GPLUS Final Report for Osram Sylvania[1]

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Problem

Metallic crystals formed during the fabrication of quartz tubes are currently the major limitation on the lifetime of the refractory metal quartz melter. When metallic crystals come in contact with the extruded quartz tube, they alter the extruded tubing and the melter must ultimately be shut down, disposed of and a new one constructed. The metallic crystals nucleate and grow just below the orifice, inside the cooling area where the molten quartz is formed into a continuous quartz tube. The nucleation site within the cooling chamber, the growth direction and rate of crystal growth determine when they will intercept the quartz tubing and consequently lead to halting production. Thus this study is to investigate the nucleation, growth and source of the metallic crystals in hopes of determining methods for controlling crystal growth and extending the lifetime of the quartz melter.

Experimental

Two ORNL staff joined Osram-Sylvania staff at the Exeter, NH facility to refine a test plan and identify samples to be studied. Samples from two types of quartz melters, types 1 and 2, were subsequently shipped to ORNL for examination. Samples from type 1 & 2 quartz melters included: two type 1 and two type 2 molybdenum orifices, one type 1 and one type 2 skirt samples, a piece of container tube from type 1 and samples of fabricated glass tubing. Inside the molybdenum orifices were quartz remnants solidified during quartz melter shutdown.

Experimental Methods

Samples were examined using various techniques: X-ray diffraction (XRD), optical microscopy and the scanning electron microscope (SEM). XRD examinations were carried out on a Scintag PAD V powder diffractometer with Cu Kα radiation ($\lambda_{K\alpha_1}$ = 1.5406 Å). Specimens cut from both the type 1 and type 2 samples were mounted in epoxy for examination using optical microscopy and

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SEM. SEM examinations were carried out on a Hitachi S4100 with computer interfaced energy dispersive spectrometer, digital imaging, and image analysis capabilities. Simple thermodynamic calculations were undertaken using the Chem Sage program package to help determine possible chemical sources for crystal formation and growth.

Results

Results of XRD, optical and SEM examinations on individual specimens are reported by location within either the type 1 or type 2 quartz melter. Thermodynamic calculations are reported in a separate section.

Crystals from type 1 & 2 quartz melter

Visual observations indicated that crystals formed on the bottom of the molybdenum orifice and on the inside of the molybdenum or tungsten skirt of the type 1 and 2 samples, respectively. The size of the crystal formations were considerably larger for the type 1 than for the type 2 quartz melter. XRD examination of ground crystals from the type 1 and 2 orifice identified them as molybdenum. In addition a minor amount of a $Mo_2O_5(OH)$ phase was present along with the molybdenum crystals on the bottom of the type 2 orifice.

A representative prismatic molybdenum crystal from the type 1 orifice was mounted on an adjustable goniometer head and placed on a four circle diffractometer with the long axis roughly perpendicular to the x-ray beam (fig. 1). During a $\theta / 2\theta$ scan, only the (222) reflection was seen to diffract. This face was aligned so that the maximum peak intensity was located at Chi and Phi values of 0. Pole figures of the (100) and (110) reflections were then collected and the intensities observed corresponded to angles consistent with a single molybdenum crystal and confirmed that the growth direction of the crystals was in the (111) direction.

Type 1 #2 Orifice

The type 1 orifice #2 was first cut in half. A wedge section was subsequently cut off and four representative samples were cut from different sites on the type 1 orifice #2. The samples were mounted in epoxy so that the cross section of the orifice could be examined.

Site 1, was cut from the lip of the orifice and included the crystals attached to the bottom of surface. In micrographs of this sample, these crystals appeared to have initiated growth at individual grains and then proceeded to grow over adjoining grains on the bottom surface (fig. 2). Crystal growth with the same orientation continued and covered adjoining grains.

Site 2 was cut off the top of the orifice and is at the juncture of the orifice and the container lining. Both the outer wall which is radiant heated by the heating coils

and the inner wall which is exposed to molten quartz are included in the examined cross section. Grain size appears to increase towards the surface exposed to the molten quartz (fig. 3). Changes in grain size could be due to fabrication or recrystallization due to temperature gradients or exposure to molten quartz.

Site 3 is a cross section from the outer wall to 1/2 inch within the orifice. While there was a wide range in grain size through out the sample, there appeared to be no systematic variations (fig. 4).

Site 4 was located at the outer wall at the bottom of the orifice and included the attachment of the skirt to the orifice (fig. 5). Crystals growing on the bottom surface of the orifice were included in the sample. Again, crystal formation was observed to be initiated at single grains and preferentially grew in one direction.

Crystals from sites 1 and 4 were analyzed for elemental content using the SEM (fig. 6-7). Molybdenum, oxygen and trace amounts of carbon, from the epoxy mounting, were the only elements present.

Type 1 Container Tube

On visual examination of the inner surface of the type 1 container there appeared to be dimples of glass. Five samples were cut from the melt zone of the container tube and mounted so that the cross section of the container from the outer to the inner surface could be examined. Upon etching the sample surface, it was observed that etch resistant material was present on the inner surface (fig. 8). Optical microscopy examination indicated that grain size varied within the samples, but there were indications that grain size increased near the molten quartz surface (fig. 9).

The interior of a piece of the melt zone sample was mounted on a X-ray diffractometer and the x-ray beam was centered on a glass dimple. XRD examination identified diffracted peaks as glass, Mo and Mo_3Si, a molybdenum silicide (fig. 10). Similar dimples of glass also appeared on the interior of the type 1 orifice. Since site 2 of the type 1 orifice connects the orifice to the container, it was examined using the SEM (fig. 11). Along with molybdenum and oxygen, silicon was present throughout the sample and is consistent with the formation of molybdenum silicides.

Type 2 #1 Orifice

The Type 2 orifice #1 was cut in half and a slice removed. Two cross sections of the sample, including crystal formations, were removed and mounted in epoxy and labeled site 1 and 2. Large grains were observed in both samples upon visual and microscopic examination (fig. 12). Microscopic examination of Site 1 and 2 revealed crystals again initiating growth at single grains (fig. 13-14, respectively). SEM analysis indicated only molybdenum, carbon and oxygen

were present in the base metal and crystals (fig. 15-16). While carbon was found in the base metal it was most likely from the epoxy mounting.

Type 2 #2 Skirt

The tungsten skirt is composed of three separate tungsten sheets. Two samples were cut from the skirt and mounted in epoxy so that the edge of the sheets was exposed. One sample was from the top, near the orifice, and the other was from the bottom .

The bottom skirt sample is composed of three tungsten sheets held close together by rivets. The outer and inner surface are smooth but sandwiched between the layers is a formation of small crystals (fig. 17). The crystals between the tungsten sheets are attached only to the inner sheet and appear to have the same orientation as the grain to which they are attached. SEM analysis indicated that the crystals were tungsten (fig. 18).

The top skirt sample is similar to the bottom skirt sample with the exception of a formation of a layer of very small crystals on the inner surface (fig. 19). The small crystals on the outer surface (between tungsten sheets) had the same orientation as the grains to which they were attached. However, the layer of very small crystals on the inner layer were randomly oriented with respect to the grains in the base metal. There also appeared to be a series of holes between base metal and the crystal formation. SEM analysis indicated that the small crystals on the outer surface and the sheet up to the formation of holes were tungsten while the crystals from the hole formation on the inner surface were a mixture of tungsten and molybdenum (fig. 20).

Type 2 #2 Orifice Liner And Orifice Shell

Specimens were cut from tungsten shell and the orifice liner that backed the type 2 #2 molybdenum orifice. For definition, the "shell" of refractory metal sheets is on the outer surface while the "liner" metal sheet is the inner surface. The tungsten shell is an extension of the tungsten skirt and also has three sheets of tungsten riveted together (fig. 21). The orifice liner lies between the tungsten shell and the Molybdenum orifice. There are small crystallites on the surface of the liner that abuts the tungsten shell. These crystals do not have the same orientation as the grains of the base metal (fig. 22). SEM revealed that the crystals were a tungsten / molybdenum mixture on what was probably was a pure molybdenum liner (fig. 23). Tungsten appears to have migrated part way into the orifice liner.

Type 2 Glass

A sample of glass found on the inside of the type 2 #2 orifice was mounted on carbon tape for examination. On the black carbon tape, the white inclusions in the glass were now clearly visible (fig. 24). A SEM analysis of the white

inclusions and the dark glassy areas revealed a marked difference in composition. The dark glassy areas were composed of silicon, oxygen and carbon. The white inclusions were composed of molybdenum, sodium, oxygen and carbon.

Powder From Inside Glass Tubing

Sticky carbon tape was used to wipe off the inside of an extruded glass tube from the type 1 quartz melter. SEM examination of the powder deposited on the carbon tape revealed the presence of silicon, oxygen and molybdenum (fig. 25).

Thermodynamic Modeling

The Chem Sage program was used by Dr. Ted Besmann for thermodynamic equilibrium calculations. Equilibrium conditions were calculated using the same initial constituents and varying the equilibrium temperature from 1800°C to 2000 °C in 50 °C steps. Constituents initially present were: Molybdenum as a solid, quartz as a liquid and H_2, O_2 and Ar as gases. The elemental mole fraction of each component was calculated for the product gas phases and were graphed (fig. 26). For Molybdenum, the elemental mole fraction in the gas phase increased from 1.66E-07 at 1800 °C to 2.99E-05 at 2000 °C. The molybdenum-containing phases present in the vapor included: Mo, MoO_2, $HMoO_2$, MoO_3, MoO, H_2MoO_4, H_2MoO_3, Mo_2O_6, H_2MoO_2, Mo_3O_9, $Mo4O_{12}$ and Mo_5O_{15}. The oxygen containing vapors are termed Mo-O_x. In all cases, the mole fraction of oxygen present in the gas phase was much higher than the maximum level of oxygen impurity to be expected in the gas stream obtained from the high purity Ar and H_2 sources. This indicated that the oxygen is supplied by a reaction of molten quartz with the reducing atmosphere (fig. 27). The potential reduction of SiO_2 to silicon and oxygen was observed at all calculated temperatures. At 1800°C, and under steady state equilibrium conditions , a rough estimate of the amount of molybdenum that could be transported by Mo-O_x gas phases which are then reduced and deposited under reducing conditions is roughly 6.25kg or 13.8 lbs. per year.

Key Results

The findings from the type 1 and 2 quartz melters, while quite similar, did have some distinct differences thus providing complementary information. The major difference between the type 1 and 2 melters is the use of tungsten and the operating temperature. The type 1 melter is constructed entirely from molybdenum components while the type 2 melter uses tungsten for the container wall and skirt. The use of tungsten alters some of the crystal formations in the type 2 melter, but not all them (fig. 28). First is the reduction in size of the molybdenum crystals. Second is the formation of a thin molybdenum / tungsten layer at the top of the tungsten skirt. In addition, the absence of tungsten crystals in the type 2 orifice and skirt is a key finding that is supported by thermodynamic modeling which shows a very low tungsten content in the vapor phase under the

same conditions that produced a substantial molybdenum content in the vapor phase.

Discussion

Optical microscopy indicated that crystal formation was initiated at single grains. The crystal then grew to extend over adjoining grains and in some cases extended to over 50 mm in length. Within the metal of the molybdenum orifice, the grain size appeared to be larger for the type 2 samples than for the type 1 samples . This difference can be attributed to either fabrication methods or dependent on operating temperatures since the type 2 quartz melter operates at a higher temperature than type 1. Examination of the interface between the crystal and the bottom of the orifice surface confirmed that molybdenum crystals were vapor deposited on the orifice in the type 1 and the type 2 quartz melters. Preference in growth direction for the molybdenum crystals also suggests vapor deposition over an extended time period. The growth direction for type 1 crystals is in the (111) direction and is the same plane reported for the growth of numerous BCC metals.

Optical and SEM examination of orifice and skirt samples indicate vapor deposition is the active mode of crystal formation and growth (fig. 29). Observations of several crystal metal interfaces indicate nucleation at surface of the type 1 and type 2 orifice occurred at single grain sites and the crystal then appeared to grow over the adjacent grain sites. This is consistent with vapor depositions observed during production of thermal barrier coatings and thin films on single crystal substrates. Single crystal substrates initiate growth of the thin films and coatings with respect to the substrate lattice and subsequent growth of the film or coating is highly ordered.

Vapor deposition is also indicated by the size and location of crystal formations on the type 1 and type 2 skirts. Crystal formation occurred preferentially at cooling vents cut in the skirt and was more pronounced at the vents near the top of the skirt. Refractory metal crystals diminished in size, length and number with increasing distance from the bottom surface of the orifice. The largest concentration of crystals occurred where the hotter gas at the bottom of the orifice first encounters the Ar/H_2 gas mixture entering the skirt from outside the molten glass container and skirt. This also indicates that molybdenum is reduced from the vapor phase and then deposited on the skirt or orifice.

Thermodynamic modeling shows that the reaction of molten quartz in a reducing environment provides a source of oxygen that is then available to react with the molybdenum orifice and inner surface of the molybdenum container walls at type 1. Three observations tend to confirm the model. First is the observation using several different analytical methods of the presence of silicon and Mo_3Si on the inside of the Type 1 container. The second is the presence of Molybdenum in the powder on the inside of the type 1 quartz tube. The third is the presence of molybdenum in the solidified glass from the type 2 quartz melter. This glass was

solidified during the quartz melter shutdown and thus has the composition of molten quartz present during operation. All these observations confirm the thermodynamic modeling.

Conclusions

Thermodynamic calculations, supported by analysis of specimens from the type 1 and type 2 quartz melters, indicate the source of molybdenum oxides present below the orifice is from inside the molten quartz container (fig. 30). The most likely pathway is for the oxidized molybdenum vapor species from the melter container to pass through the orifice either as a gas layer between the molybdenum surface and the molten quartz, as bubbles in the molten quartz, or as a dissolved component of the molten quartz. As the molten quartz passes through the orifice the glass begins to cool and any absorbed gas species or bubbles would escape from the just formed quartz tube. If the gas forms a layer between the solid molybdenum surface and the molten glass it would easily cool after passing through the orifice. If a gas interface is formed, it may assist the flow of molten glass through the orifice. Once the molybdenum oxides from the melt container pass through the orifice it is exposed to an Ar/H_2 atmosphere. In this environment, molybdenum oxides reduce to molybdenum and condense out as crystals preferentially on a cooler molybdenum (111) surface.

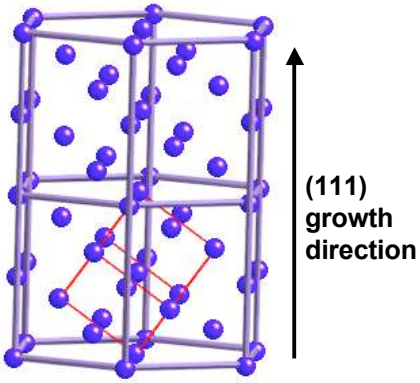
Analysis of specimens from the type 1 and type 2 quartz melters lead to the following findings (fig. 31). Molybdenum crystals on the bottom of the orifice and skirt are formed when molybdenum oxide vapors are reduced to $Mo_{(S)}$ by the Ar/H_2 gases below the orifice. Upon cooling, molybdenum then condenses out of the vapor phase and deposits on the cooler surfaces. Nucleation of molybdenum crystals occurs on single grains and crystals grow is in a preferred direction. The dramatically smaller Mo crystals on the type 2 orifice indicates that $Mo\text{-}O_x$ formation occurs primarily at the inner melt container surfaces which are only present at type 1. Much less $Mo\text{-}O_x$ must form along the melt side of the orifice surface.

Recommendations

The formation of molybdenum crystals on the orifice and skirt of the quartz melter is the main reason for failure and subsequent shutdown of quartz melters. Considering the finding above, two potential methods to extend quartz melter lifetime can be considered (fig. 32). The first is to eliminate molybdenum crystal formation. The second is to control the location and or direction of crystal growth.

XRD Examination of Representative Mo Crystal Determined Growth Plane

Diffraction pattern of a representative Mo crystal determined the growth direction as the (111) direction for the body centered cubic cell.



The BCC cell shown above is an idealized atomic arrangement of molybdenum atoms within the crystal.

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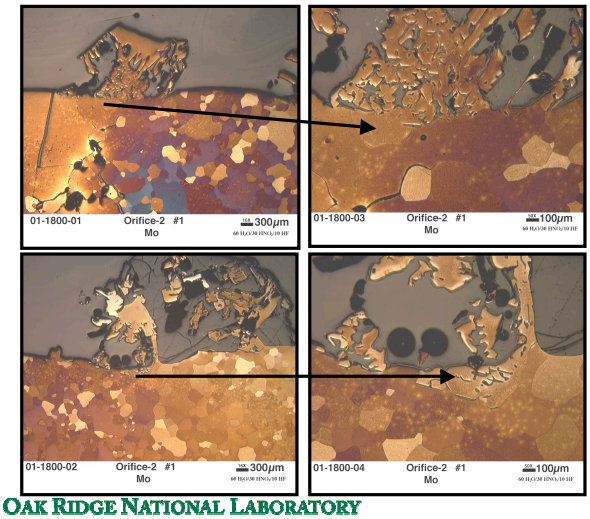
Figure 1

Crystal Formations on Orifice 2 #1 Appear to Have Single Nucleation Site



Examination of crystal formations on the orifice indicate that crystal growth is initiated and coherent with that of individual grains.

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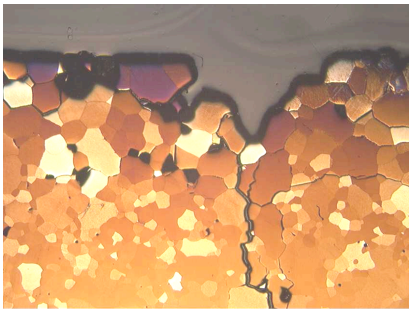
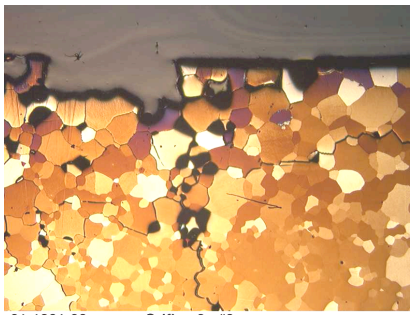
Figure 2

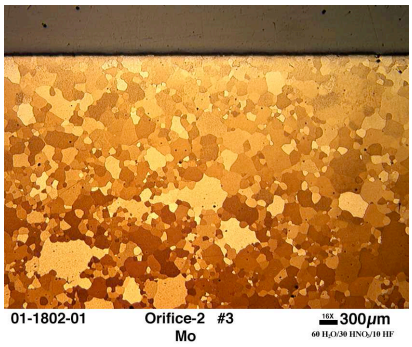
Larger Grains Formed on the Inner Surface of Type 1 Orifice #2 Site 2

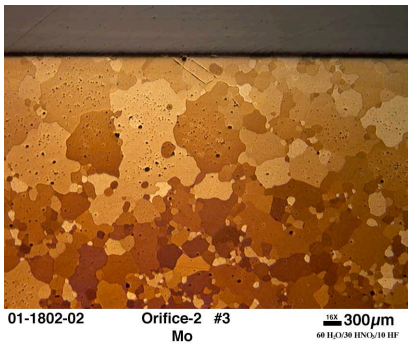




Section 2 where the orifice and container wall merge had larger sized grains near the surface exposed to molten quartz. Changes in grain size could be due to fabrication or recrystallization due to temperature gradients or exposure to molten quartz.

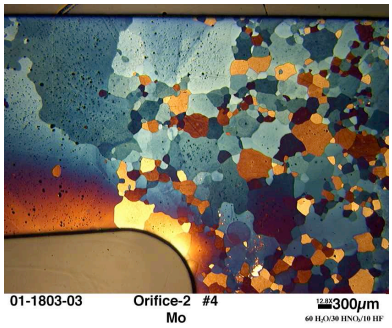
Grain Size Varied Within the Type 1 Orifice #2 Site 3 Sample

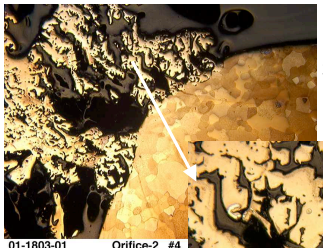




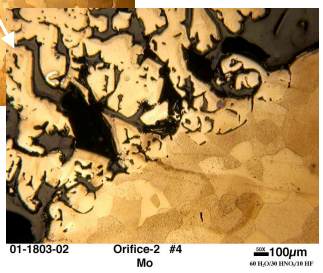
While multiple grain sizes appear in specimens from the type 1 orifice, no systematic change appears in the outer and interior metal matrix.

Orifice 2 #4 Was Located Where the Skirt Attaches to the Orifice





Expanded View: 300□m to 100□m



Grain size varied within this sample.

Crystal growth was initiated at specific grains.

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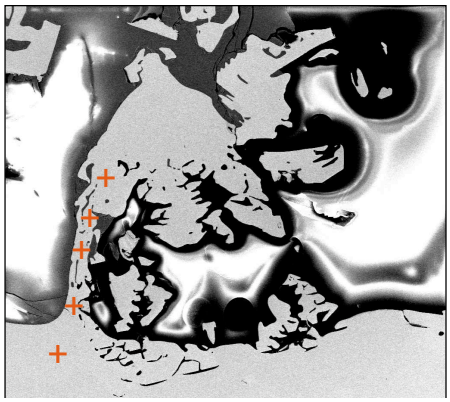
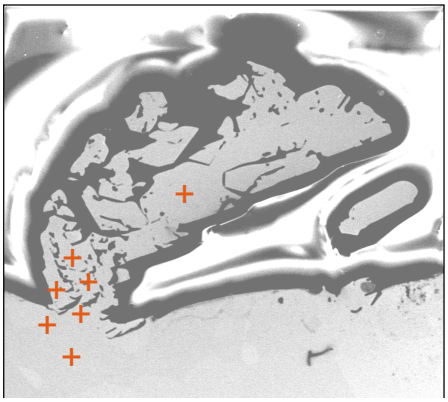
Figure 5

SEM Examination on Type 1 Orifice #2 Sample 1 at Different Locations





Molybdenum, oxygen and a trace of carbon (from the epoxy mounting) were observed in the crystals and the bottom of the orifice (red + marks).

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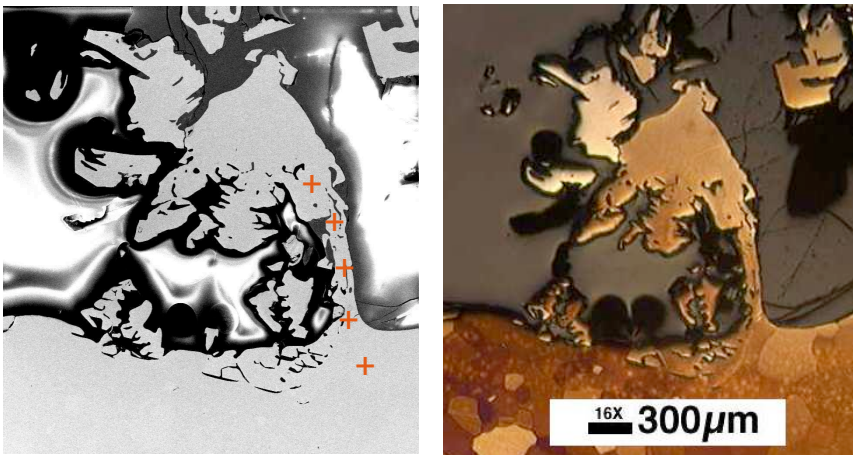
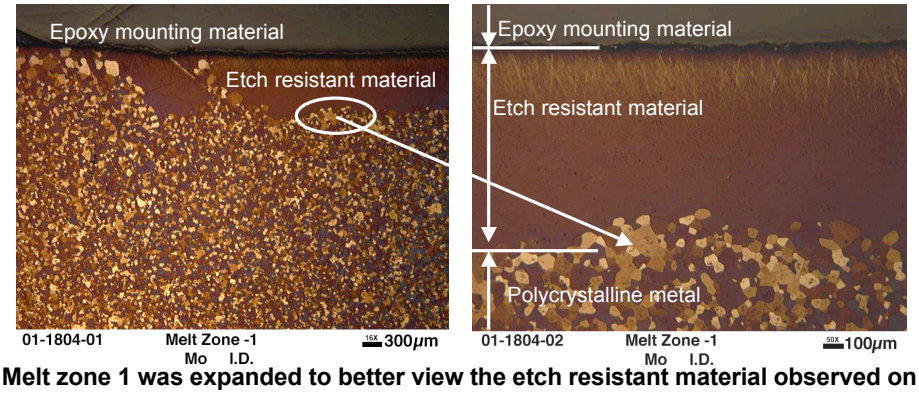
Figure 6

SEM and Optical Micrographs Are Similar for Type 1 Orifice #2 Site 1

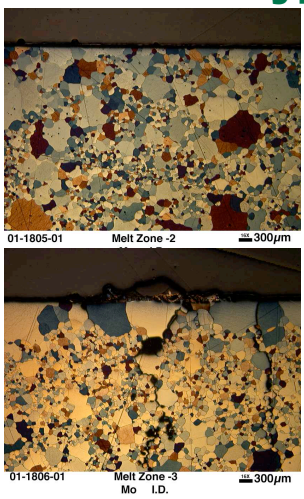


Melt Zone #1 Has Regions on the Hot Face That Are Resistant to Etching



Melt zone 1 was expanded to better view the etch resistant material observed on the hot face surface of some of the prepared samples. This resistant material may be molten quartz solidified on the surface during furnace cooling.

Micrographs Were Taken at Different Type 1 Melt Zone Sites



Grain size varies within each of four different sampling locations near the melt zone of the container tube.

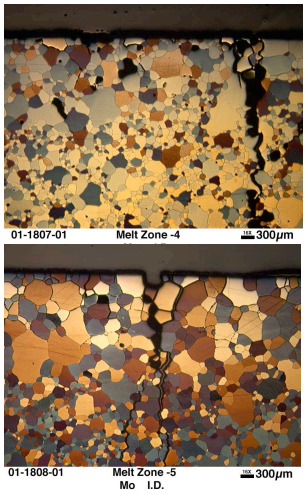


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Figure 9

XRD Revealed the Presence of Glass, Mo_3Si & Mo on the Interior Melt Zone

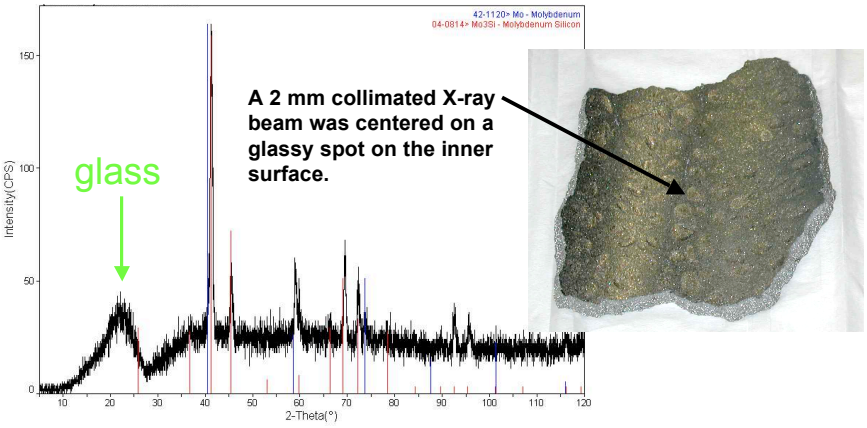

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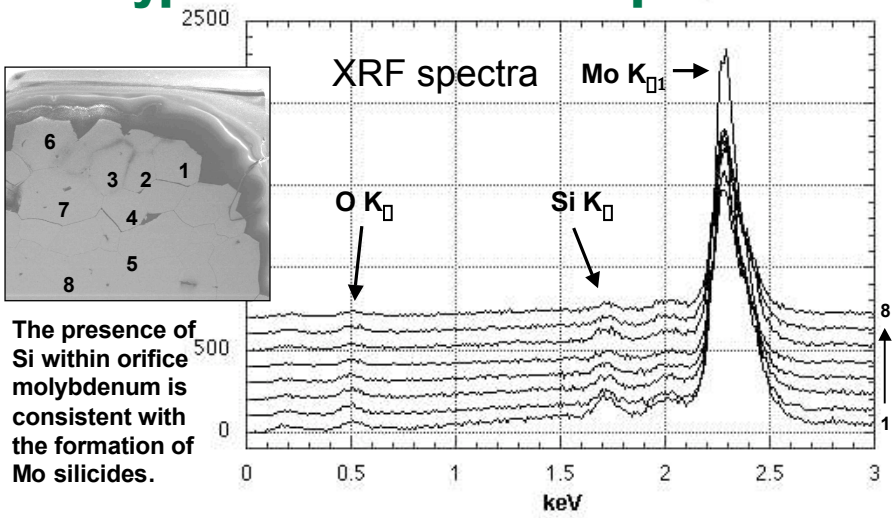
Figure 10

Si was Present in SEM Examination on Type 1 Orifice # 2 Sample 2



The presence of Si within orifice molybdenum is consistent with the formation of Mo silicides.

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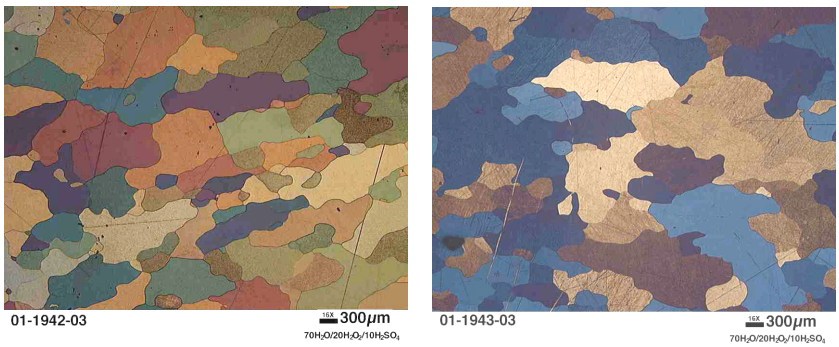
Figure 11

Large Grains Were Observed in the Sample Interior of the Type 2 Orifice



After polishing, grains were large enough to be observed by visual inspection. Micrographs of the interior of the sectioned samples reveled only large grain boundaries.

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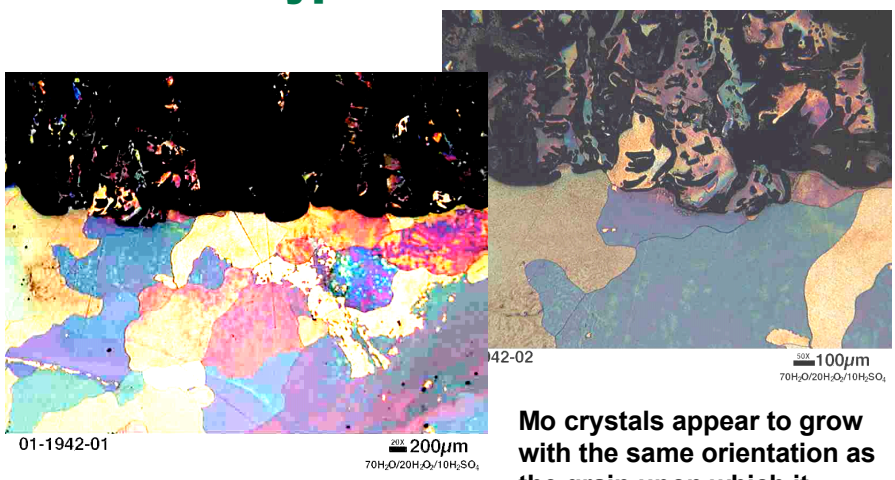
Figure 12

Small Individual Crystals Grow on the Surface of Type 2 Orifice #1 Site 1



Mo crystals appear to grow with the same orientation as the grain upon which it nucleated.

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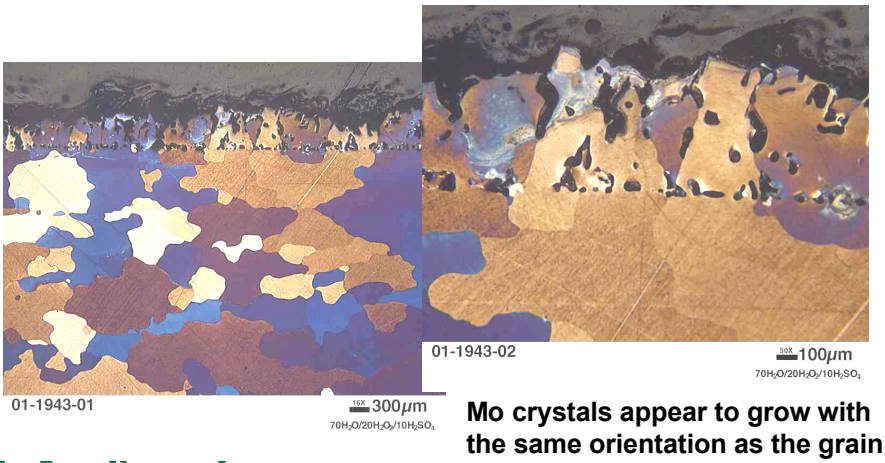
Figure 13

Small Individual Crystals Grow on Surface of Type 2 Orifice #1 Site 2



Mo crystals appear to grow with the same orientation as the grain upon which it nucleated.

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Figure 14

Molybdenum, Oxygen and Carbon Are Present in Type 2 Orifice #1

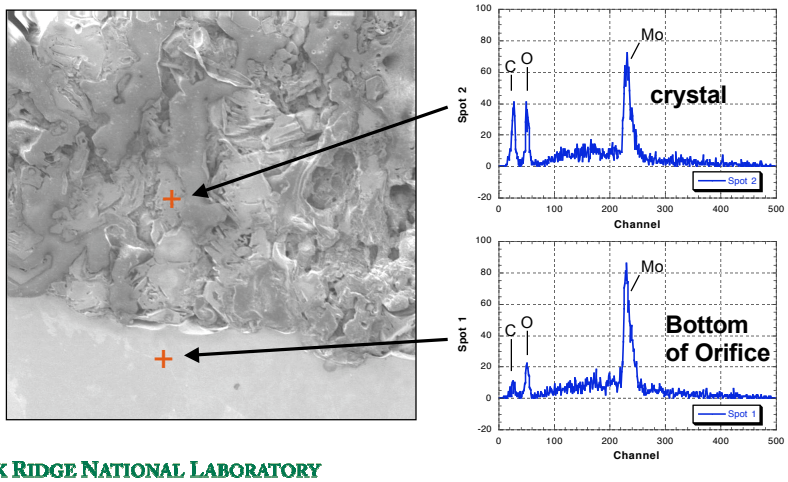

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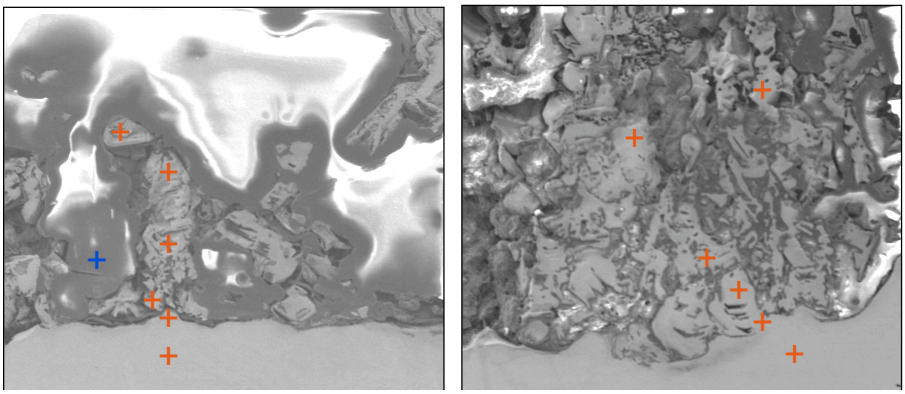
Figure 15

Only Mo Was Identified in Crystals on the Bottom of Type 2 Orifice #1 Site 1



Molybdenum, oxygen and carbon (from the epoxy mounting) were observed using SEM analyses for the crystals and the bottom of orifice (red). Carbon and a trace of oxygen was observed for the epoxy mounting (blue).

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Figure 16

Crystals Lie Sandwiched Between the 3 W Sheets of the Type 2 #2 Skirt

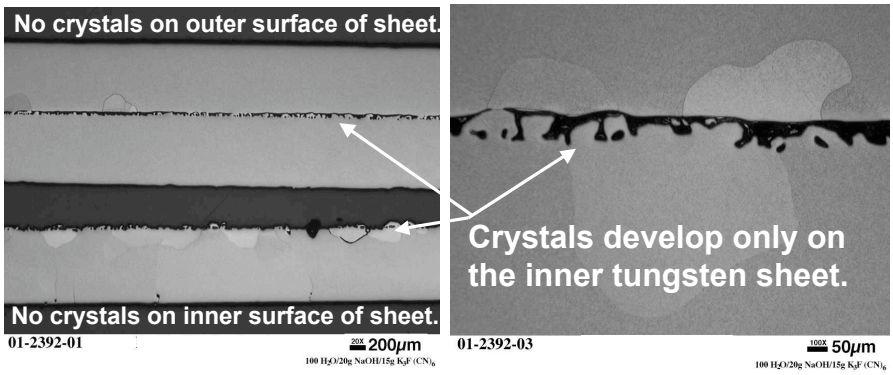

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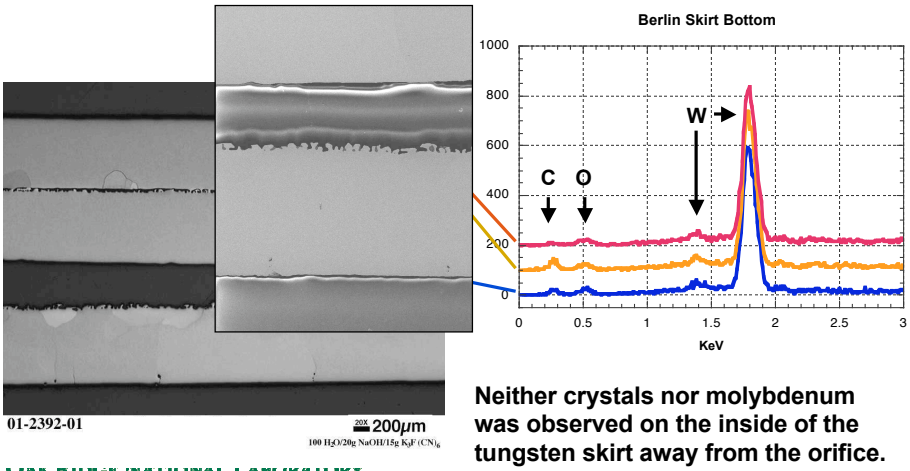
Figure 17

Only W Crystals Form Between the Sheets at the Bottom of Type 2 Skirt



Neither crystals nor molybdenum was observed on the inside of the tungsten skirt away from the orifice.

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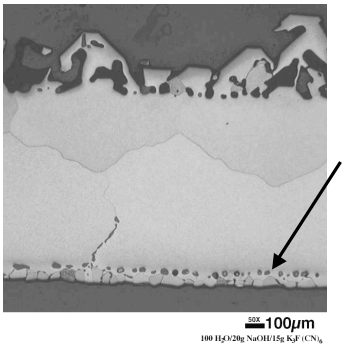
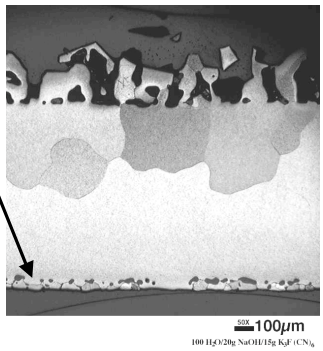
Figure 18

Crystals Form on Both Sides of the Inner W Sheet at Top of Type 2 Skirt

Oriented crystals grow on the outer surface of the tungsten sheet.



Holes are present between the base metal and the very small crystals on the inner surface



Very small crystals grow in random directions on the inner surface of the tungsten sheet.

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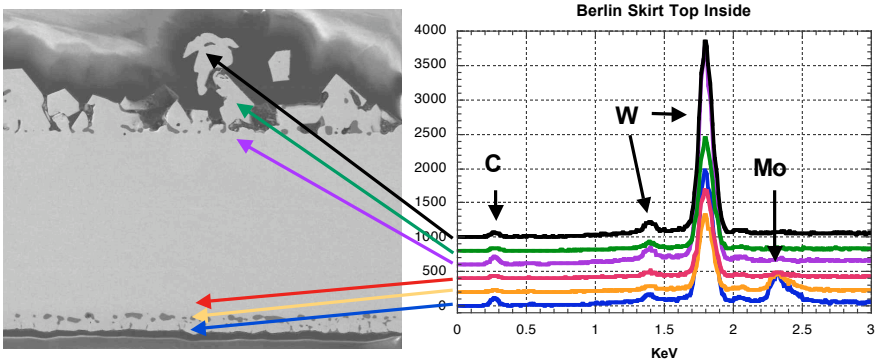
Figure 19

W or W/Mo Crystals Grow on Opposite Surfaces at the Top of the Type 2 Skirt



SEM shows crystals growing on opposite sides of the W sheet are either W for the outer surface or W /Mo for the top inner surface of the Berlin skirt.

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Figure 20

Three Tungsten Sheets Form the Orifice Shell of the Type 2 Melter

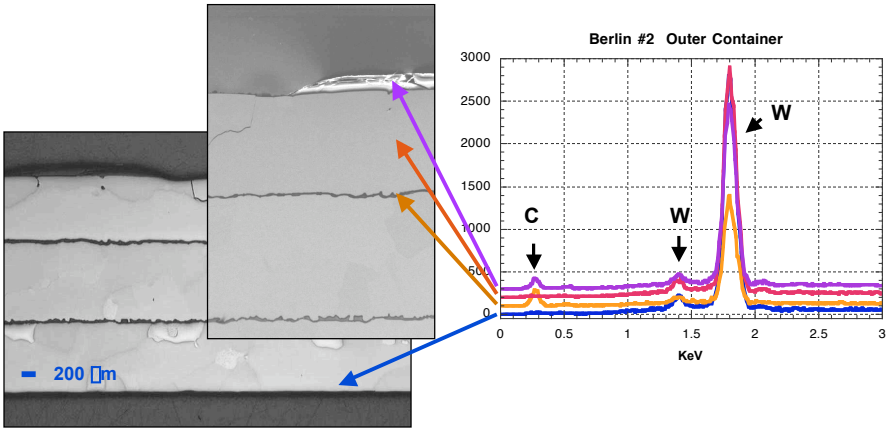

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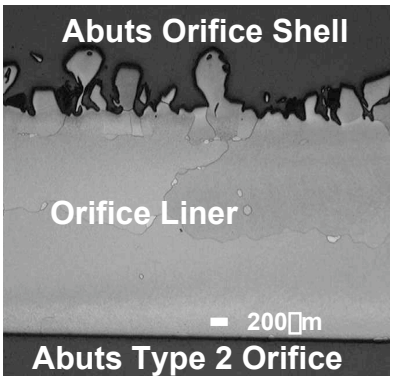
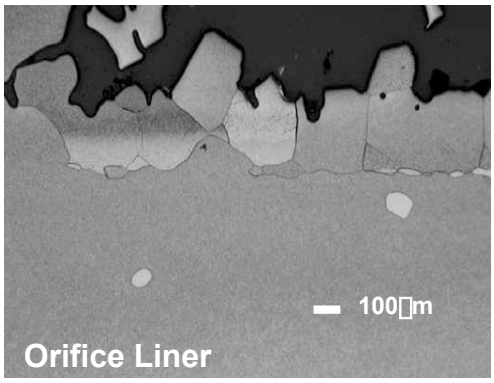
Figure 21

Crystals Grow at the Interface of the Shell & Liner Abutting Type 2 #2 Orifice





Crystals with random orientations grow as small grains on the surface of larger grains. This differs from crystal growth on orifice.

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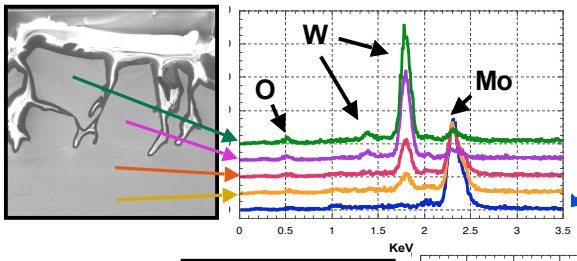
Figure 22

Tungsten Diffuses Into Molybdenum of Type 2's Orifice Liner

W / Mo crystals form between shell & liner.



Molybdenum Interior

Tungsten content decreases with depth as it diffuses into the orifice liner.

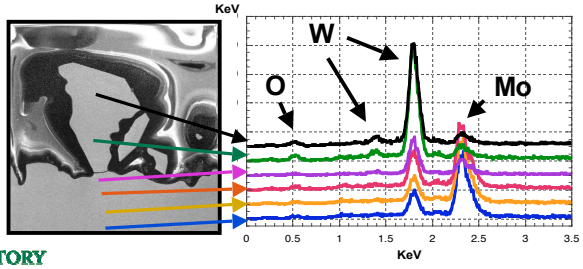


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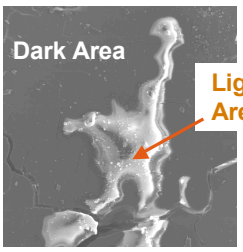
Figure 23

Light Areas in Glass on Inside of the Type 2 #2 Orifice Contain Molybdenum



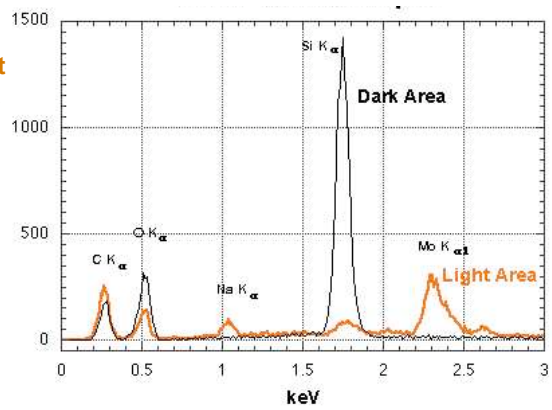


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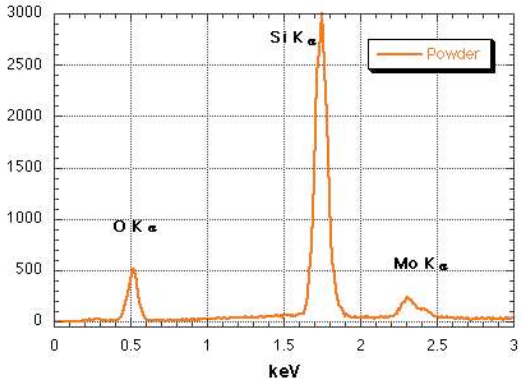
Figure 24

Powder From Inside the Extruded Glass Tube Contains Si, O and Mo



Depositions on inside of quartz tube, supports the hypothesis of Mo-O_X or Mo-Si-O_X vapor.

Figure 25

Elemental Mole Fraction Calculated Using Chem Sage

Starting Conditions

Stream Constituents	Amount/mol
Mo	1.00E+02
H2/Gas	6.00E+00
O2/Gas	1.00E-03
Ar/Gas	9.60E+01
SiO2 (Quartz)	1.00E+02

Oxygen mole fraction increases with temperature not with initial concentration.

Elemental Mole Fraction of Gas Phase Components

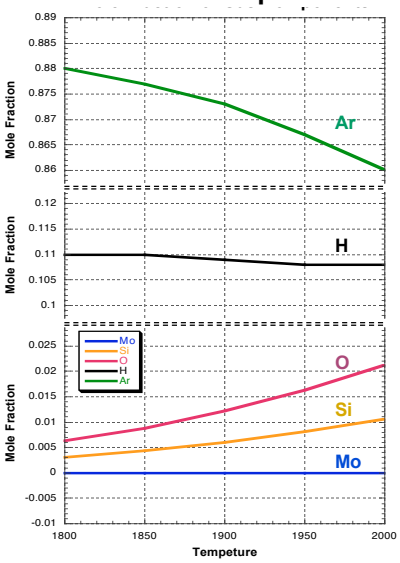


Figure 26

Thermodynamic Modeling Shows Mo Oxides Can Originate Inside Melter

- Oxygen available from SiO_2 reduced by Ar/H2
- Mo reacts with oxygen to form Mo-oxides
- MoO_X migrate through orifice & begins to cool
- Mo oxides reduce in Ar/H_2 on cooling
- Reduced molybdenum condenses out forming crystals on bottom of orifice and skirt

At 1800 °C, ~ 13 lbs of molybdenum can be transported in a year.

Use of Tungsten in Type 2 Quartz Melter Alters Crystal Formations

- Differences
  - W container limits Molybdenum available for deposition
  - Tungsten skirt alters crystal formation
    - Surface at top of skirt has an outer Mo/W layer
    - Crystals in Mo/W layer have different orientations than that of the base metal
- Similarities
  - No tungsten crystals form under current operating temperature & P_{O_2}

Experimental Results Indicate Vapor Deposition of Mo crystals

- Nucleation of crystals by single grains
  - Similar to coatings grown on single crystal substrates
- Clustering of crystals at vents
  - Mixing point of Mo rich and poor gases
  - Potential temperature variation in gases
- Growth of crystals on the (111) plane
  - Preferred growth plane in BCC metals

Thermodynamic Modeling shows Mo Most Likely from Inside the Melter

- Gas from outside melter would not pick up MoO_X at 2000 °C even with minor impurities of O_2 (1000 ppm)
- Addition of SiO_2 into calculations forms SiO which releases O to form MoO_X at equilibrium at 2000 °C
- As the saturated gas cools to 1800 °C, then Mo is deposited out of the reducing gas phase

Conclusions

- Vapor deposition of molybdenum occurs upon reducing gas phase Mo oxides on bottom of orifice and skirt
- Source of Mo oxides is most likely from transported through the orifice by the molten quartz or as a gas stream
- Below the orifice the MoO_X phases is releaxed from the molten quartz and reduced in the Ar/H_2 environment
- Molybdenum crystals nucleate at single grains and grow in a preferred direction

Recommendations Are to Either Eliminate or Direct Crystal Growth

- Eliminate Crystal Growth
  - All tungsten components
  - Tungsten lining on the inside of the melter
  - Mo sequestering agent in Ar/H_2 gas below orifice
- Direct Crystal Growth
  - Attach seed crystals on skirt vents and orifice
  - Attach a textured Mo surface on bottom of orifice and skirt
  - Develop a coating to inhibit crystal growth