XRD PEAK BROADENING EFFECTS IN U-Mo $\alpha''$ PHASE

E. Dabush, J. Sariel, I. Dahan and G. Kimmel

Nuclear Research Center, Negev, P.O. Box 9001, Beer-Sheva, 84190 Israel

ABSTRACT

The U-Mo system has been extensively studied due to its use in the past as a nuclear fuel. This system has several metastable phases, depending on the Mo content and the cooling rate after solution treatment at high temperature. One of these phases $\alpha''$ is characterized by its high strength and ductility. X-ray diffraction of mechanically polished specimens of U-Mo exhibits broadened lines associated with the surface grinding. Broadened diffraction lines are unfavorable for structural analysis. Yet, the broadening effect indicates a valuable information about the microstructure. It was found that the broadening effect is sensitive to the cooling rate and that the broadening as a function of the cooling rate has an opposite trend for mechanically and electrolytically polished. The presentation of this phenomenon together with its implications is given in this work.

INTRODUCTION

In equilibrium, above 650°C, U-Mo system has a cubic structure (cI2) denoted as $\gamma$ phase, which is a solid solution in the range of 0-40% at. Mo. Below 650°C there is a separation into two phases: U-$\alpha$ which dissolve less than 0.1%at. Mo and an ordered MoU$_2$ - $\gamma$s intermediate phase. However, it is easy to obtain single phases at room temperature in a wide solubility range by rapid cooling from the high temperature solid solution to the room temperature. These phases are metastable with various crystal structures depending on the Mo content and cooling rate.

In this work we are concentrating on the monoclinic $\alpha''$ which has a deformed U-$\alpha$ structure. The $\alpha''$ phase is obtained by rapid cooling from temperature of 800-900°C in the composition rang of 6-12 at. % Mo. Due to the small penetration of X-rays in uranium base alloys it is necessary to conduct electrolytic polishing in order to expose the bulk structure. In order to inspect the effectiveness of the electrolytic polishing procedure we decided to examine X-ray diffractograms from specimens underwent mechanical polish (MP) with those from specimens underwent electrolytic polish (EP).

The structure of $\alpha''$ phase is closely related to U-$\alpha$ orthorhombic. The four atoms in the unit cell as has been given by Stewart and Williams [1], can be generated in an artificial space group C2/m [2] from the asymmetric unit which include one atom. The natural space group should be P2$_1$/m with two atoms in the unit cell. Since some programs cannot handle unusual settings, a conversion between both settings for U 9%at. Mo is shown in Table 1.
This document was presented at the Denver X-ray Conference (DXC) on Applications of X-ray Analysis.

Sponsored by the International Centre for Diffraction Data (ICDD).

This document is provided by ICDD in cooperation with the authors and presenters of the DXC for the express purpose of educating the scientific community.

*All copyrights for the document are retained by ICDD.*

Usage is restricted for the purposes of education and scientific research.

**DXC Website**
- [www.dxcicdd.com](http://www.dxcicdd.com)

**ICDD Website**
- [www.icdd.com](http://www.icdd.com)
Table 1: Crystal structure of the monoclinic $\alpha''$ in two settings

<table>
<thead>
<tr>
<th></th>
<th>U $\alpha$ like</th>
<th>Standard setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>space group</td>
<td>C2$_1$/m</td>
<td>P2$_1$/m</td>
</tr>
<tr>
<td>Axis</td>
<td>c</td>
<td>b</td>
</tr>
<tr>
<td>cell parameters</td>
<td></td>
<td></td>
</tr>
<tr>
<td>a</td>
<td>0.2899 [nm]</td>
<td>0.3278 [nm]</td>
</tr>
<tr>
<td>b</td>
<td>0.5781 [nm]</td>
<td>0.4977 [nm]</td>
</tr>
<tr>
<td>c</td>
<td>0.4977 [nm]</td>
<td>0.3185 [nm]</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>90°</td>
<td>90°</td>
</tr>
<tr>
<td>$\beta$</td>
<td>90°</td>
<td>126.9°</td>
</tr>
<tr>
<td>$\gamma$</td>
<td>92.1°</td>
<td>90°</td>
</tr>
</tbody>
</table>

Atomic positions

<table>
<thead>
<tr>
<th>Site</th>
<th>x</th>
<th>y</th>
<th>z</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>0.46</td>
<td>0.61</td>
<td>$\frac{1}{4}$</td>
</tr>
<tr>
<td>2</td>
<td>0.855</td>
<td>0.069</td>
<td></td>
</tr>
</tbody>
</table>

EXPERIMENTAL

Seven solid specimens, in the form of disks ($\Phi$25X5 mm) of uranium with 6 to 12 % at. molybdenum were prepared by arc melting in argon atmosphere. The specimens were polished mechanically and electrolytically for metallography and XRD characterization. All the specimens were cut into four parts each. One part used for the comparison between EP and MP. They were treated at 900°C for 8 hours for homogenization and slowly cooled to room temperature. At the next stage the specimens were reheated at 800°C for 20 min and then were quenched into cold media: water, oil and sand and also cooled in flowing cold helium. Each specimen was mechanically polished, examined by XRD and then electrolytically polished and examined again by XRD.

The X-ray system was a Bragg-Brentano diffractometer with copper anode and graphite monochromator.

RESULTS

Crystal structure of as-cast specimens:
The specimens within the composition range from 6 to 9 %at. Mo were identified as a single phase, ($\alpha''$ monoclinic). Those with higher Mo concentration found to be a mixture of a $\alpha''$ monoclinic phase (major) with another phase (minor) with a gamma-like structure. These results agree with the reported solubility range of Mo in U $\alpha''$ structure [3]. Figure 1 presents typical diffractograms of specimen with 9% and 12 at.% Mo. The diffractogram from the specimen with 9 at. % Mo shows pure monoclinic $\alpha''$ phase. The gamma-like phase was indexed as a cubic bcc (c12).
Fig 1. Diffractogram from as-cast specimens. The marked peaks belong to the gamma phase, which was indexed as cubic. All unmarked peaks belong to the monoclinic $\alpha''$ phase.

The specimens were almost randomly oriented. The unit cells and the atomic positions were refined by Rietveld method [4]. An example of Rietveld plot for as-cast U-9%at. Mo specimen is shown in Fig. 2.

Fig. 2: Rietveld plot for as-cast specimen with 9%at. Mo, after electrolytic polish

The cell parameters of the monoclinic phase varied with the Mo concentration. Yet, only two parameters b and gamma showed a significant modification (see Figs. 3a and 3b respectively). The results in this work are in good agreement with previously reported [5].

Fig. 2: Rietveld plot for as-cast specimen with 9%at. Mo, after electrolytic polish
ROADENING EFFECTS

As can be seen in Fig. 4 there is a severe line broadening effect in mechanically polished specimen in the monoclinic phase after dissolution in gamma phase followed by water quenching. Electrolytic polishing found to be effective for removing the surface cold worked layer, resulting in much less broadened diffraction peaks. Yet, the peaks are still broadened relatively to the instrumental broadening.
In metastable solid solutions broadened X-ray diffractograms are expected due to non-uniform distribution of the solute. The clustering phenomenon is attributed to early stages of precipitation. This process is depressed by rapid cooling. Therefore, higher broadening effect is expected in case of lower cooling rates.

Examining several specimens it was found that after mechanical polishing the broadening effect was lower in specimens with lower cooling rates. For example, in Fig. 5 it is shown that with mechanical polishing after cooling at sand (low cooling rate), the diffraction lines are sharper than after water quenching. This can be explained by considering the contribution of the cold work to the broadening effect. Higher cooling rate leads to more homogeneous material, which is also softer. Mechanical polishing of softer material brings upon greater amount of cold work. Normally in solution heat treated material, the higher the cooling rate is, the sharper the peaks obtained. In our case, because of the greater amount of the surface cold work while mechanically polished, the higher the cooling rate is the higher the broadening effect obtained.
Fig 5. Comparison between water quenched and sand cooled (-low cooling rate) specimens, both mechanically polished (MP).

REFERENCES


