, pronounced grains of the annealed structure while the bottom half shows a finer, less well defined structure observed in the AR samples. This structure indicates that the transformation does not occur simultaneously throughout the material thickness. The cause of this nonuniform structure could lie simply in the structure resulting from the rolling process or a more complicated combination of matrix structure and particle distribution.

This structure supports the observation that there were greater differences observed for several properties between the 1500-2 and 1500-6 conditions than between the 1800-2 and 1800-6 conditions. The transformation occurs faster at higher temperatures so that two hours at 1800°C is enough to cause complete transformation, while in the lower temperature 1500°C heat treatment a time longer than 2 hours must be used to achieve complete transformation.

The second type of unexpected microstructural feature occurred near the surface of samples from a second, smaller lot of material initially included in the matrix to validate results from early tests and allow additional experimental variables. The etched cross sections from these samples displayed rapid, abnormal grain growth along both surfaces of the material with a typical microstructure in the bulk of the material, shown in Figure 4-31. The region near the surface of the sheet shows small, equiaxed grains and the thickness of this abnormal band increases with increasing heat treatment temperature.

One sample from this lot cracked unexpectedly during bending. Based on results before this test it was expected that this sample, having undergone
Figure 4-30: In low temperature heat treatments only select regions of some samples transformed to show grains indicative of heat treatment. This sample is from the 1500-2 group.

the 1800-2 heat treatment, would produce a smooth bend. Since the behavior of this sample was uncharacteristic, additional analysis was performed. Figure 4-32 shows the complete fracture surface for this sample. Two regions, labeled A and B, were selected for analysis at higher magnification, shown in Figure 4-33. This figure indicates that near the surface intergranular fracture dominates while in the bulk of the material transgranular fracture is evident. Regardless of cause, it appears that the formation of these surface grains negatively impacts the bending process.

4.2.2 Scanning Electron Microscopy with EDX

Representative images for both orientations and conditions are shown in Figure 4-34. In these images the elemental map indicating the location of lanthanum in the structure in red is overlaid on a secondary electron SEM image of the same region. White particles visible in the SE image correspond to the strongest lanthanum signal and are taken to be lanthanum oxide particles
Figure 4-31: A small number of samples, from a second lot of material, developed an abnormal structure near the surface of the sheets.
Figure 4-32: SEM image of fracture surface for material exhibiting abnormal surface structure. Higher magnification of areas A and B is found in Figure 4-33.

Figure 4-33: Higher magnification of areas A and B noted in Figure 4-32 indicating intergranular fracture near surface.
exposed during etching.

There are also regions with a weaker lanthanum signal that mimic the size and shape of the particles but without corresponding particles visible in the SE image. This observation is explained by considering the relative sampling depth of the two imaging techniques. Secondary electrons are have only enough energy to escape from a thin surface layer several nanometers thick while the primary electrons penetrate much deeper resulted in x-ray generation from a greater volume of material. Using Equations 4.5 and 4.6 it was determined that primary electrons penetrate $2.55\mu m$ and x-rays can escape from a depth of $0.95\mu m$. This means that oxide particles below the surface of the material
Figure 4-35: This magnified view of the AR plan view shows particle shape and size in greater detail.

can still show up in the elemental map.

\[
D_{\text{primary}} = \frac{0.0276 A_w E_0^n}{Z^{0.889} \rho} \\
D_{\text{x-ray}} = \frac{0.064(E_0^n - E_C^n)}{\rho}
\]  

(4.5)  

(4.6)

\[A_w = \text{AtomicWeight} \]

\[Z = \text{AtomicNumber} \]

\[E_0 = \text{IncidentEnergy} \]

\[\rho = \text{density} \]

\[E_C = \text{CharacteristicEnergy} \]

\[n = 1.67 \]
On initial inspection differences between the cross section and plan views are noted. Both cross sections contain a relatively even distribution of particles irrespective of the location of grain boundaries while the plan view images show lines of particles aligned along the rolling direction.

Further analysis of the plan view images reveals that in the AR condition the lanthana phase exists in extended bands and rods, magnified in Figure 4-35, while after heat treatment this dispersed phase exists in discrete particles closely spaced in these long bands. This observation supports the previous discussion about the grain growth patterns. Long stringers of oxide particles exist along the rolling direction limiting grain growth perpendicular to the rolling directions in which the moving ground boundary would move though these particle strings.

4.2.3 Transmission Electron Microscopy

Figures 4-36 and 4-37 show representative microstructural images recorded during the TEM analysis of material in AR and 1800-2 conditions. The circles in both images denote the location of observed particles. In all cases the particles are sub-micrometer in size, consistent with results reported in literature for rolled and drawn ODS material.

The appearance of the particles in the AR sample is not clearly defined but the particles appear to have a rough texture. At the particle-matrix interface the matrix is similar in appearance to the bulk of the grain containing the particle.

Several differences are observed in the 1800-2 condition. First, the shape of the particles is better defined, appearing faceted and symmetric. The two particles in the circle labeled ‘A’ are cubic in appearance while the particles in areas ‘B’ and ‘C’ appear faceted. A significant difference is also observed in the appearance of the particle-matrix interface, shown most clearly in the
Figure 4-36: TEM image showing the structure of molybdenum matrix and oxide particles for the AR condition.

Figure 4-37: TEM image showing the structure of molybdenum matrix and oxide particles for the 1800-2 condition.
Figure 4-38: This diffraction pattern from the molybdenum matrix shows many discrete but closely spaced diffraction spots for each reflection.

high magnification image on the right in Figure 4-37. Here an elliptical region appears around the particle, lighter than the surrounding matrix. A dark ring is also observed around the particle separate from this ellipse. These features could point to the voids noted in some alloys by Bianco and Buckman[10]. This could be the result of a change in microstructure to accommodate the particles and leading to a change in the diffracted beam intensity near the particles.

Figure 4-38 shows a selected area diffraction pattern recorded for the molybdenum matrix in the 1800-2 condition. Highlighted in this figure is the apparent shifting of the diffraction spots with multiple spots composing each reflection. This result is similar to the that reported by Wright indicating the presence of subgrains with slight misorientations[13].
Chapter 5

CONCLUSIONS

A range of time-temperature regimes have been investigated to improve the formability of a La$_2$O$_3$ dispersion strengthened molybdenum alloy. Heat treatment at 1500°C for two and six hours improved ductility but still lead to cracking in some transverse specimens. Treatment at higher temperatures eliminated this cracking but treatment at 1800°C for more than two hours resulted in sufficient grain growth to cause visible roughness on the bend surface. The best bending results were obtained from 1700-2 and 1800-2 heat treatments. This research has met the objective of developing a post-processing heat treatment routine to improve the ductility of this material.

In addition to conclusions related directly to the research objective, the following conclusions were reached about material behavior during heat treatment:

- Heat treatment causes a reduction in the yield strength and ultimate tensile strength. For the conditions tested the treatment time at a given temperature did not seem to matter with two and six hour treatments producing similar tensile strength results.

- The elongation of material treated at temperatures less that 1800°C
decreased with increasing test temperatures. Samples treated at 1800°C had improved ductility and the ductility increased with increasing test temperature.

- The strength of the material at room temperature is significantly lower in a heat treated condition. This difference in strength is reduced as the test temperature is increased toward the application temperature for the alloy.

- Stress relief and grain growth do not occur uniformly throughout the material. The 1500-2 treatment was not sufficient to create an annealed structure through the thickness of the sheet.

- Grain growth occurs in all directions during heat treatment, but the growth happens fastest along the rolling direction.

- Heat treatment causes elongated oxide ribbons and rods found in the as-rolled material to separate into many smaller particles distributed as stringers with the same orientation.

- TEM results reveal that the nature of the particle and matrix interface changes with heat treatment. In the as-rolled condition the matrix is uniform until the interface while in the heat treated material elliptical regions with different apparent structure are observed around the particles.
REFERENCES


