OXIDATION, VOLATILIZATION, AND REDISTRIBUTION OF MOLYBDENUM FROM TZM ALLOY IN AIR

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Published January 2000

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ABSTRACT

The excellent high temperature strength and thermal conductivity of molybdenum-base alloys provide attractive features for components in advanced magnetic and inertial fusion devices. Refractory metal alloys react readily with oxygen and other gases. Oxidized molybdenum in turn is susceptible to losses from volatile molybdenum trioxide species, $(MoO_3)_m$, in air and the hydroxide, $MoO_2(OH)_2$, formed from water vapor. Transport of radioactivity by the volatilization, migration, and re-deposition of these volatile species during a potential accident involving a loss of vacuum or inert environment represents a safety issue. In this report we present experimental results on the oxidation, volatilization and re-deposition of molybdenum from TZM in flowing air between 400 and 800ºC. These results are compared with calculations obtained from a vaporization mass transfer model using chemical thermodynamic data for vapor pressures of $MoO_3(g)$ over pure solid MoO_3 and an expression for the vapor pressures of $MoO_2(OH)_2$ from the literature. Calculations correlate well with experimental data. The volatilization process is dominated by MoO_3 above 550°C and by $MoO_2(OH)_2$, formed from the small ingress of water vapor, at temperatures below 550°C. Partial saturation of gaseous species of (MoO_3) near specimen surfaces accounts for observed reductions in volatilization rates at lower flow rates at 700ºC. We have thus demonstrated predictive capabilities of the model to account for volatilization as influenced by temperature, humidity (vapor content), and flow rate.

We obtained oxidation rates (mm/h) as indicated by the recession into the base metal. These rates which accounted for the concurrent processes of oxidation and volatilization showed trends similar to other refractory metals, namely, niobium and tantalum.

Deposition of MoO_3 downstream at lower temperatures was calculated with a model using saturation ratios of $(MoO_3)_m$ within segmented regions. Calculated locations of peak distributions and maximums within the temperature gradients generally correlate reasonable well with experimental measurements. Scanning electron microscopy showed that deposits collected in a final filter consisted of small agglomerated particles. We might expect such nucleation, growth and agglomeration of particles to result from the supersaturation of the $(MoO_3)_m$ upon cooling during transport. Hydroxide molecules also decompose back into $MoO_3(s)$ and water vapor at low temperatures. These latter two processes and increased surface areas due to extensive crystal growth from $(MoO_3)_m$ are plausible explanations for differences in peak height and distribution predictions between the model and experiments..

The oxidation-driven mobilization data, along with activation calculations determining radioactive isotope inventories, were used to determine airborne dose rates. These calculations showed that site boundary doses from TZM alloy would be one to two orders of magnitude lower than tungsten at comparable temperatures.

SUMMARY

In this report we present the results of experimental and modeling approaches used to define the oxidation, oxidation-driven vaporization, and re-condensation behavior of TZM alloy tested in air environments between 400 and 800°C. Experimental procedures are presented in Section 2. This section describes the test system used to expose specimens at various temperatures and flow rates in an air environment. Included are descriptions of the system and procedures used to collect, quantify, and examine the volatilized and re-deposited oxide products.

Experimental results are presented in Section 3. This includes the integration of mass change and inductively coupled plasma-atomic emission spectroscopy (ICP-AES) results to account for the concurrent processes of oxygen pick-up and oxide volatilization. These integrations provide oxidation rates (mm/h), the relative amount of the oxide that is volatilized, and the mobilization rate of molybdenum (g/m^2-h). Features of the oxidized specimens based on physical appearance, optical metallography, scanning electron microcopy (SEM), and x-ray diffraction (XRD) are given in Section 3.2. Illustrations of re-deposited oxides are shown in Section 3.3. Detailed descriptions of deposited oxide products on various system components, the chemical analyses procedures, and ICP-AES results providing mass flux calculations and distribution information are given in Appendices A, B, and C, respectively.

Experimental results are discussed in Section 4. Oxidation rates, mechanisms and the relative amount volatized at various temperatures are compared to the available information in the literature in Sections 4.1 and 4.2. A description of our vaporization mass transfer model including the procedure to handle partial saturation of volatilizing oxide species near specimen surfaces is given in Section 4.3. The treatment for partial saturation helped to explain flow rates effects. In this section we also present equilibrium vapor pressures for both volatilizing polymers of $(MoO_3)_m$ and $MoO_2(OH)_2$ as a function of temperature and various water vapor concentrations. The combined results from these two species, with $MoO_2(OH)_2$ dominating at low temperatures and $(MoO_3)_m$ dominating at high temperatures, provide a very good correlation with experimental measurements. Our model predicting the re-deposition from $(MoO_3)_m$ vapors is described and compared with experimental molybdenum profiles in Section 4.4. The model provides good correlation with the locations of peak deposition for tests performed at and above 600°C. Plausible reasons for differences between predicted and measured peak heights and subsequent deposition profiles include: 1) nucleation, growth, agglomeration, and transport of particles due to supersaturation of $(MoO_3)_m$ upon cooling, 2) extra surface areas for re-deposition resulting from crystal development, 3) the role of $MoO_2(OH)_2$ in the mobilization process at lower temperatures. A comparative study showing mobility-based doses derived for TZM alloy and tungsten is given in Section 4.5. This comparison shows that doses from the activated products of TZM alloy are one to two orders lower than tungsten for similar accident conditions of a conceptual fusion device.

ACKNOWLEDGMENTS

CONTENTS

1.0 INTRODUCTION

The potential of high temperature strength has motivated much interest in the oxidation behavior of refractory metals. Many of these studies have been oriented toward extremely high temperatures, e.g., up to 1700ºC, often under vacuum or environments with low oxygen activities [1,2,3,4]. Studies [1,5,6] on molybdenum at lower temperatures in high levels of oxygen report: 1) parabolic rate law at 250 to 450ºC, 2) linear behavior above 400ºC, 3) a role of MoO_2 and other oxides (MoO_Z), where $2 < Z < 3$, between 450 to 650ºC. At temperatures above 650ºC, oxidation rates are largely influenced by the high rates of the vaporization of the various polymers of MoO_3. Water vapor will also react with $MoO_3(s)$ to form a volatile hydroxide. Vapor pressures of this $MoO_2(OH)_2$ species have been documented by Speiser and St. Pierre [6]. We performed this study to explore conditions more typical to future fusion devices and to demonstrate predictive capabilities of a vaporization mass transport model. The model had provided excellent mass transport predications for the highly volatile species produced in the tungsten-steam system.[7]

2.0 EXPERIMENTAL PROCEDURES

We tested specimens of TZM alloy between 400 and 800ºC in air flowing at a rate of 1000 sccm. Flow rates were varied from 10 sccm to 2500 sccm for tests at 700°C. Gas velocities for these flow rates at 700°C, the pressure at the INEEL, and the test chamber cross section ranged from 0.033 to 8.3 meters per minute. The specimens were prepared from a 22-mm diameter bar made by vacuum arc casting and then hot extrusion. The composition was Mo – 99.25 wt%, Ti - 0.50 wt%, Zr - 0.102 wt%, C - 0.018 wt%, Si – 0.0034 wt%, Fe – 0.001 wt%, Ni - 0.001 wt%, O - 0.0009 wt%, and N – 0.0005 wt%. Disc-shaped specimens of either 1.75- or 3.5-mm thickness were sectioned from the bar. Surfaces were polished to a 600-grit finish.

The specimens were tested in a dual furnace arrangement as shown in Figure 1. The test system consisted of three regions, the reaction furnace, a transition zone, and a furnace in which deposition occurred. The dimensions of the quartz components in the system were as follows: reaction chamber (4.0 cm I.D. x 36 cm long), the transition zone (1.5 cm I.D. x 13 cm long), and the deposition tube (1.5 cm I.D. x 90cm long). Air from a compressed cylinder flowed through a column of Drierite and was preheated to 400ºC before passage into the reaction furnace containing the specimen. Upon exiting the reaction furnace the gas stream then passed through a transition zone and a three-zone furnace with a prescribed temperature gradient. Oxidation products transported from the specimen were re-deposited in these components downstream at temperatures between 800 and 25ºC. The quartz collection tube in the three-zone furnace was lined with sections of 1-in., or 3-in., long quartz tube. These were used to determine the distribution to the re-deposited molybdenum oxide products along the temperature gradient. A final filter of quartz wool was used to ensure the complete capture of products at the end of the collection tube.

Vented to hood

Three zone furnace (800 to 400C) to condense volatilized products.

Quartz wool used to collect any remaining volatile oxide particles

Deposition furnace (32-in.)

Quartz chambers
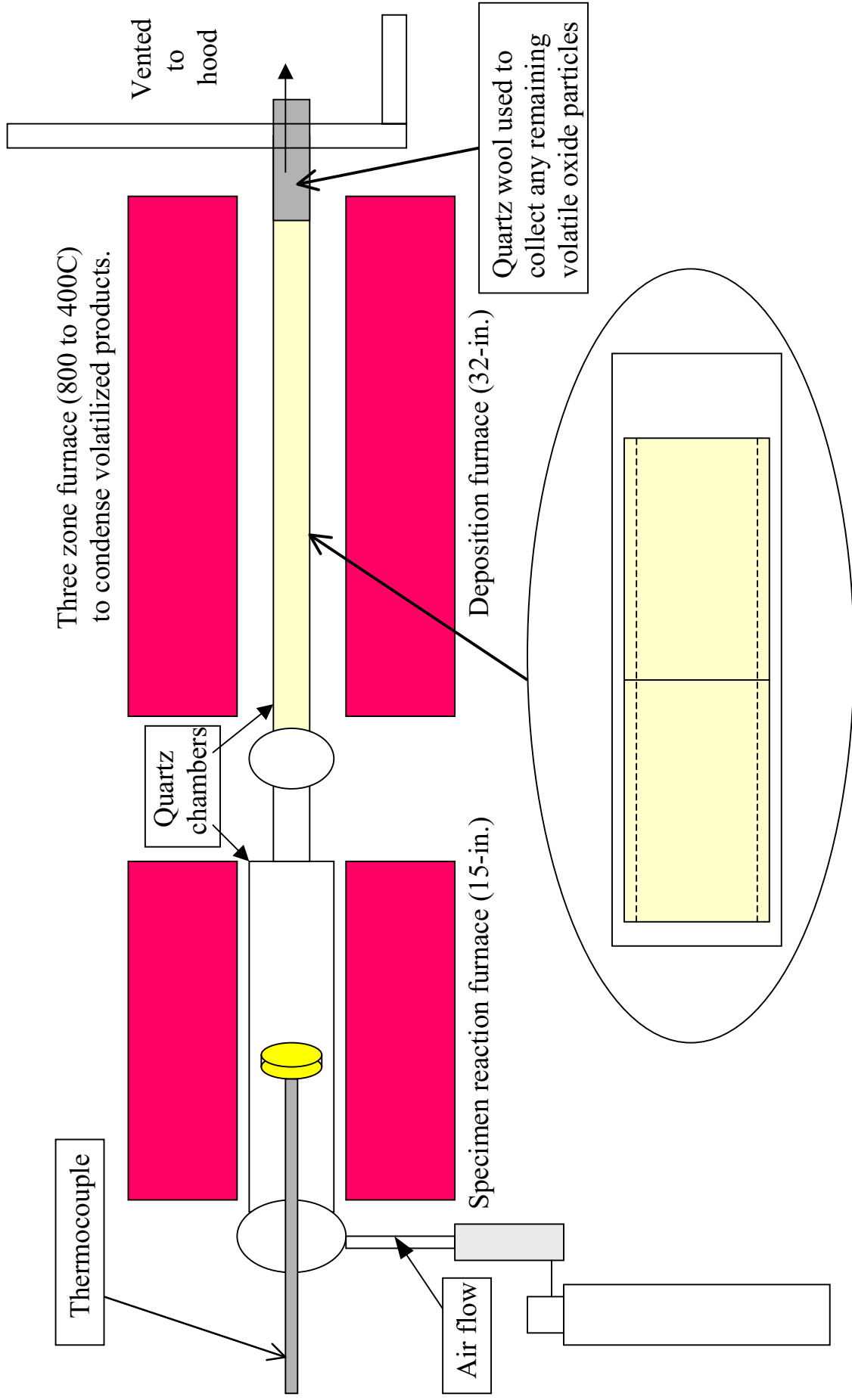
Specimen reaction furnace (15-in.)

Thermocouple

Air flow

Figure 1. Schematic of test system to study the mobilization/deposition of MoO_3 from TZM molybdenum alloy in flowing air.

Specimens were weighed before and after testing to obtain mass change. The quartz components were visually inspected following the tests. Descriptions of the oxide products deposited on the components are given in Appendix A. The quartz components were then separately processed as described by the procedures in Appendix B. This involved cleaning the components with 2.9 molar $NH_4(OH)$ solution to remove the molybdenum oxide products. The solutions were heated to drive off NH_3 and then acidified with nitric acid and diluted to 100 ml or 250 ml samples. The diluted solutions were analyzed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). We examined oxidized specimens with optical metallography, scanning electron microcopy (SEM), and x-ray diffraction (XRD).

3.0 EXPERMENTAL RESULTS

3.1 OXIDATION AND MOBILIZATION RATES. The test matrix with temperatures, times, and flow rates, along with mass changes and total amounts of molybdenum transported as measured by ICP-AES are given in Table 1. Oxidation rates and molybdenum mass flux rates can be obtained from the data in Table 1. However, mass changes need to be corrected to allow for concurrent oxygen pick-up and losses by molybdenum oxide volatilization. For these calculations we assume that the oxidation process essentially produces molybdenum trioxide by the reaction: $Mo + (3/2) O_2 \rightarrow MoO_3$. The total mass of molybdenum (M_t^{Mo}) reacted can then be determined by the relationship expressed in Eq.(1):

$$(M_t^{Mo}) = (M_V^{Mo}) + (96/48)[\Delta m + (M_V^{Mo})], \qquad (1)$$

where Δm is the mass change and (M_V^{Mo}) is the amount of volatilized molybdenum as measured by ICP-AES.   The results of these calculations are given in Table 2. We used these values to determined recession rates (mm/h) and the fraction of oxidized molybdenum that volatilized. Recession considered as the depth of alloy converted to oxide was calculated using 10.2 g/cm^3 as the density of molybdenum and initial surface area.   We used metallography to measure recession rates (mm/h) for a limited number of specimens. Average mass flux values were obtained from the sum of the ICP-AES measurements for all system components for a given test, the initial surface area of the specimen, and the test duration. The detailed calculations from the ICP-AES measurements are reported in Appendix C. The calculations in Appendix C show the average mass flux from the specimen, molybdenum distributions, and temperature profiles. Essentially there was no indications of molybdenum mobilization at 400ºC. We use a flux value, which we refer to as the Engineering Maximum Flux Value (EMFV), at this temperature. It is determined from the ICP-EAS detection limit from a blank solution. The flux values at 500ºC are orders of magnitude higher than the detection limit and more than ten times greater than the uncertainty based on ICP-AES limitations. A summary of the measurements and calculations refer to above are shown in Table 2.

Table 1. Test matrix with mass changes and ICP-AES measurements.

Specimen	Temp., ºC	Flow rate, sccm	Time, h	Initial area, cm^2	Initial weight, g	Final weight, g	Mass change, g	ICP-AES total, g
TZM14	400	1000	24	10.345	13.6641	13.6684	0.0043	1.5E-6(a)
TZM15	500	1000	24	10.369	13.8827	13.94643	0.06373	1.02E-4
TZM1	500	1000	24	9.101	6.6733	6.7429	0.0696	5.97E-5
TZM16	550	1000	24	10.402	14.0636	14.4353	0.3717	9.04E-4
TZM11	602	1000	8	10.235	13.15934	13.35724	0.19790	3.25E-3
TZM7	599	1000	24	9.136	6.88303	7.65951	0.7765	3.56E-3
TZM8	650	1000	8	9.123	6.8263	7.3607	0.5344	2.51E-2
TZM4	699	10	2	8.994	6.1262	6.1887	0.0625	4.50E-3
TZM17	700	10	8	10.311	13.4955	13.7485	0.2530	1.91E-2
TZM5	699	50	2	9.111	6.7467	6.7988	0.0521	4.55E-3
TZM18	700	50	8	10.326	13.5565	13.7589	0.2024	5.75E-2
TZM10	699	100	2	9.112	6.71857	6.74414	0.02557	3.08E-2
TZM6	700	500	2	9.075	6.4623	6.4560	-0.0063	6.49E-2
TZM12	700	1000	2	10.343	13.67683	13.67773	0.0009	5.43E-2
TZM9	700	2500	2	9.069	6.52977	6.52907	-0.0007	5.67E-2
TZM3	750	1000	1	9.056	6.48254	6.1071	-0.3754	3.46E-1
TZM2	800	1000	1	9.150	6.9709	3.6501	-1.9056	2.22E+0

(a) The value reported for 400ºC is a maximum as established by the detection limit from ICP-AES.

**Table 2.** Oxidation and mass flux rates determined for molybdenum oxidation tests.

Specimen	Temp ºC	Flow rate, sccm	Time h	Total Mo reacted, g	Calculated recession rate, mm/h	Measured recession rate, mm/h	Fraction of reacted Mo volatilized	Measured mass flux g/(m^2-h)	Predicted mass flux g/(m^2-h)
TZM14	400	1000	24	0.0086	3.40E-5		1.74E-4	6.04E-5(a)	8.22E-5
TZM15	500	1000	24	0.1277	5.03E-4		8.00E-4	4.10E-3	2.97E-3
TZM1	500	1000	24	0.1393	6.25E-4		4.30E-4	2.73E-3	2.97E-3
TZM16	550	1000	24	0.7457	2.93E-3		1.21E-3	3.62E-2	2.28E-2
TZM11	602	1000	8	0.4053	4.85E-3		8.02E-3	3.97E-1	2.97E-1
TZM7	599	1000	24	1.5628	6.99E-3	6.24E-3	2.28E-3	1.62E-1	2.97E-1
TZM8	650	1000	8	1.1434	1.54E-2		2.19E-2	3.43E+0	4.17E+0
TZM4	699	10	2	0.1384	7.55E-3		3.25E-2	2.50E+0	7.06E-5
TZM17	700	10	8	0.5631	6.69E-3		3.40E-2	2.32E+0	7.06E-5
TZM5	699	50	2	0.1178	6.33E-3		3.86E-2	2.50E+0	4.60E-1
TZM18	700	50	8	0.5745	6.85E-3		9.95E-2	6.95E+0	4.60E-1
TZM10	699	100	2	0.1435	7.72E-3	1.12E-2	2.15E-1	1.69E+1	2.81E+0
TZM6	700	500	2	0.1820	9.83E-3		3.57E-1	3.57E+1	2.79E+1
TZM12	700	1000	2	0.1645	7.80E-3		3.30E-1	2.62E+1	4.93E+1
TZM9	700	2500	2	0.1687	9.12E-3		3.36E-1	3.13E+1	8.67E+1
TZM3	750	1000	1	0.2873	3.11E-2	5.46E-2	1.20E+0	3.82E+2	4.75E+2
TZM2	800	1000	1	2.8435	3.05E-1	3.85E-1	7.80E-1	2.42E+3	3.79E+3

(a) The value reported for 400ºC is a maximum as established by the detection limit from ICP-AES.
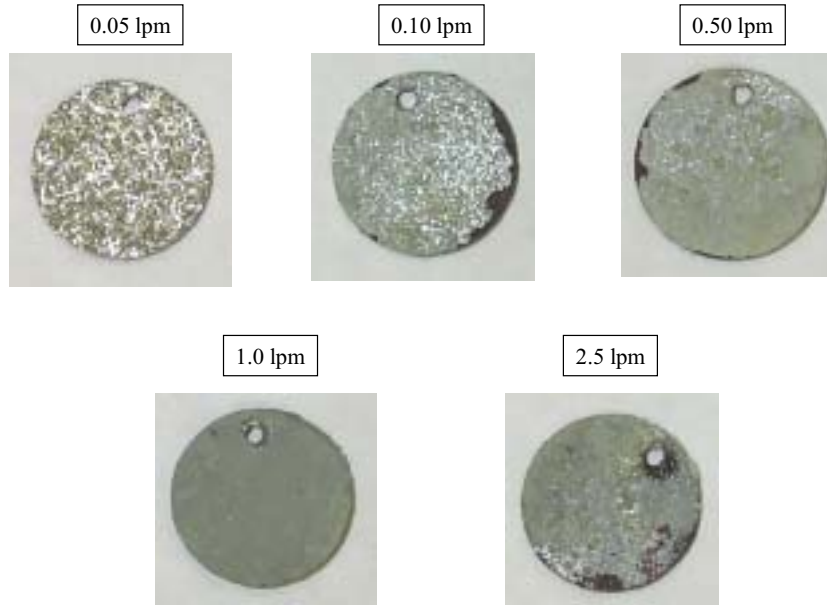
3.2 FEATURES OF OXIDIZED SPECIMENS.

3.2.1 SPECIMEN APPEARANCE. The oxidized specimens of the TZM alloy were photographed at low magnification. Photographs in Figure 2 show specimens tested at various temperatures. Increasing amounts of residual oxide are apparent with increasing temperatures and times for up to 650ºC. Above 700ºC the amount of residual oxide is notably less due to oxide evaporation. The samples exposed at 750 and 800ºC have only a thin darker oxide layer. The oxide on the specimen exposed at 800ºC shows evidence of having been molten. It flowed down the surfaces of the specimen and formed a glob at the bottom. Figure 3 shows photographs of specimens tested at 700ºC under various flow conditions. Data in Table 2 show that there were comparable amounts of molybdenum metal oxidized for all flow conditions, however, significantly greater amounts were volatilized with the higher flow rates. This is reflected in the appearance of the specimens in Figure 3. The specimens with the lower flow rates have crystals developed on the surfaces. Volatilizing molybdenum trioxide species apparently were not swept away but instead re-deposited on preferentially oriented seed crystals located on the specimen surfaces. The specimens with higher flow rates had smoother surfaces and evidence of a darker underlying oxide showing that more of the oxide was volatilized and transported from the specimens in agreement with the ICP-AES measurements.

Oxidized Specimens: Air Flow (1 liter/minute)
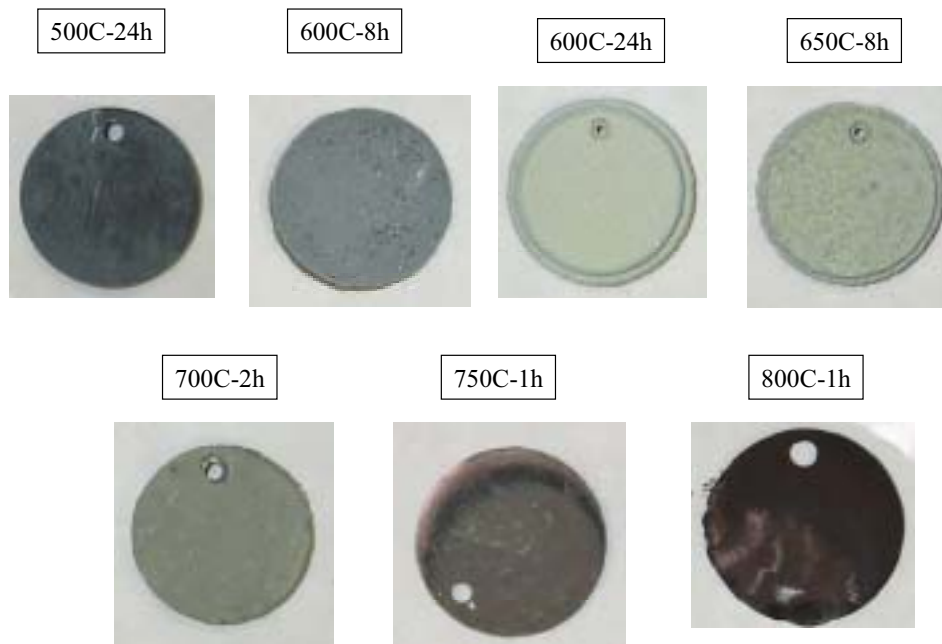
Figure 2. Specimens of TZM alloy oxidized at various temperatures and times in air.

## Effect of Flow Rate on Specimens Oxidized at 700°C

Figure 3. Specimens of TZM alloy tested in air at 700ºC with different flow rates.

3.2.2 MICROSTRUCTURAL OBSERVATIONS OF OXIDIZED SPECIMENS. Oxides formed on specimens were examined with the aid of optical metallography (OM), SEM and XRD. Metallographic cross sections showed both thicknesses of residual oxide layers and profiles of oxide formations on the specimens. A specimen exposed for 24 hours at 500ºC possessed a 0.02-mm thick oxide layer with some irregularly distributed crystals extending to a height of 0.065 mm. A SEM image of surface oxides formed under such conditions is shown in Figure 4. Thicker oxide scales with multiple layers were apparent for higher temperature tests. A dense oxide layer with intrusions into the metal matrix and cusps-like formations on the outer oxide layer is shown for the 24-hour 600ºC test in Figure 5. The outer regions of the oxide also show numerous cracks caused by the growth stresses in the oxide. This suggests that the surface area available for evaporation could be significantly higher than the initial geometric area of the specimen. However, the specimen tested for 8 hours at 600ºC not showing such exaggerated oxide growth had a higher mass flux rate (see Table 2). This indicates that instead of the cracks within the oxide contributing to mobilization, the cusp-like formation may influence the flow pattern and reduce volatilization. The inner oxide layer and the extent of external oxide crystal growth depended upon the flow rate for 700ºC tests. Figure 6 shows that the oxides existed primarily as externally protruding crystals on the specimens with a flow rate of 10 sccm. The specimen with a flow rate of 100 sccm had a more continuous 0.06-mm thick layer but still some external crystal growth (see Figure 7). Figure 8 shows only a 0.04-mm thick oxide layer with no evidence of external crystal growth for the specimen tested with a flow rate of 1000 sccm. This

correlates with the higher mass flux shown for TZM12 in Table 2. The thickness of the residual oxide layer was significantly less for tests at 700ºC and higher. OM and SEM in Figure 9 show that the oxide layer for the 750ºC is about 0.013-mm thick. OM provides evidence of crystal structure in the oxide layer while SEM provides better resolution of the porosity and layers within the oxide. A cross section of the very thin oxide layer (less than 0.01 mm) and glob-like formation near the bottom of the specimen tested at 800ºC is shown in Figure 10. The thin residual oxide layers for the 750ºC and 800ºC tests agree with information in Table 2 showing that most of the oxide that formed on the specimens evaporated.
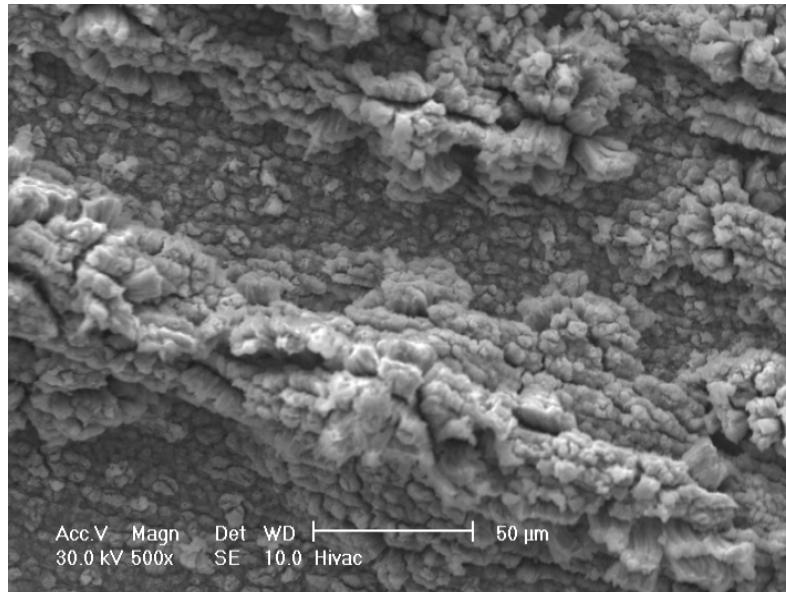
Figure 4. Oxides formed on the surface of specimen oxidized at 500ºC for 24 hours.
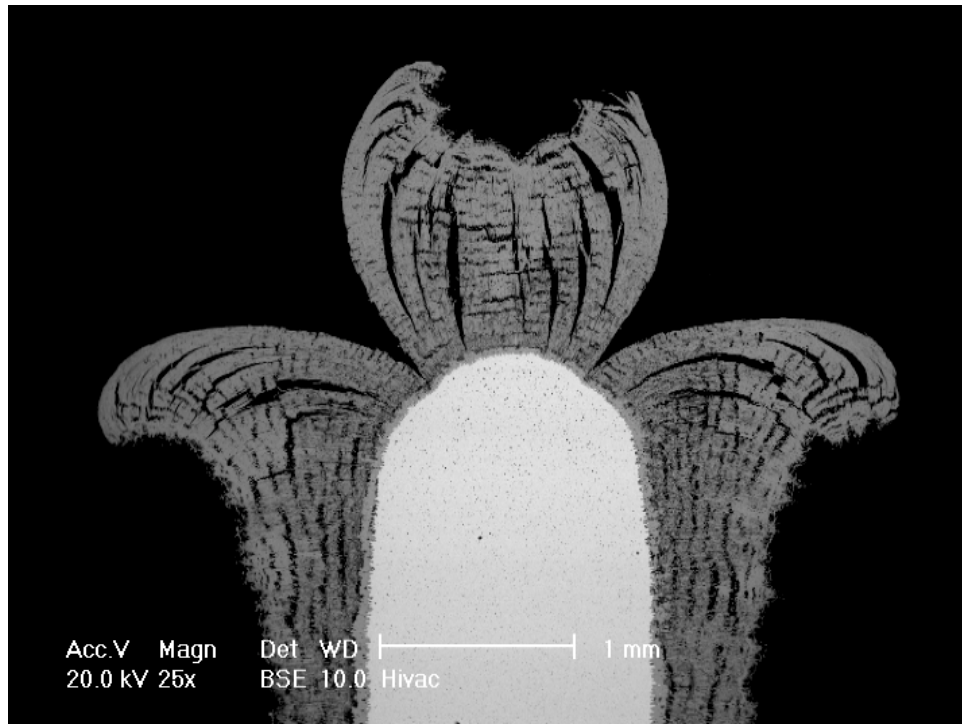
(a)
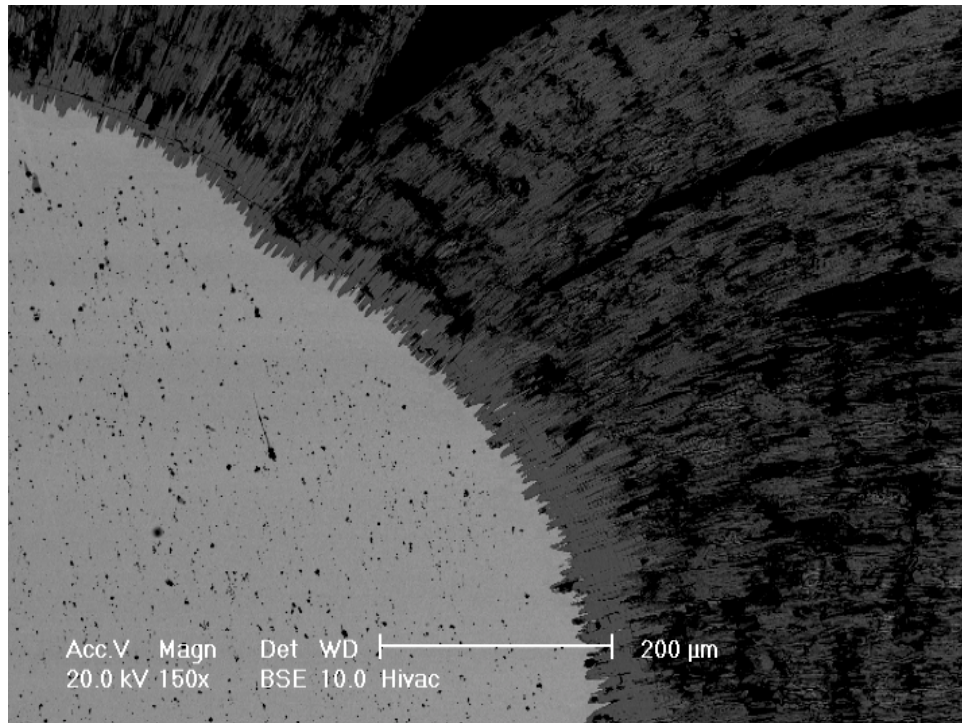
(b)

Figure 5. Cross section of specimen oxidized in air 24 hours at 600ºC. (a) Cusp-like oxide scale showing numerous cracks from growth stresses. (b) Layer at oxide to metal interface.
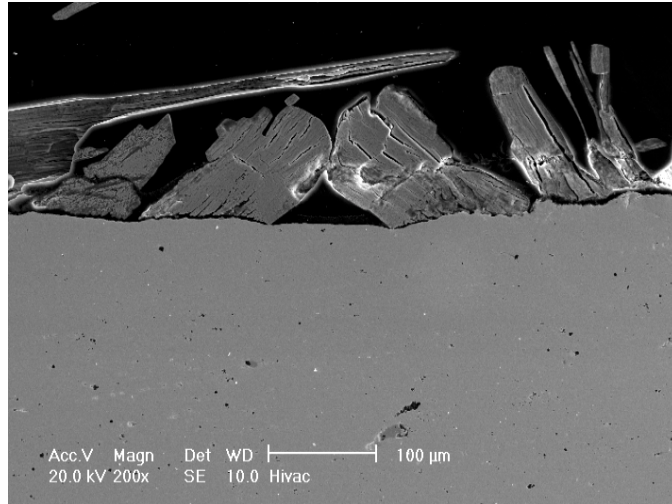
Figure 6. Oxide crystals on specimen oxidized at 700ºC for two hours with 10 sccm flow.
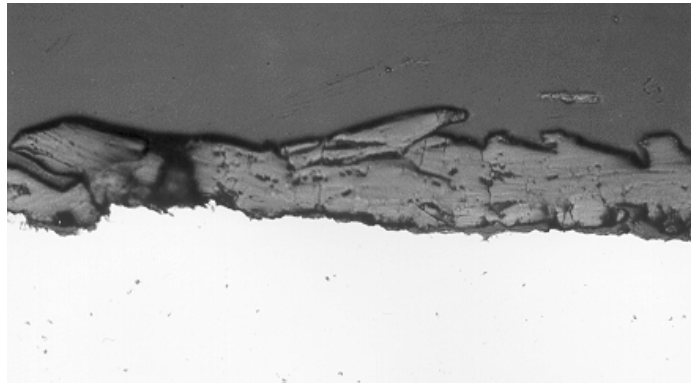
Figure 7. Oxide formed on specimen oxidized at 700ºC for two hours with 100 sccm flow.
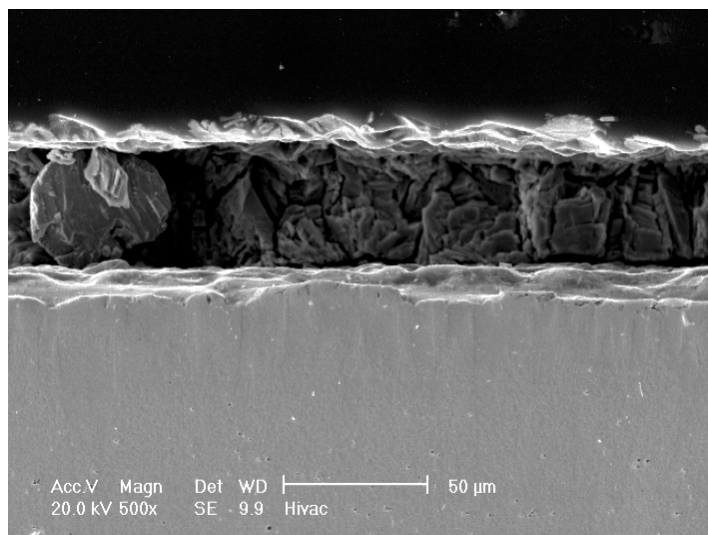
Figure 8. Oxide scale formed on specimen at 700ºC after two hours with 1000 sccm flow.

(a)


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Acc.V   Magn   Det  WD ├──────────┤   20 µm
20.0 kV 1000x   SE   14.9  Hivac
```
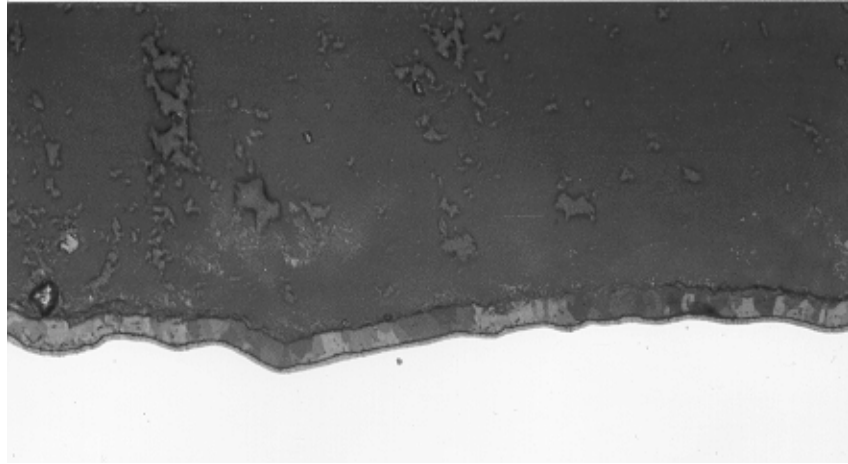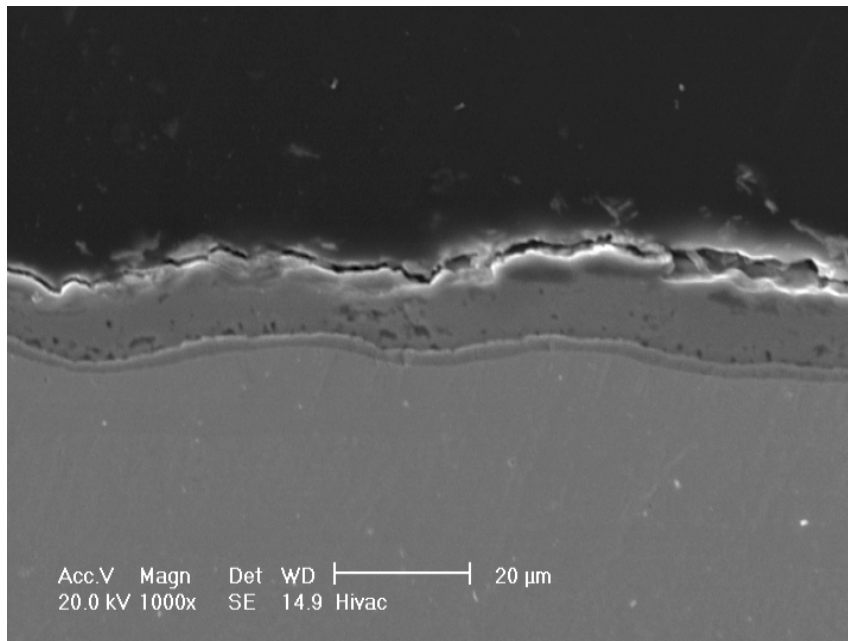
(b)

Figure 9. Residual oxide scale on specimen oxidized at 750ºC for one hour with a flow rate of 1000 sccm. (a) optical micrograph (magnification: 400X). (b) SEM at 1000X.
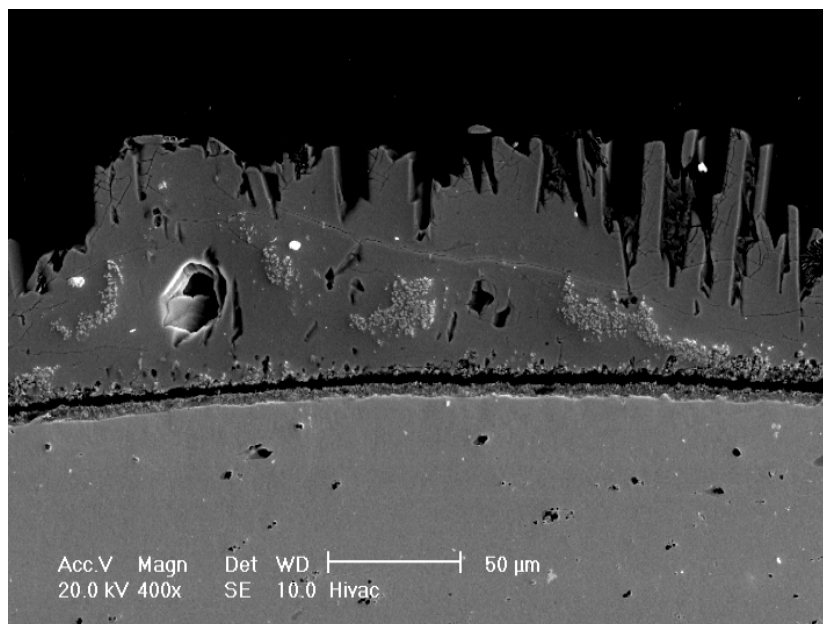
Figure 10. Thin oxide layer and glob-like formation from molten oxide on specimen oxidized at 800ºC for one hour with 1000 sccm flow rate.

3.2.3 PHASE AND CHEMISTRY DETERMINATIONS. Semi-quantitative analyses by energy dispersive x-ray spectroscopy (EDS) were obtained with the SEM from both cross sections and surfaces of oxidized specimens. In nearly all cases, i.e., including analyses near the oxide to metal interface and at outer regions of the oxide the molybdenum to oxygen ratio most closely indicated MoO_2. In addition, many analyses within the base metal indicated high oxygen level, e.g. up to 25 at%. The oxygen content in the base metal does not agree with phase diagram information showing no solubility of oxygen in molybdenum. XRD analyses from the surfaces of specimens tested at 550ºC (TZM16) and 600ºC (TZM7) indicated only MoO_3 as shown by the spectrum in Figure 11. We could not detect any MoO_2. Its presence, however, as a thin oxide layer at the oxide to metal interface as suggested by some authors cannot be discounted since XRD will not detect minor phases. The confirmation of MoO_3 by XRD and oxygen indications from the base metal causes us to question the validity of the EDS analyses. High molybdenum concentrations produce an excitation peak, which is located very near the oxygen peak. Perhaps the EDS software cannot appropriately handle background subtractions or other analytical corrections associated with the excitation peak.
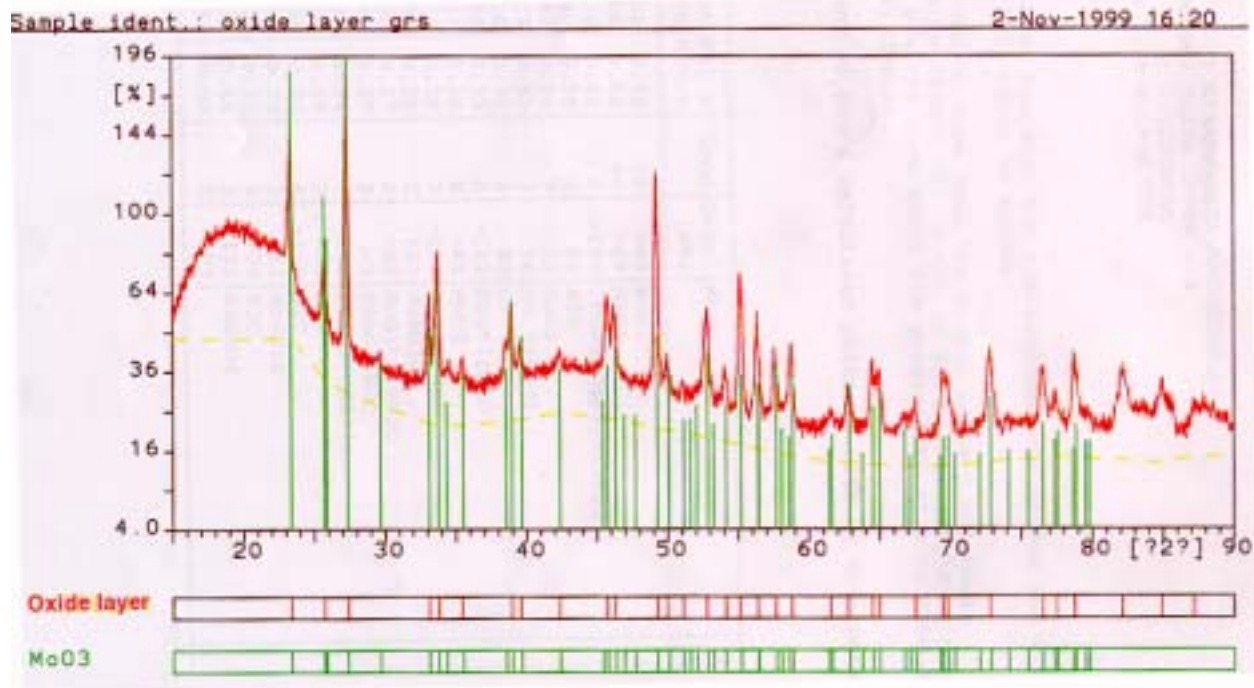
Figure 11. XRD spectrum from specimen oxidized at 600ºC for 24 hours showing only the presence of MoO_3.

3.3 FEATURES OF RE-DEPOSITED OXIDE.

The test components were examined after the test and the type, color and location of re-deposited oxide were recorded. Detailed records for each test are given in Appendix A. Photographs were taken of the collection tube for some of the tests. Some of these tests showed a start, or heavy concentration, of crystal growth at about 12 inches along the collection tube where temperature decreased to around 550 to 600ºC. This region corresponded to the highest concentration of re-deposited molybdenum as determined by ICP-AES. Downstream from this region showing distinctive crystal growth there was often another type of deposit having either a smoky or sometime bluish hue. Sometimes a fairly heavy blue deposit collected at the front end of the quartz wool filter. Figure 12 shows the re-deposited oxides and molybdenum distribution for a test run at 700ºC for two hours with a flow rate of 500 sccm. The SEM micrograph in Figure 12 shows that the product collected in the quartz wool consists of agglomerations of very small particles. Figure 13 shows an extremely heavy crystalline deposit for the 800ºC tests. The deposit bridges and appears to nearly fill the collection tube at this location. Descriptions of deposits, molybdenum deposition profiles, and temperature profiles for all of the other tests are given in Appendices A and C.
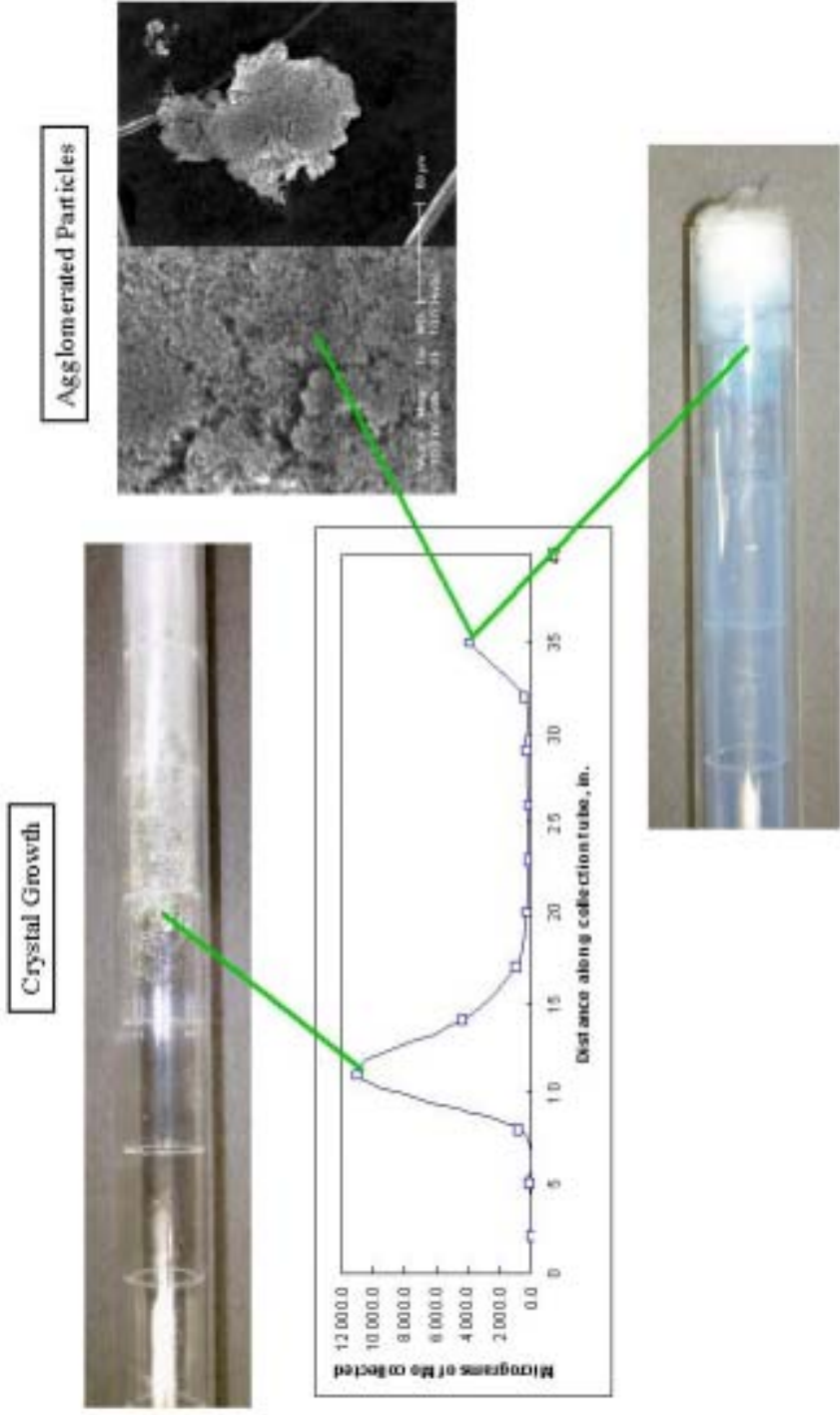
Figure 12. Oxide deposition products from specimen tested in air at 700°C for two hours with a flow rate of 500 sccm.

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Figure 13. Yellow crystalline deposit and white smoky deposit formed in the collection tube for a test run at 800ºC for one hour with a flow rate of 1000 sccm.

4.0 DISCUSSION

4.1 OXIDATION RATE OF TZM ALLOY. We have plotted the recession rates, fractions of "reacted" metal volatilized, and mass flux rates from Table 2 with respect to reciprocal temperature. The plot for the recession rates is shown in Figure 14. The plot shows a good correlation between rates obtained by the (Δm + ICP-AES) and metallographic methods. The plot shows a maximum at 650ºC (the rate at 650ºC is 2 to 3 times higher than at 600 and 700ºC), and then sharply increasing rates from 700 to 800ºC. This trend in oxidation rate is very similar to those observed for the other refractory metals of niobium[2] and tantalum[3]. The irregularities are attributed to various non-stoichiometric phases other than Nb_2O_5 or Ta_2O_5. Speiser and St. Pierre[6] reported that they sometimes observed a thin MoO_2 layer, or an external MoO_3 layer with a thin sub-layer of MoO_2 or other oxides during the oxidation of molybdenum in air between 450 to 770ºC. The other oxides were reported as being non-stoichiometric, i.e., MoO_z with "Z" varying between 2 and 3. The similar characteristics of the refractory metals and the observations above suggest the maximum at 650ºC in Figure 14 is real and likely caused by different types of oxide. Although we have little data to show kinetic behavior, the two tests at 600ºC indicate linear or even slightly accelerating behavior (Table 2).

4.2 VOLATILIZATION PROCESS FROM OXIDIZED TZM ALLOY. The plot of calculated fraction of "oxidized" molybdenum volatilized in Figure 15 show a marked increase above 650ºC. Generally 20 to 33 percent of the oxide that formed volatilized at 700ºC except for the tests with the lowest flow rates of 10 and 50 sccm. Although all 700ºC tests formed similar amounts of oxides (Table 2), only about one-tenth as much of the oxide volatilized at the lower flow rates. Our model subsequently presented shows this is the result of partial saturation of the gas phase with the MoO_3, effectively reducing the volatilization at low flow rates. Most of the oxide was volatilized at 750 and 800ºC. This corresponds with our metallographic observations of only a thin (3 to 10 µm) oxide on these specimens. The high rate of volatilization is apparently linked to the increase in oxidation rate for these two temperatures as shown in Figure 14. Our data agree with that of Gulbransen, et al.[1], where all of the oxygen reacted at 800°C in 76 torr oxygen and formed volatile oxides. The specimen tested at 800°C showed evidence of a molten layer. The sides were smooth and parallel indicative of molten oxide flowing down the specimen. A thicker region at the bottom of the specimen contained
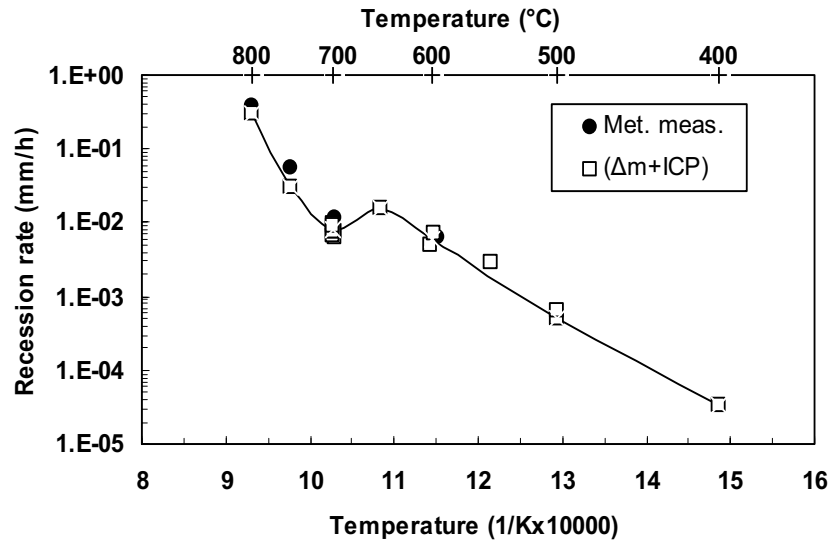
Figure 14. Oxidation rates of TZM alloy as shown the rate of metal reacted (mm/h).
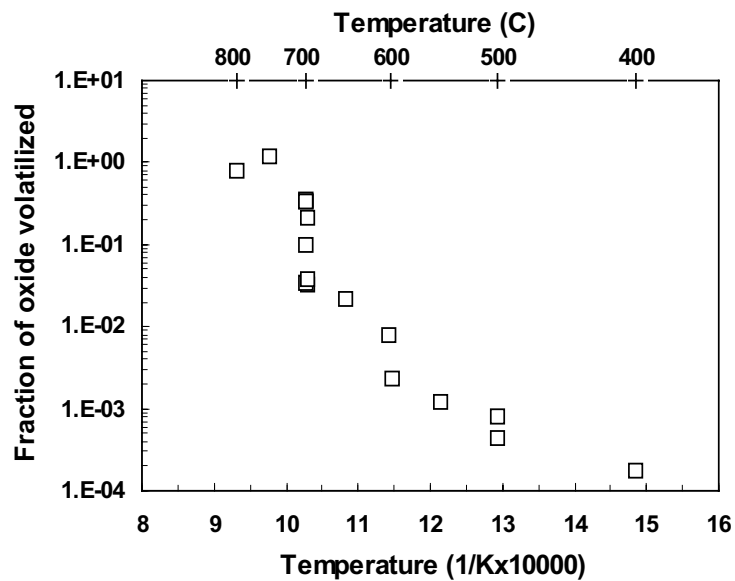
Figure 15. The fraction the MoO_3 volatilized plotted with respect to temperature.

some oxides as revealed in the metallographic cross section. This agrees with temperatures reported for the melting points of MoO_3 (795°C) and a MoO_3-MoO_2 eutectic (778°C)[6]. The higher vapor pressure above the melting point would increase volatilization rates, which in turn would increase oxidation rates by removing the oxide barrier.

Insight into the mechanisms governing oxidation and volatilization processes of molybdenum metal at temperatures from 827 to 1127°C is provided by Olander and Schofill[4]. They believe that very thin coatings of MoO_2 exist even at high temperatures where rapid evaporation occurs by the overall reaction: $Mo(s) + 3/2\ O_2(g) = 1/m\ (MoO_3)_m(g)$. Although the influence of oxygen adsorption on the oxidation behavior of refractory metals has been widely reported,

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Olander, et al.[4] relate the dissociative adsorption of oxygen as the controlling mechanism via the reaction: $O(ads) + MoO_2 \rightarrow MoO_3 (ads)$. The available active sites facilitating the adsorption of oxygen in turn depend upon the anion vacancy concentration in the n-type semi-conductor structure of molybdenum dioxide. The above theory may help explain some of our data at lower temperatures. The volatilization rate for the 24-h test of 0.162 $g/(m^2$-h) was less than the rate of 0.397 $g/(m^2$-h) for the 8-h test at 600ºC. Perhaps the stoichiometry of the oxide changed as the oxide scale grew with time and influenced the number of active sites. It is also possible that as the oxide thickened the flow pattern around the specimen changed decreasing the volatilization rate.

4.3 MODEL OF VOLATILIZATION RATES. We used a vaporization mass transfer model to predict the mobilization of molybdenum from the specimens tested in air. The model based on the experimental geometry, thermal hydraulic conditions and vapor pressures of the volatile trioxides, $(MoO_3)_m$, and hydroxide, $MoO_2(OH)_2$, was used to predict the mobilization mass flux of molybdenum , J, as expressed by Eq.(2).

$$J = k(p^{Mo}- p^{bulk})/RT \qquad (2)$$

where

  k = mass transfer coefficient (m/s),
  p^{Mo} = partial pressure of volatile molybdenum species at surface of the specimen,
  p^{bulk} = partial pressure of volatile molybdenum species in bulk steam flow.

Since the flow in the experiment is laminar, the mass transfer coefficient is given by [8]:

$$k = 1.86[Re\ Sc(l/d)]^{1/3} D/d \qquad (3)$$

D is the diffusion coefficient of the volatile molybdenum species in air based on classic Chapman-Eskong theory [8] and Re is the Reynolds number, Sc is the Schmidt number, and l and d are the length and diameter of the furnace. Surface to bulk pressures of the volatile molybdenum species are corrected for the partial saturation of the species in the bulk flow by Eq. (4), where Q is the volumetric flow rate of air (m^3/s).

$$(p^{bulk}/p^{Mo}) = [1 - exp(-hA/Q)] \qquad (4)$$

The partial pressure of $(MoO_3)_m$ is determined as an effective value for the various polymeric forms of (MoO_3) existing at different temperatures over pure solid or molten MoO_3. Vapor pressures for the various polymer species were obtained from the HSC thermochemical computer code[9]. Correlations [10] for the effective average polymer number (m) in $(MoO_3)_m$ were also determined. The primary species within our temperatures of interest are $(MoO_3)_3$, $(MoO_3)_4$, and $(MoO_3)_5$. Average polymer numbers above solid (MoO_3) are given by Eq. (5).

$$m = 6.785-0.013193T+1.432x10^{-5}T^2 – 4.4235x10^{-9}T^3 \qquad (5)$$

The total vapor pressures of these average effective polymers over the solid phase up to the melting point as derived from the HSC thermochemical code are shown by Eq. (6).

$$log_{10}P(Torr) = 8,599.81/T – 186.495 + 63.1654\ log_{10}T –0.0078447T – 4,154,834/T^2 \qquad (6)$$

Vapor pressures of the hydroxide formed by the reaction: $MoO_{3(s)} + H_2O_{(g)} = MoO_2(OH)_{2(g)}$, have been experimentally determined by Speiser and St. Pierre.[6] They reported an equilibrium constant, K_m, for the reaction as expressed by $K_m = P_{hydroxide}/P_{water}$, to be given by Eq. (7).

$$\log K_m = -7731/T + 5.45 \text{ (for } T = 873 \text{ to } 963 \text{ K)} \qquad (7)$$

We obtained vapor pressures for the hydroxide species by using the relationship for K_m, the saturation pressure for water vapor in ambient temperature air of 17.55 Torr (2340 Pa), and the relative humidity. We used a parametric analysis to show vapor pressures of the hydroxide species at various relative humidities. The humidity at the INEEL is typically around 40 percent. Although we dried the air prior to introduction into the test system we postulate that there was some backflow and/or leakage of ambient air into the test system. We show comparisons of the vapor pressures of $(MoO_3)_m$ in dry air and $MoO_2(OH)_{2(g)}$ in environments with the various relative humidities, including a postulated test condition with a ten percent ingress, in Figure 16. The plots show that the vapor pressure of $(MoO_3)_m$ in air and $MoO_2(OH)_{2(g)}$ in the postulated experimental condition are about equivalent at 550°C. In higher relative humidities, e.g., 80 to 100 percent, the temperature at which the vapor pressures of the two different species become equivalent would increase to about 650°C.
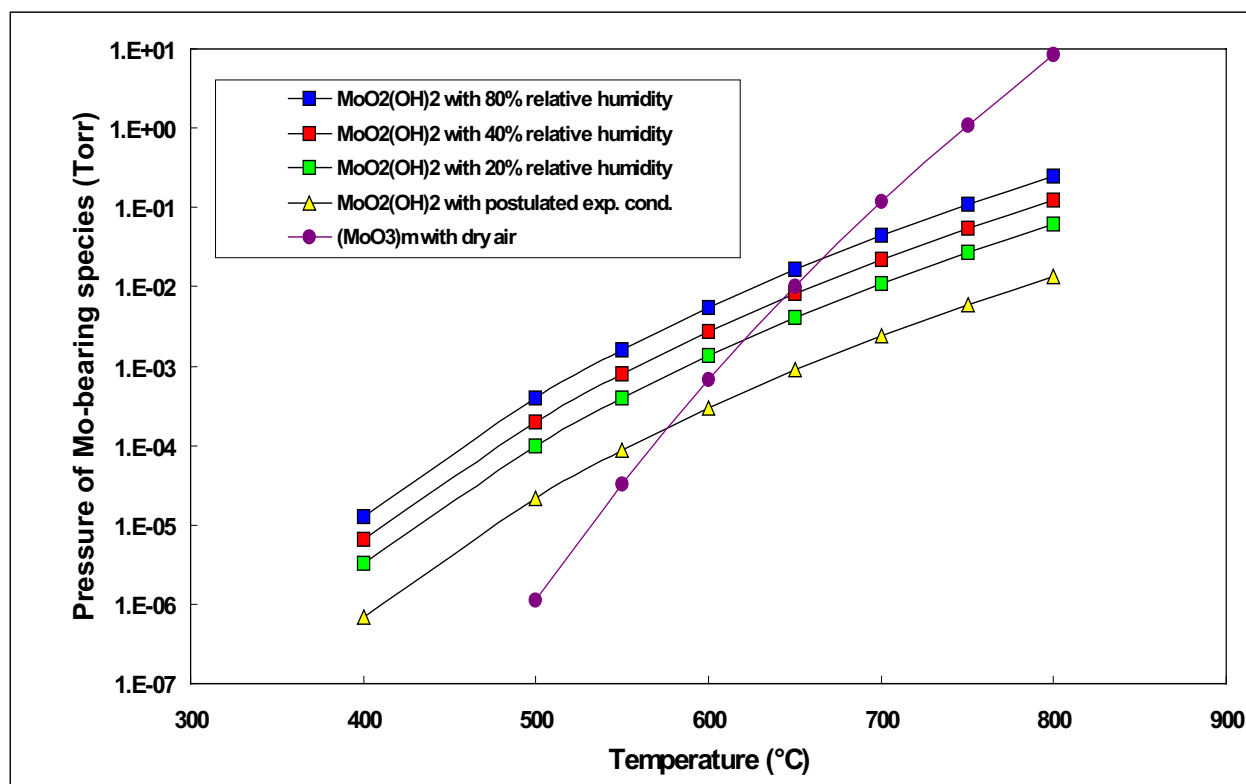
Figure 16. Vapor pressures of molybdenum trioxide and hydroxide species at various relative humidities and temperatures.

We calculated contributions to the molybdenum mass flux from both the trioxide and hydroxide species using Eq. (2) through Eq. (7). Comparisons of predicted total mass flux of molybdenum with experimental measurements for the various times, temperatures, and flow rates are given in Table 2. The predictions for tests with a flow rate of one liter per minute are plotted along with the experimental data in Figure 17. The calculations show that below 550°C contributions from $MoO_2(OH)_{2(g)}$ dominate the volatilization process, whereas $(MoO_3)_m$ is the major contributor above this temperature. The model incorporating both species agrees quite well with experimentally derived mass flux measurements over the entire temperature range. Simnad and Spilners [5] showed a very similar trend for molybdenum metal oxidized between 500 and 770ºC including an inflection in volatilization rate at 600ºC. They reported activation energies of 53.0 kcal/mole and 89.6 kcal/mole below and above 650ºC, respectively. We obtained activation energies of 41.4 kcal/mole between 400 and 500°C and 85.3 kcal/mole between 600 and 800°C, respectively. We believe this supports the proposal that the hydroxide and trioxide species, respectively, are the active mechanisms in the two different temperature regions.

The trend of lower mass flux with lower flow rates at 700°C (shown in Table 2) is supported by our predictions using Eq. (3). The measured values, however, are still somewhat higher than predicted values for the lowest flow rates. Protruding crystals were observed growing from specimens tested in the low flow rates as evident in Figure 3 and Figure 6. We would expect environments with higher partial saturation to be more conducive to grow such crystals from preferential sites. Some of these crystals were quite needle, or ribbon-like, and would easily break off. In fact, ribbon and needle-like crystals were observed in the reaction tubes for 700ºC tests with 10 and 50 sccm flow rates and molybdenum measurements were higher compared to the other components (see Appendices A and C). This supports the suggestion that the high measurements for mobilized material compared to predicted values result from spalled crystals.
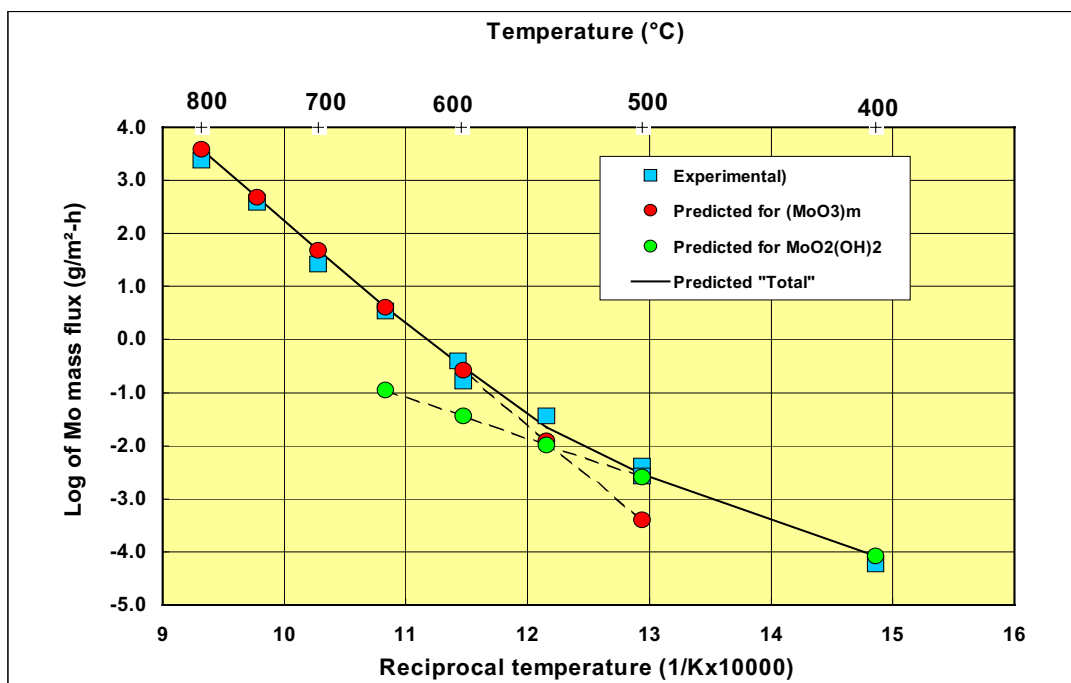
Figure 17. Mass flux of molybdenum showing experimental and predictions from the $(MoO_3)_m$ and $MoO_2(OH)_{2(g)}$ vapor species at various temperatures.

4.4 MODEL FOR RE-DEPOSITION OF MOBILIZED MOLYBDENUM.

In this section, we present a simple condensation model that predicts the condensation profiles in the downstream components in the experiments. The $(MoO_3)_m$ vapors are assumed to be the primary species vaporized from the sample particularly at 600°C and higher as shown by the preceding section. For a given flow rate in the experiment, a concentration of $(MoO_3)_m$ in the gas phase is established by Eq. (8):

$$C_{gas} = (\Gamma * A)/Q \qquad (8)$$

where,

C_{gas} = concentration of $(MoO_3)_m$ in gas (kg/m^3),
Γ = measured mass flux from surface $(kg/m^2\text{-s})$,
A = initial surface area of sample (m^2), and
Q = volumetric flow rate of air (m^3/s).

As a packet of gas travels down the tube, it cools and the $(MoO_3)_m$ becomes supersaturated. The saturation ratio expressed by Eq. (9) is that defined by Friedlander[11]:

$$S = C_{gas}(R*T)/(p_{sat}(T)* MW) \qquad (9)$$

where,

S = saturation ratio,
MW = molecular weight of $(MoO_3)_m$ (kg/kgmole),
R = gas constant $(Pa\text{-}m^3/kgmole\text{-}K)$,
T = temperature (K), and
$p_{sat}(T)$ = vapor pressure of $(MoO_3)_m$ at temperature T

The vapor pressure of $(MoO_3)_m$ was obtained from the HSC thermochemical computer code[9].

Condensation to the wall will occur when the concentration in the gas exceeds the equilibrium vapor concentration at the given temperature. The rate of condensation given by Eq. (10) in that presented in Ref. [8]:

$$J_{cond} = k (C_{gas} - C_{equ}) \qquad (10)$$

where,

J_{cond} = condensation mass flux $(kg/m^2\text{-s})$,
k = mass transfer coefficient (m/s), and
C_{equ} = equilibrium vapor concentration (kg/m^3).

Using the ideal gas law to relate the equilibrium vapor concentration to the vapor pressure and the definition of supersaturation ratio, this relationship can be written as Eq. (11).

$$J_{cond} = k \, A_s \, MW \, p_{sat}(T) \, (S-1) \, /(R*T) \qquad (11)$$

The mass transfer coefficient expressed as k in Eq. (12) is given by Bird[8]:

$$k = 1.86 * (D/d) * [Re *Sc \, (L/d)]^{0.333} \qquad (12)$$

where

Re = Reynolds number of the flow,
Sc = Schmidt number,
D = diffusion coefficient of $(MoO_3)_m$ in air given by the Chapman-Eskong theory which is also presented in Bird[8],
d = diameter of tube, and
L = length of tube from sample to location of interest along the tube.

Thus, the mass balance equations that describe the mass of $(MoO_3)_m$ condensed and in the vapor phase are:

$$dC_{gas}/dt = -J_{cond}*A_{wall}/V \qquad (13)$$

$$dC_{cond}/dt = J_{cond}*A_{wall}/V \qquad (14)$$

where

A_{wall}/V = surface to volume ratio of pipe equals (4/d).

Converting the concentration in the gas to supersaturation yields Eq. (15)

$$dS/dt = (kA_{wall}/V)*(S-1) \qquad (15)$$

Integration yields Eq. (16):

$$S(t) = 1- (S_o-1)exp(-kA_{wall}*t/V) = 1- (S_o-1)exp(-4kt/d), \qquad (16)$$

where S_o is the initial supersaturation at the start of the collection tube. The collection tube is then discretized into an equal number of uniform cells (usually 100 over the length of the collection tube each 1 cm each). In this case S_o is the initial supersaturation as the packet of gas enters the cell and S(t) is set to the final value as it exits the 1-cm long cell. This final value is given by Eq. (17):

$$S_{final}= 1- (S_o-1)exp(-4k\Delta t/d) = 1- (S_o-1)exp(-4kV_i/Q*d), \qquad (17)$$

where

Δt = time for the packet to transit the cell,
V_i = volume of segment i, and
Q = volumetric flow rate.

And the mass condensed in a given cell is given by Eq. (18):

$$[S_{final}-S_o]*p_{sat}(T)*MW/[R*T] \qquad (18)$$

The final supersaturation exiting a cell is the initial supersaturation entering the next cell in the collection tube. Temperatures in the tube for each test were linearly fit (piecewise into two or three pieces in some cases to get the best fit) and then the linear fit was used in the calculations. The $(MoO_3)_m$ deposition was converted to molybdenum by multiplying by the ratio of the molecular weights. The measured and calculated depositions were converted to deposition per unit length given the limited number of measurements and the large variation in the calculated deposition along the tube.

The results for various cases of temperatures, flow rates and times as listed in Table 3 are shown in Figures 18(a) through Figure 18(h).

Table 3. Cases calculated by deposition model.

Case Number	Temperature (°C)	Flow rate of Air (slpm)	Duration (hr)
1	800	1	1
2	700	0.5	2
3	700	2.5	2
4	700	1	2
5	650	1	8
6	600	1	8
7	600	1	24
8	500	1	24

The results show that the start of condensation is very well predicted with the model for all the cases. The peak deposition is well predicted in the majority of cases (e.g., Cases 2, 3, 5 and 7) and in the worst cases overpredicts the peak deposition by between 50 and 100%. The deposit model predicts a broad tail of deposition beyond the peak deposition location. This broad tail in most cases overpredicts the observed deposition in the tube. Deviations in the peak heights and the broad tail distribution can be explained by deposition occurring at preferential macroscopic sites provided by growing crystals. For example, this is most apparent for the 800°C test that had crystals bridging the I.D. and nearly plug the tube. Deposition on these crystals would substantially deplete the $(MoO_3)_m$ concentration causing lower than predicted deposition in downstream locations. The experimental data also showed significant molybdenum deposition near the end of the tube and in the glass wool. This deposition was bluish colored and SEM showed that the deposition in the quartz wool consisted of agglomerated small particles. We might expect particle nucleation and transport upon cooling the supersaturated gas. Such aerosol formation was not considered in the model. Had nucleation been incorporated into the model, some of the condensation would have occurred onto those nucleated particles decreasing the

calculated condensation onto the tube in regions where supersaturation is high enough to support both nucleation and condensation onto both particle and walls. The role of $MoO_2(OH)_{2(g)}$ was also not considered. Decomposition of this vapor at lower temperatures may have produced the oxide with the bluish color. This latter mechanism also provides an explanation for the large discrepancy for the 500°C test, i.e., Case 8. Most of the molybdenum is shown to be volatilized as $MoO_2(OH)_{2(g)}$ in Section 3.3 rather than $(MoO_3)_m$. Analyses from the ICP-AES showed that most of the re-deposited oxide was in the reaction chamber as reported in Appendix C. In conclusion, given the simplicity of the model, it does a reasonable job at broadly describing deposition behavior at 600°C and higher where volatilization is dominated by $(MoO_3)_m$.
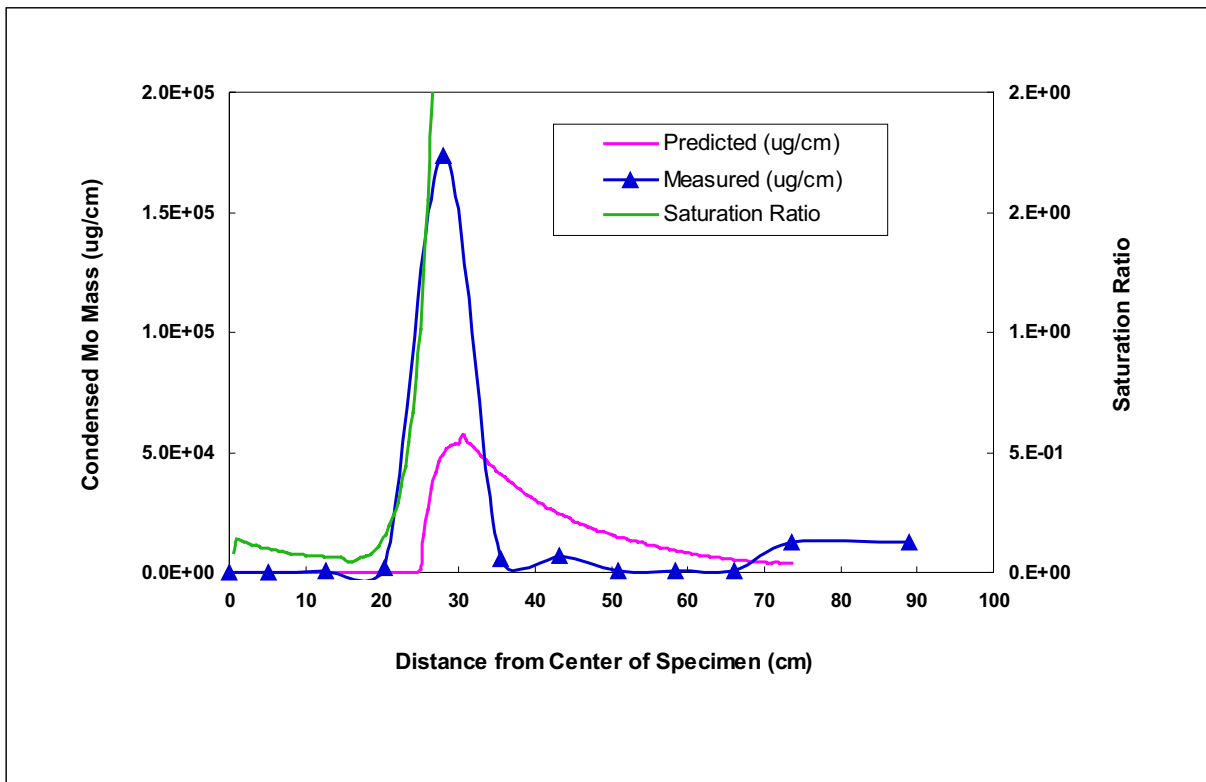
Figure 18(a). Case 1—Test at 800°C with one liter per minute flow rate for one hour.
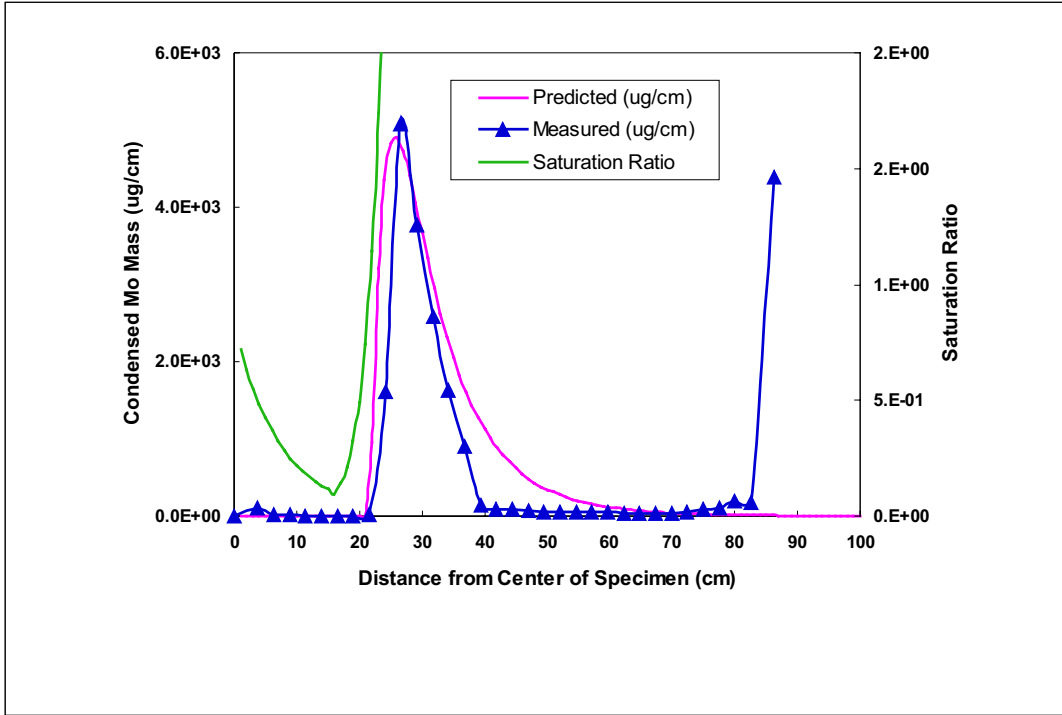
Figure 18(b). Case 2 – Test at 700°C with 0.5 liter per minute flow for two hours.
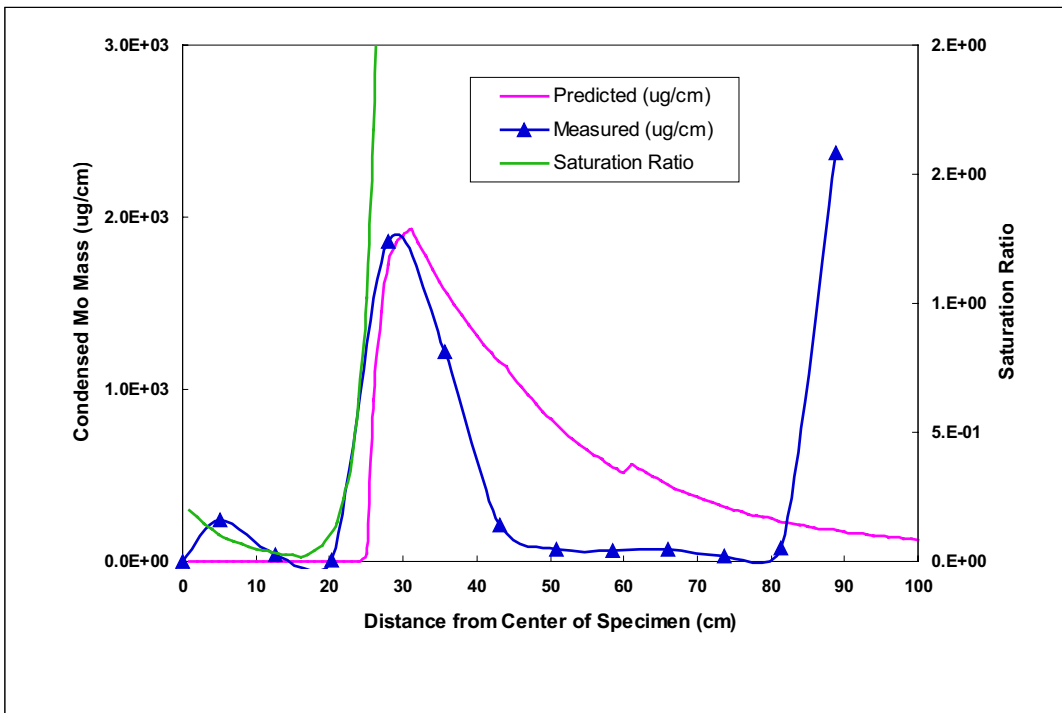
Figure 18(c). Case 3 – Test at 700°C with 2.5 liter per minute flow for two hours.
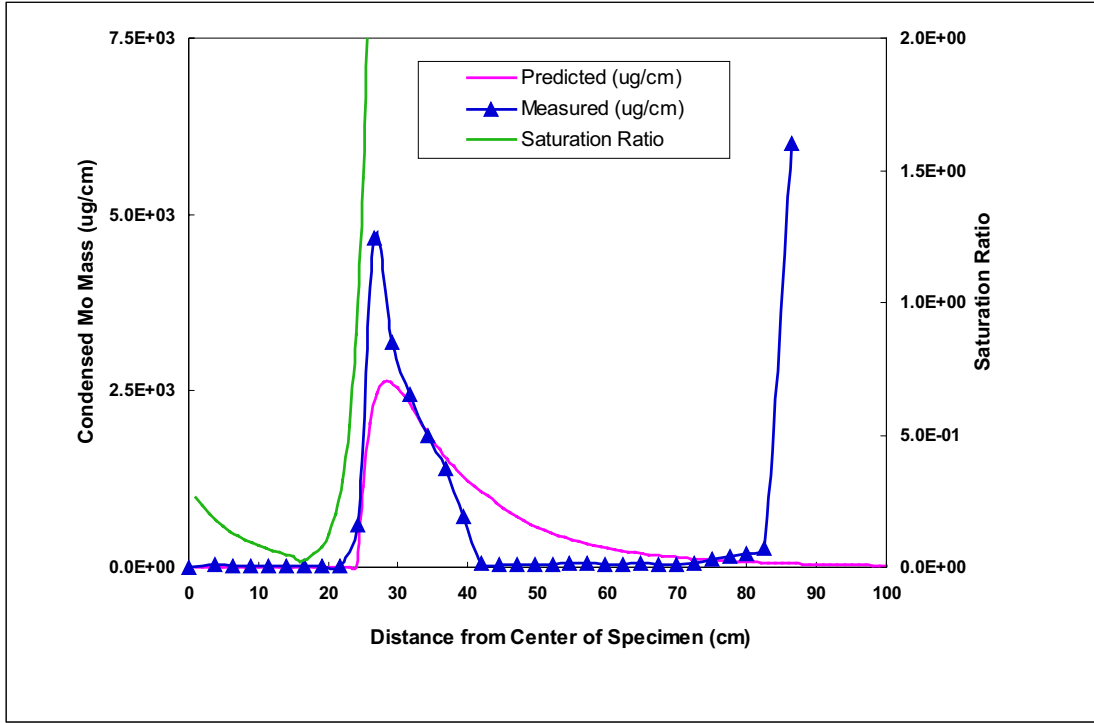
Figure 18(d). Case 4 – Test at 700°C with one liter per minute flow for two hours.
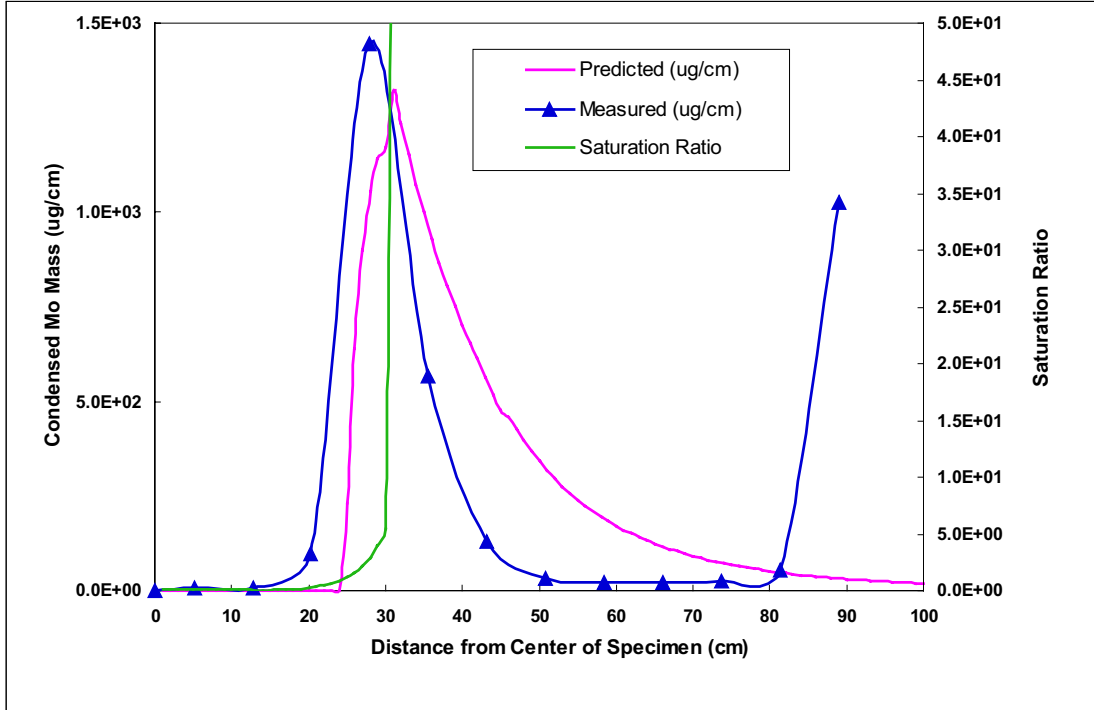
Figure 18(e). Case 5 – Test at 650°C with one liter per minute flow rate for 8 hours.
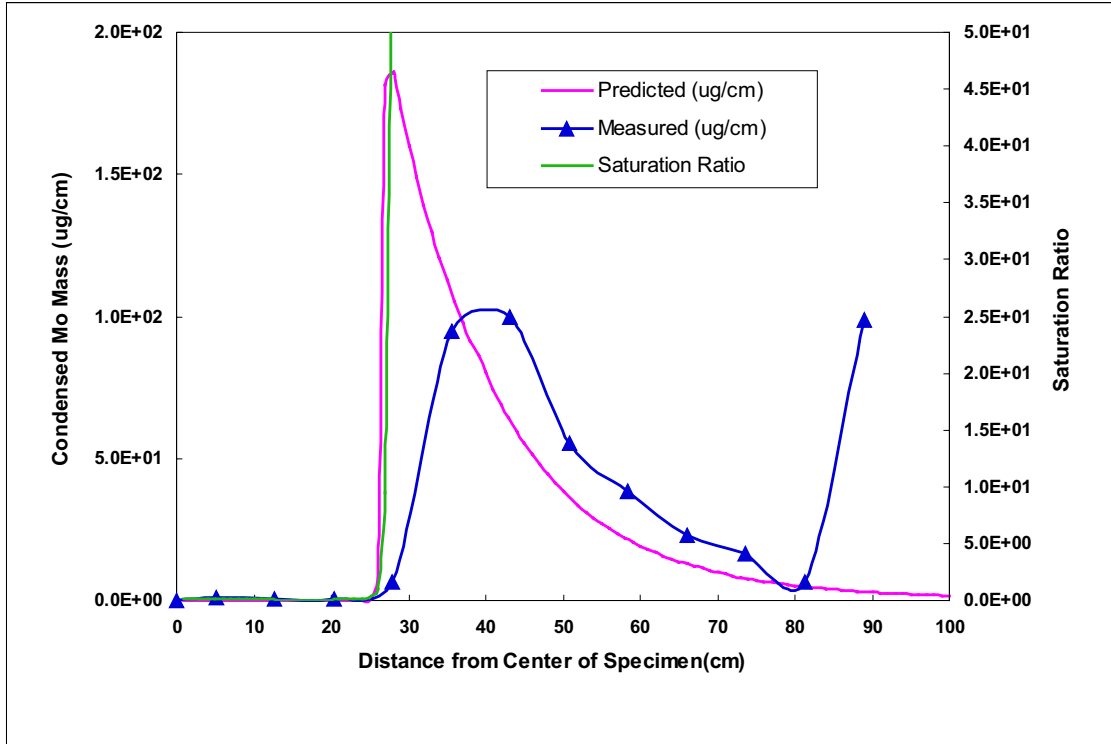
Figure 18(f). Case 6 – Test at 600°C with one liter per minute flow for 8 hours.
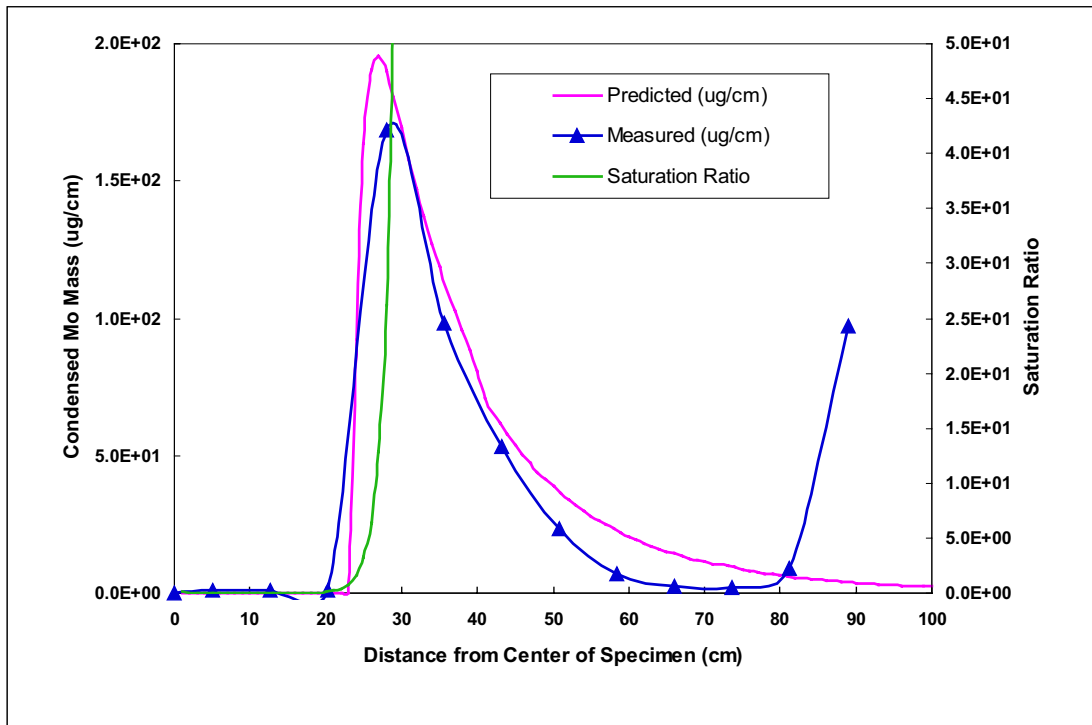
Figure 18(g). Case 7 – Test at 600°C with one liter per minute flow rate for 24 hours.
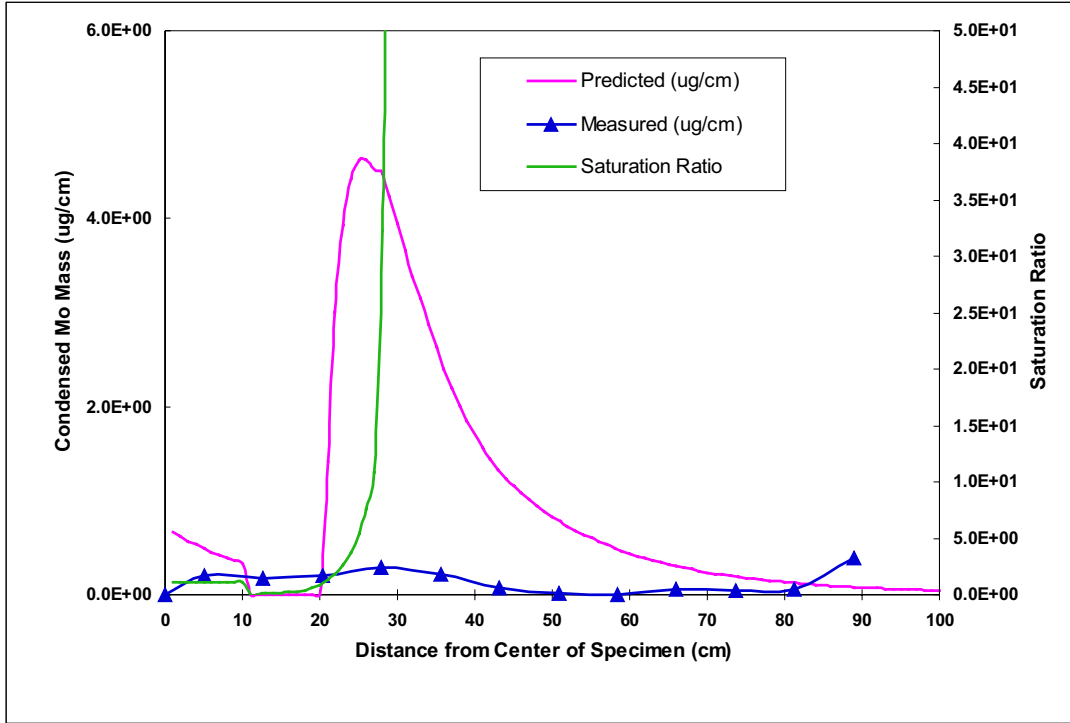
Figure 18(h). Case 8 – Test at 500°C with one liter per minute flow rate for 24 hours.

4.5 MOBILITY-BASED DOSE DERIVED FROM OXIDATION DRIVEN VOLATILITY

We can use the oxidation-driven mobilization data to calculate a mobility-based dose. This dose can be used as a metric to compare with other fusion materials or with appropriate confinement factors used in a safety analysis of a conceptual fusion design.

The fractional rate of mobilization of a particular element in an alloy at a given temperature is calculated using the Eq. (19):[12]

$$\dot{MF} = \frac{\Gamma}{WP \times \rho \times \delta} \qquad (19)$$

where $\dot{MF}$ is the mobilization fraction per unit time, Γ is the cumulative mass flux of the element (kg/m^2-s) based on the test data, WP is the weight fraction of the element in the material, ρ is the density of the material (kg/m^3), and δ is the thickness of the component (m) that is being analyzed.

The oxidation-driven mobility-based dose rate is then given by the following equation:

$$DoseRate = [RI \times DI] \times \dot{MF} \times A_{FW} \times \rho \times \delta \qquad (20)$$

where,

RI = radioactive inventory of isotope per unit mass of material (Bq/kg) in the component,
DI = dose impact of isotope (Sv/Bq),
A_{FW} = surface area of first wall (m^2).

The inner summation is over all isotopes produced and the outer summation is over all of the elements in the alloy.

Thus, the dose rate is simply given by:

$$DoseRate = \frac{[\sum RI \times DI] \times \Gamma \times A_{FW}}{WP} \qquad (21)$$

For the TZM alloy, an activation calculation for the first wall based on the EVOLVE design assuming TZM as the first wall structural material was used.[13] The dose impact of each isotope was calculated for both ground level and elevated releases using conservative meteorology (class F and 1 m/s wind speed as needed in a traditional "design basis" safety analysis), and using best estimate meteorology (class D and 4 m/s wind speed as needed in a no-evacuation assessment).[14] A one-kilometer site boundary and the regulatory-accepted Pasquill-Gifford dispersion set were used in the evaluation. All results are early doses (7-day exposure) to the maximum exposed individual (at the site boundary for the ground level cases and where the plume touches the ground for the elevated releases). These data are reproduced in Table 4.

Table 4. Radioactive inventory and Dose Impact of Mo and Tc Isotopes.

Isotope	Radioactive Inventory (Ci/g)	Dose Impact D and 4 m/s Ground level (Sv/TBq)	Dose Impact D and 4 m/s Elevated (Sv/TBq)	Dose Impact F and 1 m/s Ground level (Sv/TBq)
Mo 93	2.60E-03	4.10E-05	3.72E-06	4.26E-04
Mo 93m	1.35E-02	5.43E-07	4.89E-08	5.53E-06
Mo 99	2.13E-01	6.98E-06	6.38E-07	7.23E-05
Mo101	1.83E-01	5.45E-08	7.01E-09	2.69E-07
Tc 98	1.81E-07	4.66E-05	4.27E-06	4.83E-04
Tc 99	4.88E-05	8.01E-06	7.28E-07	8.34E-05
Tc 99m	2.80E+00	1.81E-07	1.97E-08	1.73E-06
Tc101	1.83E-01	7.01E-09	8.44E-10	3.20E-08
All Mo isotope contribution to dose (Sv/g)*		5.97E-08	5.46E-09	6.16E-07
All Tc isotope contribution to dose (Sv/g)*		1.88E-08	2.05E-09	1.79E-07

* Calculated by summing the product of the radioactive inventory of an isotope and its dose impact.

The mobilty-based dose rate for Mo from the TZM alloy is based on the cumulative maximum[1] method from data presented in Table 2 in Section 3.1. Mobilization of the Tc isotopes produced by activation of the Mo is assumed to follow that of the Mo at all temperatures because of the very high vapor pressure of the technetium oxides.[15] The other elements in the TZM alloy did not mobilize in the experiments because of their extremely low volatility and are not included in the evaluation. The results, based on a 500 m^2 first wall with no radiological confinement, are shown in Table 5. They are compared graphically to the mobilization from tungsten alloy for two of the dose cases studied in Figures 19 and 20.

Table 5. Oxidation-driven Mobilization-base Dose Rates as a Function of Temperature Under Different Exposure Conditions.

Temperature (°C)	Oxidation-driven Mobilization-based Dose Rate		
	D&4 m/s Ground (Sv/hr)	D&4 m/s Elevated (Sv/hr)	F&1 m/s Ground (Sv/hr)
400	2.37E-09	2.27E-10	2.40E-08
500	1.61E-07	1.54E-08	1.63E-06
600	1.57E-05	1.50E-06	1.59E-04
650	1.35E-04	1.29E-05	1.36E-03
700	1.40E-03	1.34E-04	1.42E-02
750	1.50E-02	1.43E-03	1.52E-01
800	9.51E-02	9.10E-03	9.64E-01

The results show that the mobilization dose for TZM alloy is about two orders of magnitude lower than that for tungsten below about 600°C, and decreases to about one order of magnitude up to 800°C. Above 800°C, the Mo in the TZM that converts to molybdenum trioxide melts making measurements very difficult. The results can also be used to determine the degree of radiological confinement needed in accidents involving oxidation driven mobilization of material from the first wall. In a decay heat transient involving air ingress that reaches 750°C for five days, the oxidation driven mobilization dose would be approximately 2 Sv (see Figure 20). Thus, to meet a 10 mSv no-evacuation dose would require the radiological confinement to reduce this source term by about a factor of 200. After five days at 750°C, approximately 70 percent of the affected first wall area would have been mobilized. Transients that result in shorter times at lower temperatures would require less radiological confinement to meet the no-evacuation goal. If temperatures always remained below 500°C in a transient then this would not be a significant contribution to the off-site radiological source term in an accident.

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[1] The concept of cumulative maximum is a conservative formulation used for safety assessments. The cumulative maximum mobility at temperature X is the maximum mobility measured at all temperatures up to and including X. Thus, if the maximum mobility measured at 600°C is higher than the value measured at 700°C, the value from the 600°C test is used in place of the value at 700°C.
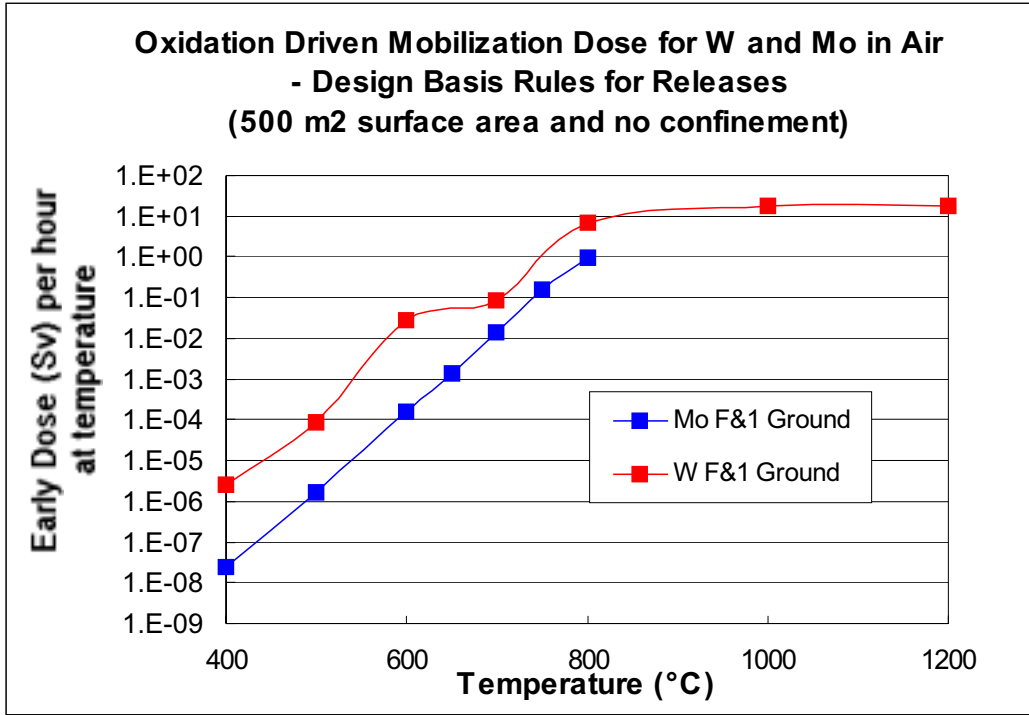
Figure 19. Oxidation driven mobilization doses for W and Mo in air. The design basis for release are 500 m^2 surface area with no confinement.
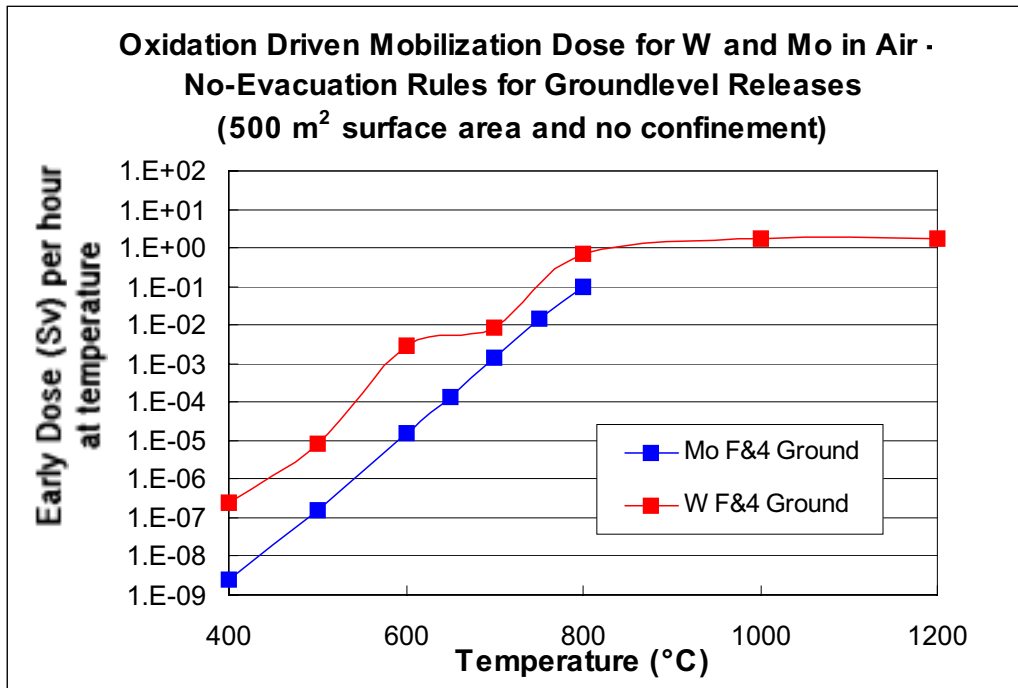
Figure 20. Oxidation driven mobilization doses for W and Mo in air. Releases are for 500 m^2 surface area with no confinement and class D and 4 m/s wind speed for no-evacuation criteria.

5. CONCLUSIONS

We have observed good correlation between experimental volatilization rates and those predicted by our mass transport model. The volatilization process occurs by two different mechanisms at low and high temperatures. At low temperatures, e.g., below 550°C volatilization is dominated by $MoO_2(OH)_{2(g)}$ formed from even small concentrations of water vapor. Above this temperature volatilization occurs predominately by the volatile (MoO_3) polymeric species. Our model accounted for both of these mechanisms in the two different temperature regions. It can therefore be used to accurately predict molybdenum mobilization for different climatic conditions, or ambient and humidity conditions of the air, involved in the ingress accident. The model also proved capable of accounting for flow rates which affected near-surface partial saturation of the volatilizing species and reduced mobilization.

The oxidation rate (mm/h) as indicated by recession, i.e., the amount of metal reacted, shows an increasing trend to 650ºC, then a slight decrease, and then sharply higher rates due to high volatilization. This trend with a maximum near 650ºC simulates that observed with other refractory metals and has been associated with different oxide types. The higher oxidation rates at higher temperature, e.g., 700 to 800°C are due to the very high volatilization rates of the molybdenum trioxide.

 Our model for the re-deposition of volatilized molybdenum species of (MoO_3) accurately predicted the downstream peak-deposition locations. The model provided best results for the higher temperatures where volatilization by the various polymeric forms of (MoO_3) is dominant. Differences between predicted and experimental peak heights and subsequent deposition profiles resulted when extensive crystal growth at the peak-deposition locations provided greatly increased surface area for re-deposition to occur.

Mobility-based dose calculations derived from oxidation-driven mobilization data showed TZM alloy to have lower radiological dose impact than tungsten at comparable temperatures and conditions. Dose levels from TZM alloy were two orders of magnitude lower below 600°C and about one order of magnitude lower at 800°C.

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APPENDIX A

**DESCRIPTIONS OF MOLYBDENUM OXIDE
PRODUCTS DEPOSITED IN
QUARTZWARE TEST COMPONENTS**

Description of Oxidation Products from TZM 14

Test temperature (ºC) 400   Time (h) 24   Flow rate (lpm) 1
Specimen mass (g): Initial 13.6641   Final 13.6684   Mass gain 0.0043

Reaction chamber
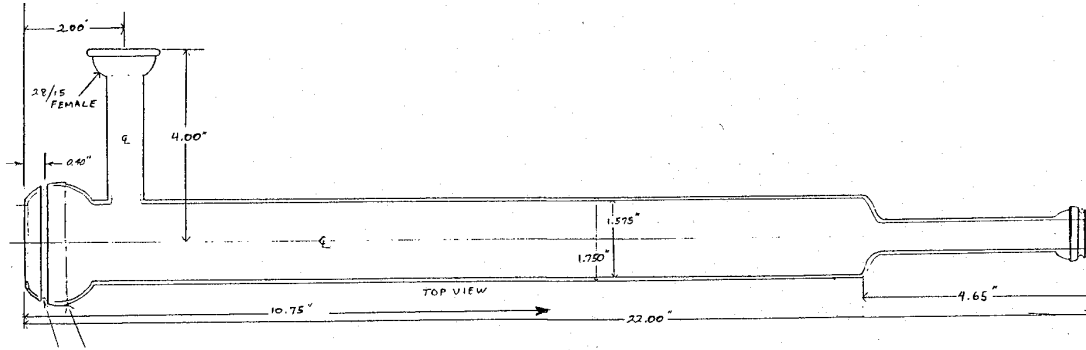
Description of oxidation products	Location (inches from left)	Temp. (ºC)

1. Clear, there is no evidence of any reaction product in test chamber.

2.

3.

4.

Deposition chamber
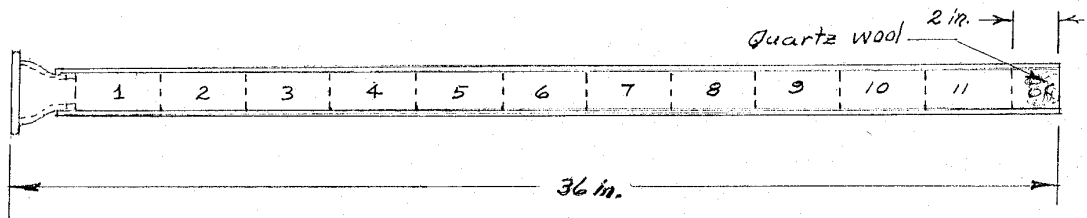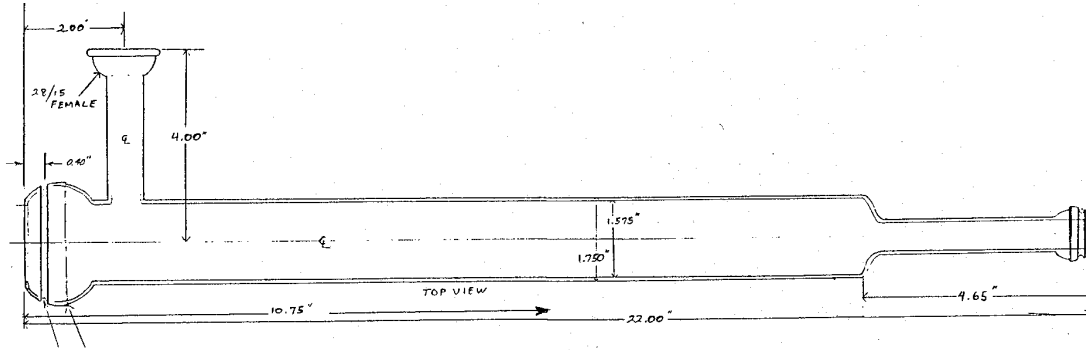
Description of oxidation products	Location (inches from left)	Temp. (ºC)

1. Clear, there is no evidence of any reaction products in collection tube, inserts, or quartz wool filter.

2.

3.

4.

5.

Description of Oxidation Products from TZM __1__

Test temperature (ºC) __500__ **Time (h)** __24__ **Flow rate (lpm)** __1__
Specimen mass (g): Initial __6.6733__ **Final** __6.7429__ **Mass gain** __0.0696__

Reaction chamber

Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Nothing apparent in the reaction chamber		

Deposition chamber
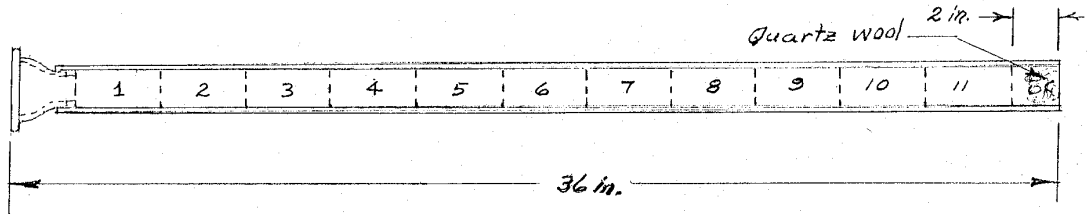
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Clear to 4.5 inches		
2. Light smoky deposit (insert I2)	4.7 to 7	
3. Lighter smoky deposit (I3 and I4)	7 to 15.5	
4. Slight color (on I5 and I6)	15.5 to 16.5	
5. Nothing apparent (I7 to I9)	16.5 to 25	
6. Very slight coloring (I9 and I11)	25 to 31.5	
7. Nothing apparent on quartz wool		

Description of Oxidation Products from TZM __15__

Test temperature (ºC) __5__00__   **Time (h)** __24__   **Flow rate (lpm)** __1__
Specimen mass (g):   Initial __13.8827__   **Final** __13.94643__   **Mass gain** __0.06373__

Reaction chamber
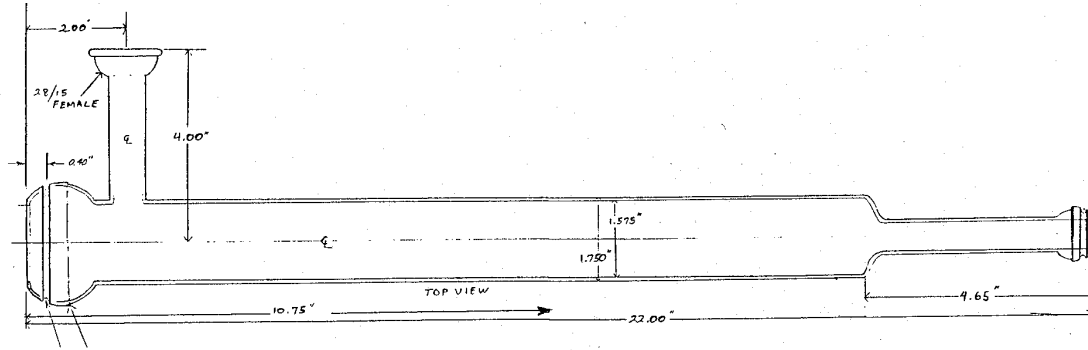
Description of oxidation products	Location (inches from left)	Temp. (ºC)

1. Clear, there is no evidence of any reaction product in test chamber.

2.

3.

4.

Deposition chamber
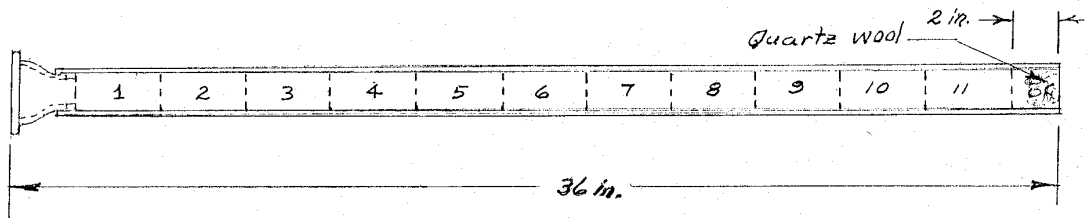
Description of oxidation products	Location (inches from left)	Temp. (ºC)

1. Clear, there is no evidence of any reaction products in collection tube, inserts, or quartz wool filter.

2.

3.

4.

5.

6.

Description of Oxidation Products from TZM __16__

Test temperature (ºC) __550__   Time (h) __24__   Flow rate (lpm) __1__
Specimen mass (g):   Initial __14.0636__   Final __14.4353__   Mass gain __0.3717__

Reaction chamber
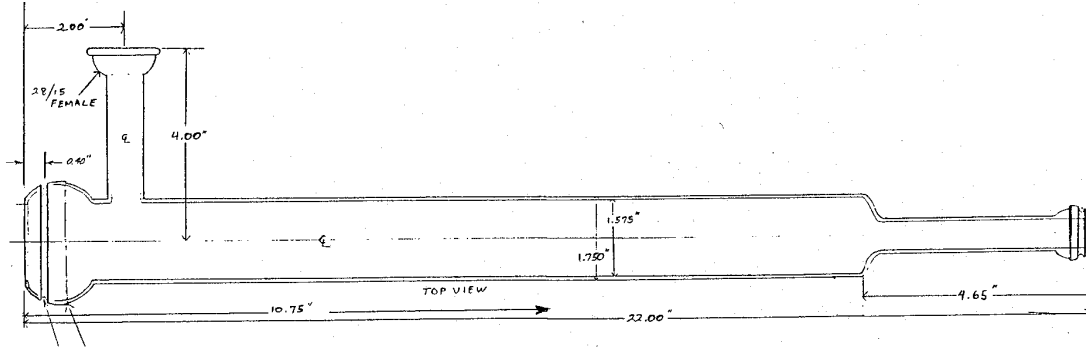
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Nothing apparent		

Deposition chamber
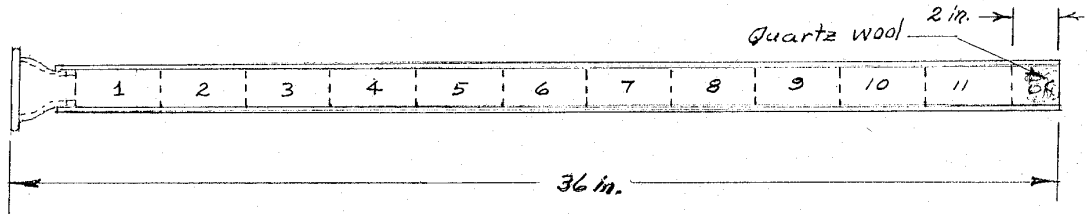
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Clear for first 12.5 inches		
2. Light smoky color (E4 thru E6)	12.5 to 18.5	
3. Light smoky to light blue	18.5 to 21	
4. Very light blue deposit	21 to 22	
5. Clear	22 to 31	
6. Very light bluish hue	31 to quartz wool	

Description of Oxidation Products from TZM __11__

Test temperature (ºC) __600__   Time (h) __8__   Flow rate (lpm) __1 lpm__
Specimen mass (g): Initial __13.15934__   Final __13.35724__   Mass gain __0.1970__

Reaction chamber
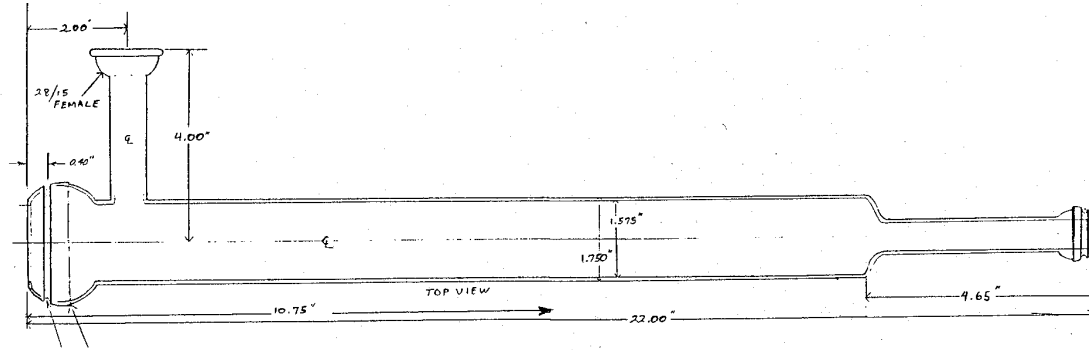
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Localized white deposit on wall	12 – 14	

(This be due to the close positioning of the specimen to the wall of the reaction tube or to a localized cold spot in the wall of the reaction tube.)

Deposition chamber
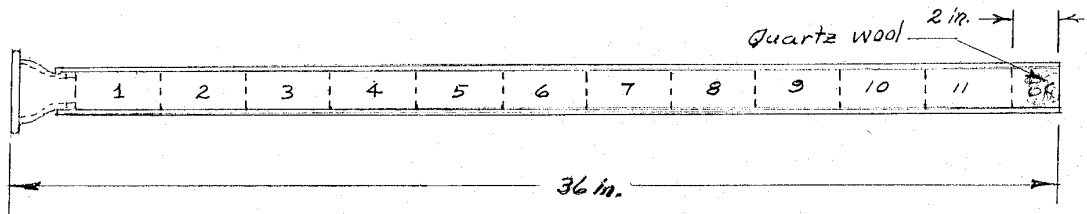
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Start of light white deposit	12.5	
2. Light, white deposit	12.5 – 15.5	
3. Heavier white, smokey deposit	15.5 – 27.5	
4. Clearer region, less deposit	27.5 - 34	
5. Blue-grey deposit in front of quartz wool	34 - 35	
6. Back of quartz wool is still white	35 – 36	

Description of Oxidation Products from TZM 7

Test temperature (ºC) 600   Time (h) 24   Flow rate (lpm) 1
Specimen mass (g):   Initial 6.88303   Final 7.65951   Mass gain 0.7765

Reaction chamber

Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Nothing apparent in the reaction chamber.		
2.		
3.		
4.		

Deposition chamber
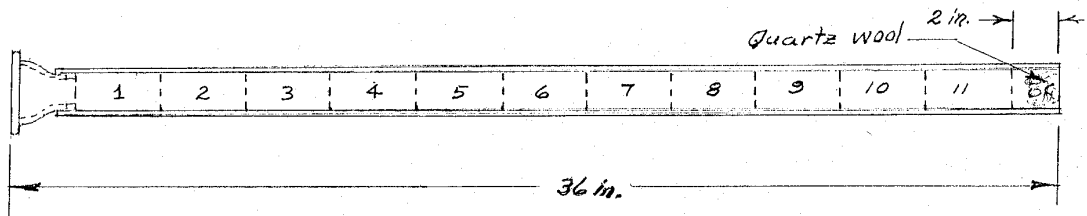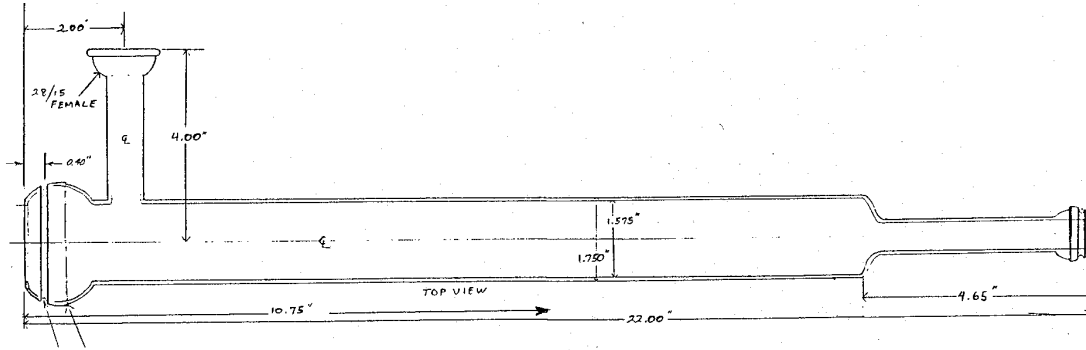
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Clear, nothing present	0 to 10	
2. Sporadic small crystals with small particles	10 to 11	
3. Smoky deposit, i.e., small particles	11 to 13	
4. White, to smoky, to light blue deposits	13 to 21.5	
5. Clear	21.5 to 34	
6. Quartz wool is blue in front and white in back		

Description of Oxidation Products from TZM __8__

Test temperature (ºC) __650__   Time (h) __8__   Flow rate (lpm) __1__
Specimen mass (g): Initial __6.8263__   Final __7.3607__   Mass gain __0.5344__

Reaction chamber

Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. clear, nothing in the reaction chamber.		
2.		
3.		
4.		

Deposition chamber
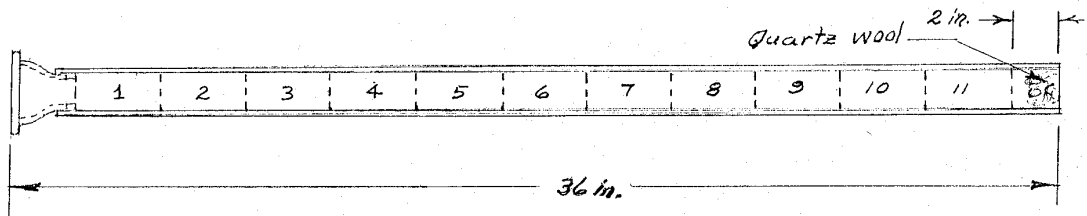
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Clear through first three inserts.	0 to 10.25	
2. Small crystals	11 to 12	
3. Heavy, white smoky deposit.	12 to 14.5	
4. Medium white smoky deposit.	14.5 to 16.5	
5. Clearing (no evidence of deposit)	17	
6. Quartz wool is very blue in front, clear (white) in back.		

Description of Oxidation Products from TZM __4__

Test temperature (ºC) __700__ **Time (h)** __2__ **Flow rate (lpm)** __0.010__
Specimen mass (g): Initial __6.1262__ **Final** __6.1887__ **Mass gain** __0.0625__

Reaction chamber
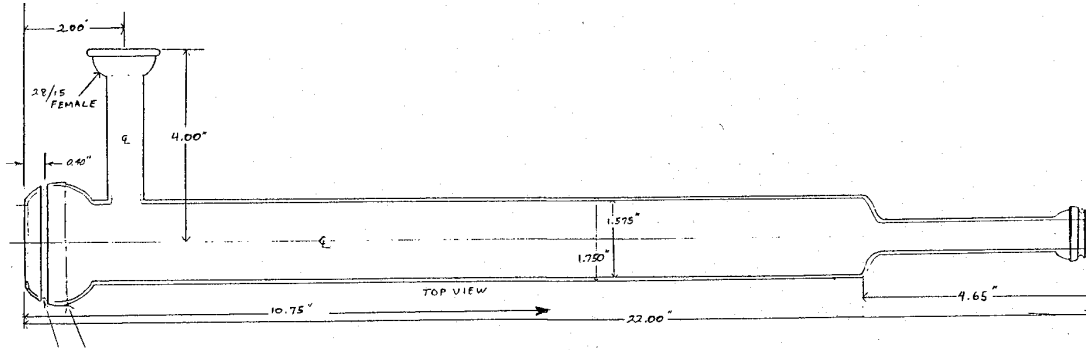
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Clear except for some silvery, flat ribbon-like crystals near outlet.	18.5 to 23	
2.		
3.		
4.		

Deposition chamber
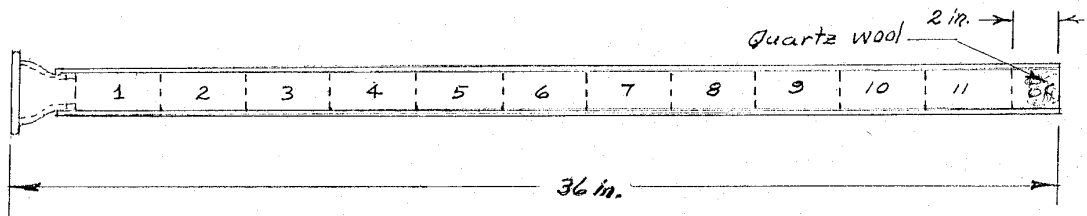
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. No apparent deposit in collection tube, inserts, or Q.W. filter.		
2.		
3.		
4.		
5.		
6.		

Description of Oxidation Products from TZM __17__

Test temperature (°C) __700__ Time (h) __8__ Flow rate (lpm) __0.01__
Specimen mass (g): Initial __13.4955__ Final __13.7485__ Mass gain __0.2530__

Reaction chamber
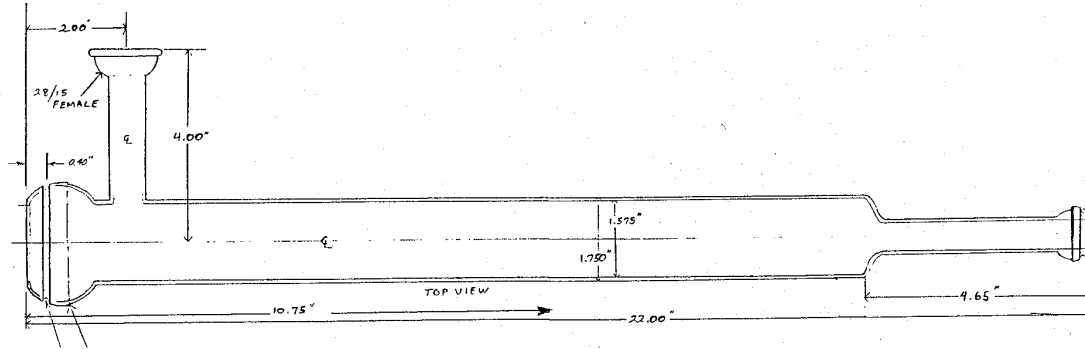
Description of oxidation products	Location (inches from left)	Temp. (°C)

1. Smoky deposit from 0 to 2.5 in., The cap has some smoky, white deposit.

2. Silvery crystals to ribbon-like needles   2.5 to 3.5

3. Thinly, distributed, very thin ribbon-like crystals 13.5 to 18.5
The crystals irradiant (showing green, blue, red or transparent depending upon angle of reflection).

Deposition chamber
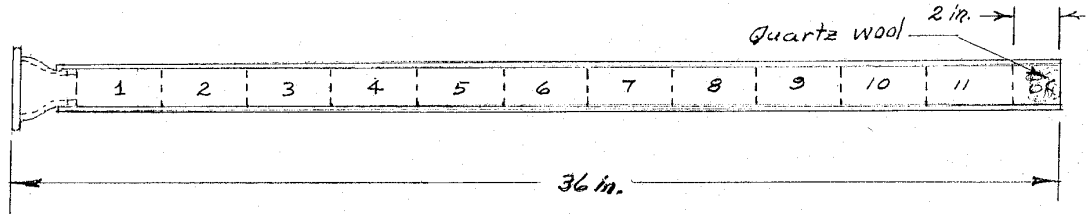
Description of oxidation products	Location (inches from left)	Temp. (°C)

1. Few, thin ribbon-like crystals.   6 to 12

2. Medium to small crystals   12 to 13.25

3. Light blue to smoky, thin deposit   13.25 to 16

4. Regions of light smoky blue deposits   18.5 to 31.

5. Nothing apparent on the quartz wool

Description of Oxidation Products from TZM __5__

Test temperature (ºC) __700__ **Time (h)** __2__   **Flow rate (lpm)** __0.05 lpm__
Specimen mass (g):   Initial __6.7467__ **Final** __6.7988__   **Mass gain** __0.0521__

Reaction chamber

Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Small silvery needle-like crystals, widely spaced	18.5 to 19.5	
2. Very large crystals (1/8 to ½ in. long)	19.5 to 20.5	
3.		
4.		

Deposition chamber
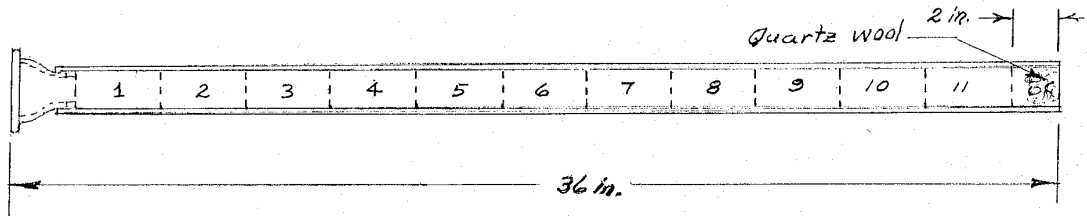
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Few speck-size crystals, widely spaced.	0 to 1	
2. Mostly clear	0 to 12	
3. Small crystals, widely spaced	10.75 to 12.25	
4. Clusters of very small particles.	11.75 to 12.25	
5. Particles become smaller and disappear.	12.25 to 13	
6. Clear thereafter.	13 to 36	

Description of Oxidation Products from TZM __18__

Test temperature (ºC) __700__   **Time (h)** __8__   **Flow rate (lpm)** __0.05__
Specimen mass (g): Initial __13.5565__   **Final** __13.7589__   **Mass gain** __0.2024__

Reaction chamber
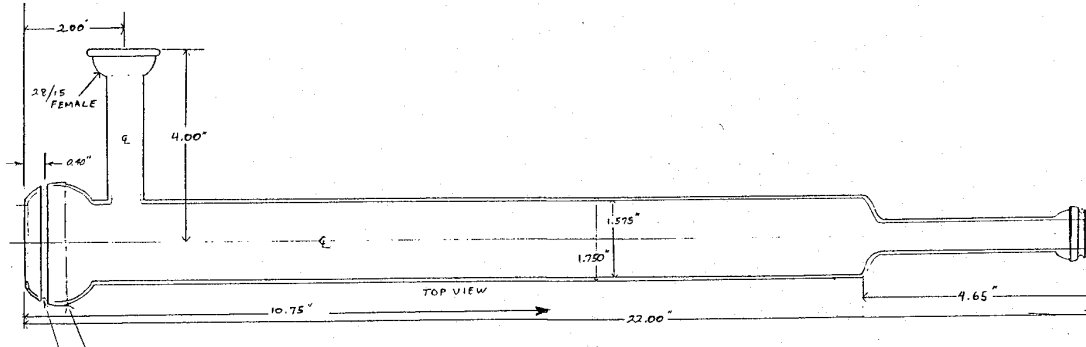
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. There may be some slight haze on walls	9.75 to 12	
2. Distinct needle-like crystals have formed	start at @ 13.5	
3. Heavier deposit of crystals.	16 to 17.5 (end of large section)	
4. Smaller less needle-like crystals	17.5 to 22	

Deposition chamber
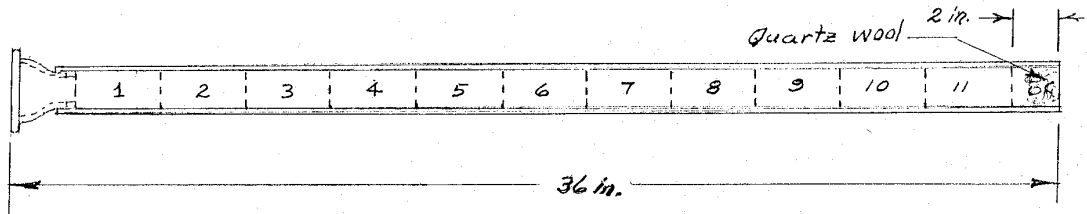
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Very light whitish, smoky haze	0 to 36	
2. Large ribbon-like crystals (some 0.5 in.)	9 to 11.5	
3. Heavier deposit which extends across I.D.	11.5 to 12.5	
4. Smaller crystals	12.5 to 14.5	
5. Just haze on wall	14.5 to 27	
6. Fewer, small crystals on wall	27 to 33.5	
7. Heavy collection of ribbon-like crystals in front quartz wool filter	33.5 to 34.5	
8. No indication on any color on quartz wool.		

Description of Oxidation Products from TZM __10__

Test temperature (ºC) __700__ **Time (h)** __2__ **Flow rate (lpm)** __0.10__
Specimen mass (g): Initial __6.71857__ **Final** __6.74414__ **Mass gain** __0.02557__

Reaction chamber
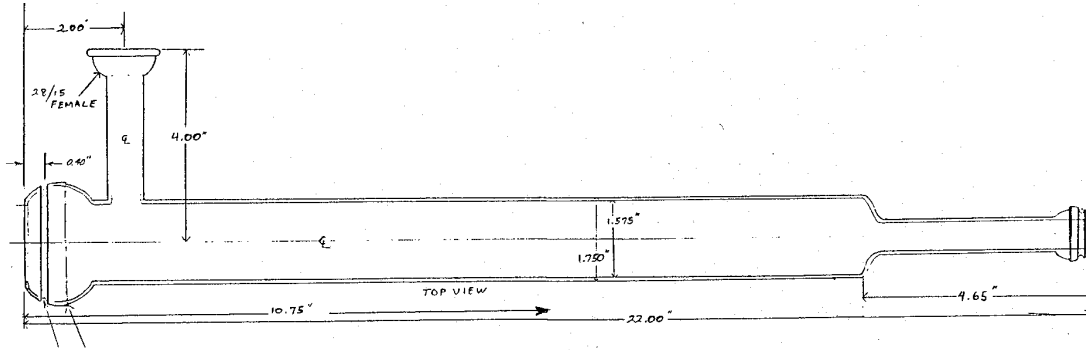
Description of oxidation products	Location (inches from left)	Temp. (ºC)

1. Large to medium shiny, silver, flat sliver-like crystals sporadically attached: 8 to 14.

2. Crystals on top of tube.   9 to 13

3. Crystals on bottom of tube.   12 to 14.5

4. Smokey deposit on top of tube.   12 to 14

Deposition chamber
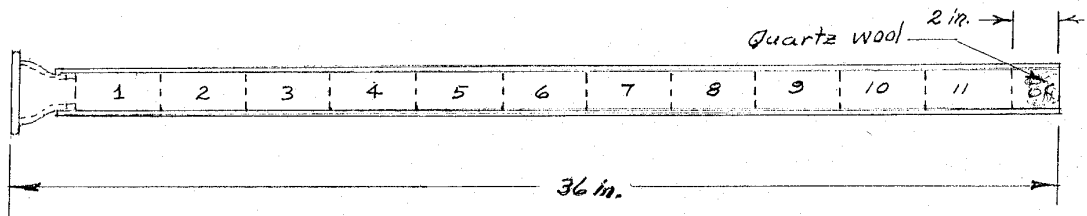
Description of oxidation products	Location (inches from left)	Temp. (ºC)

1. Clear   0 to 10

2. Silvery sliver-like crystals   10 to 13.5

3. Smoky deposit on wall   13.5 to 17

4. Smaller yellowish needle-like crystals   11 to 13

5. White smoky deposit: heavy 12.5 to 16, Becomes light to clear at about 16 inches.

6. Large silver crystals at 34 to 36 likely dislodged during transport.

7. None to barely detectable deposit in quartz wool.

Description of Oxidation Products from TZM __6__

Test temperature (ºC) __700__ Time (h) __2__   Flow rate (lpm) __0.5__
Specimen mass (g): Initial __6.4623__   Final __6.4560__   Mass loss __0.0063__

Reaction chamber
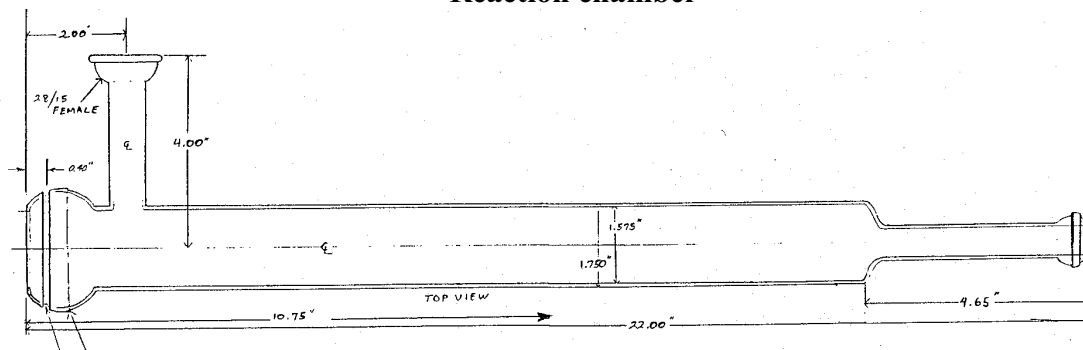
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Clear over most of the chamber.		
2. Silvery crystals on the wall	Last 2 inches	
3. The amount of crystals are less than those observed for the test with 0.05 lpm flow.		
4.		

Deposition chamber
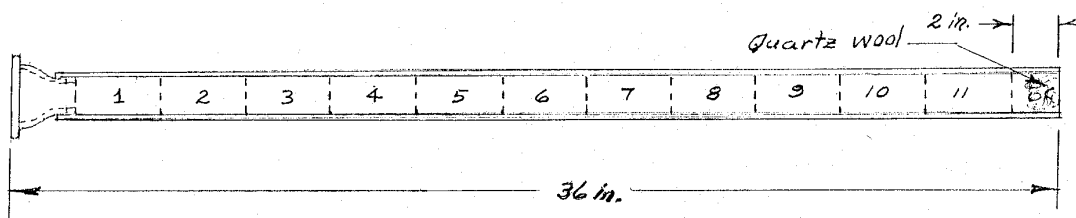
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Very small crystals on first insert.	2 to 2.5	
2. Clear	2.5 to 10	
3. Larger silvery crystals.	10 to 12.5	
4. White deposit.	12.5 to 17	
5. Very light blue deposit	17 to 31.5	
6. Heavier blue deposit	31.5 to 34.5	
7. Quartz wool is blue in front, white in back.	34.5 to 36.	

Description of Oxidation Products from TZM __12__

Test temperature (ºC) __700__   Time (h) __2__   Flow rate (lpm) _1 lpm__

Specimen mass (g): Initial __13.67683__   Final __13.67773__   Mass gain __0.0009___

Reaction chamber
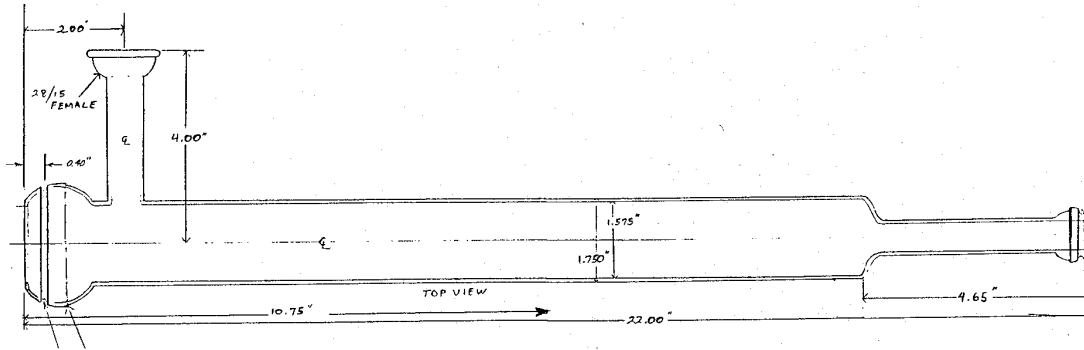
Description of oxidation products	Location (inches from left)	Temp. (ºC)

1. There is only a few small clear crystals at one location likely from when the specimen contacted the surface during removal.

Deposition chamber
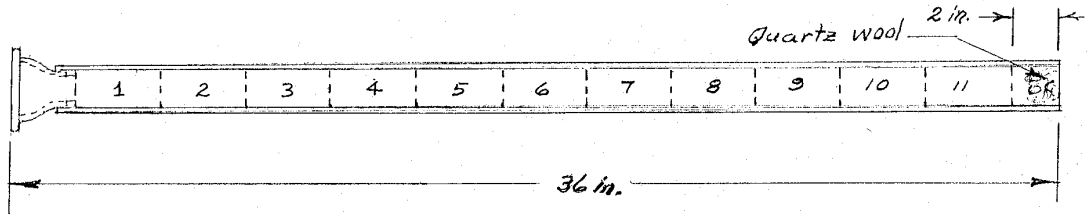
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Formation of clear crystal start	9.75 inches	
2. Crystal become larger	9.75 to 10.75	
4. Crystals become smaller	10.75 to 12.25	
5. White to smoke color deposit	12.25 to 16.25	
6. Very light haze	16.25 to 23.5	
7. Clear region	23.5 to 29	
8. Hazy deposit increases (bluish hue)	29 to 34	
9. Quartz wool is blue in front, white in back	34 to 36	

Description of Oxidation Products from TZM __9__

Test temperature (°C) __700__   **Time (h)** __2__   **Flow rate (lpm)** __2.5__
Specimen mass (g): Initial __6.52977__   **Final** __6.52907__   **Mass loss** __0.0007__

Reaction chamber

Description of oxidation products	Location (inches from left)	Temp. (°C)
1. No evidence of any deposit in the reaction chamber.		
2.		
3.		
4.		

Deposition chamber
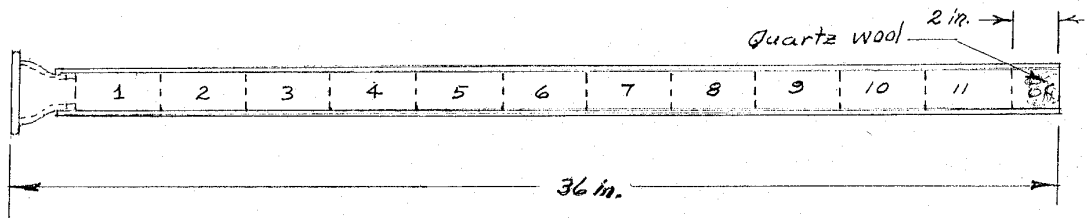
Description of oxidation products	Location (inches from left)	Temp. (°C)
1. Clear, except for a few small needle-like crystals.	0 to 10.5	
2. Small crystal, heavier deposition.	10.5 to 11.5	
3. White deposit on walls.	11.5 to 17	
4. Smokey deposit	17 to 33.5	
5. Blue deposit	33.5 to 34	
6. Blue deposit in front half, white at end of quartz wool.	34 to 36	

Description of Oxidation Products from TZM __3__

Test temperature (ºC) __750__ **Time (h)** __1__ **Flow rate (lpm)** __1__
Specimen mass (g): Initial __6.48253__ Final __6.1071__ Mass loss __0.3754__

Reaction chamber
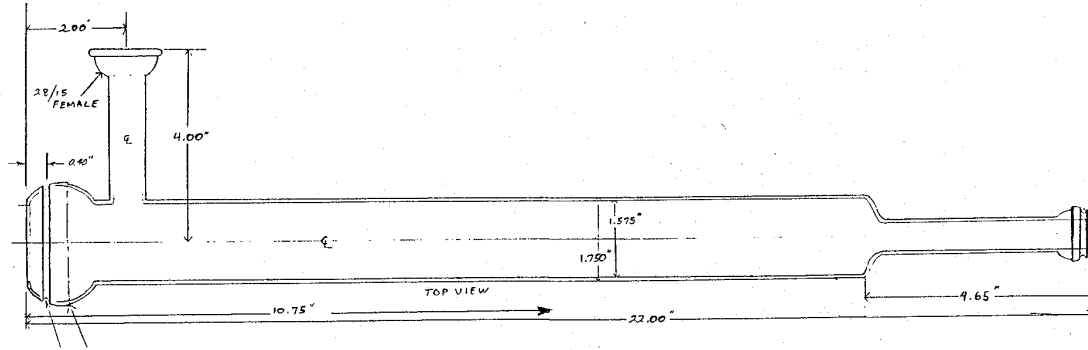
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Silvery flat needle-like crystals (1/4-in.) at exit.	20.5 to 23	

Deposition chamber
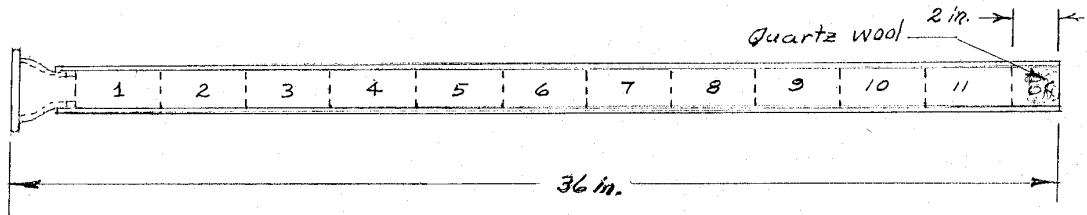
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Silvery needle-like crystals	0 to 1	
2. Clear.	1 to 10	
3. Large greenish crystals extending across tube	10 to 11.5	
4. Med. and small crystals, heavy conc.	11 to 12.25	
5. Small crystals with crystalline particles, i.e., white smoky deposit.	12.25 to 14	
6. Heavy white deposit , crystals becoming yellowish	14 to 16	
7. White smoky deposit with greenish streaks.	16 to 25	
8. Yellow-greenish smoky deposit	25 to 31	
9. Greenish smoky deposit becomes heavier.	34.25	
10. Heavy greenish deposit in quartz wool, the end is still white.	34.25 to 36	

Description of Oxidation Products from TZM __2__

Test temperature (ºC) __800__   Time (h) __1__   Flow rate (lpm) __1__
Specimen mass (g): Initial __6.9709__   Final __5.0653__   Mass loss __1.9056__
Final weight includes 3.6498 g from specimen plus 1.4152 from molten oxide.

Reaction chamber
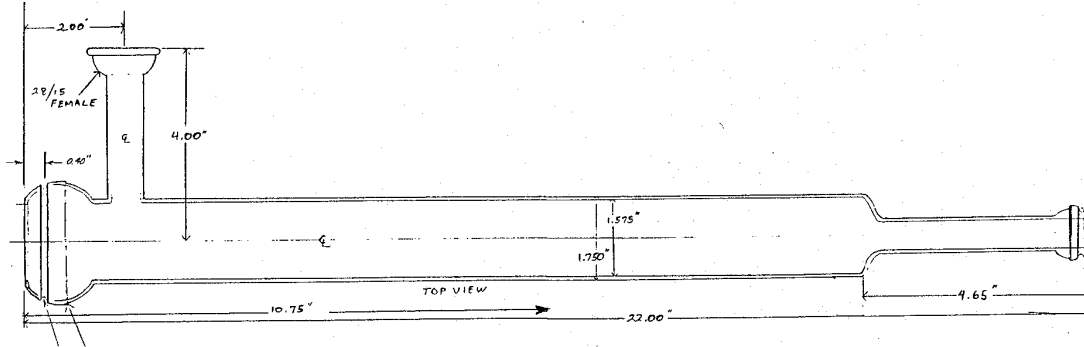
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Two pieces of molten oxide (1.4152 g)	13.4 to 17.5	
2. Crystals changing from white, to yellow, to greenish	18.5 to 22	
3. Larger greenish crystals appear to nearly block tube at outlet.		

Deposition chamber
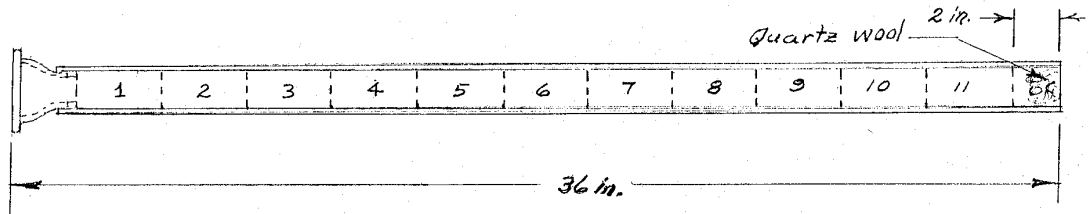
Description of oxidation products	Location (inches from left)	Temp. (ºC)
1. Clear.	0 to 11	
2. Few, yellow (silver) crystals	11 to 14	
3. Heavy yellow, greenish crystals extend across I.D.	14 to 16	
4. Heavy white smoky deposit with some crystals	16 to 20	
5. Deposit becomes yellow (or light greenish) again with some crystals	20 to 34	
6. Heavy conc. of yellow-greenish crystals in front of quartz wool.	34 to 35	
7. Heavy yellow-greenish deposit in front of quartz wool. (The back portion to the quartz wool is still white.)	35 to 36	

APPENDIX B

PROCEDURES FOR CLEANING
THE MOLYBDENUM-BEARING TEST COMPONENTS
AND PROCESSING THE SOLUTIONS
FOR ICP-AES ANALYSES

Processing and Analytical Procedures for Molybdenum –Bearing Glassware.
for
Molybdenum Oxidation Tests
IHRG# IRC-99-740

Scope: This procedure describes chemically removing molybdenum oxidation products from quartz test components. The test components the reaction chamber, collection tube, quartz tube inserts, and quartz wool filters as described in IHRG #IRC-99-740.

The following basic procedures will be used for all components (there will be only minor differences used to handle the various components).

1. Wash the component once or twice with 2.9 Molar NH_4OH solution.
2. Rinse with nanopure water.
3. Heat solutions in covered Teflon beakers on hot plate to drive off NH_3.
Transfer solution to 100-ml volumetric flask.
Add 5 ml of concentrated nitric acid (Normality of 15.8).
Dilute to 100 ml with nanopure water.
Analyze by ICP-AES.

The reaction chamber and 36-long collection tube will be sealed with Parafilm at on end. The 25 ml of the 2.9 M NH_4OH will then be added and the other end sealed with Parafilm. The component will then be manipulated to wash the inner surfaces. This solution will be transferred to a Teflon beaker and the process repeated with another batch of 2.9 M NH_4OH. The inside surfaces of the component will then be rinsed with nanopure water using a squirt bottle. The rinse water will be drained directly into the Teflon beaker. This rinsing process will be done at least twice. The solution collected in the Teflon beaker will be processes as indicated above.

There are two different sizes of quartz tube inserts: 1-in. long pieces and 3-in. long pieces. Molybdenum oxidation products will be cleaned from these pieces by rinsing them with of 2.9 M NH_4OH solution dispensed from a squirt bottle. Approximately 50 ml will be used to rinse each segment directly into a Teflon beaker. Each segment will then be rinsed with about 30 ml of nanopure water. The solution collected in the Teflon beaker will be processes as indicated above.

The quartz wool filter will be placed in a 50-ml plastic disposable beaker. It will be soaked in 25 ml of the 2.9 M NH_4OH solution overnight. The 25 ml of solution will be transferred to a Teflon beaker. Another 25 ml of 2.9 M NH_4OH solution will be added to the quartz wool for a second soak (approx. 10 minutes). The quartz wool will then be rinse with 2.9 M NH_4OH solution from a squirt bottle followed by a nanopure water rinse. All solutions will have been collected in the Teflon beaker which will be processes as indicated above.

APPENDIX C

ICP-AES ANALYSES,

MASS FLUX CALCULATIONS,

MOLYBDENUM DISTRIBUTIONS,

TEMPERATURE PROFILES

TZM14: Tested at 400°C for 24 hours (1000 sccm flow)

Test TZM14		ICP-AES measurements for 100 ml aloquits, except RXN Tube is 250 ml.			
				Mo 202.0	
Calculated per EDF SA-17, i.e., 2 sigma (S.D.) for uncertainities				========	
and 3 sigma (S.D.) for detection limits.					
For Data analyzed		% SD of Cal. Stds	sigma %	3.300	
on 6-21-99		SD of Blk Std	sigma prime ug/ml	0.005	
TZM14	400C/24h		F1, ug/ml	-0.008	
Tested 6-2-99	air		F2, ug/ml	-0.006	
time	24.000		F3, ug/ml	-0.006	
			F4, ug/ml	-0.008	
			F5, ug/ml	-0.007	
			F6, ug/ml	-0.008	
area,cm2	10.34500		F7, ug/ml	-0.011	
			F8,ug/ml	-0.013	
			F9,ug/ml	-0.013	
			F10,ug/ml	-0.012	
			F11,ug/ml	-0.013	
			QW,ug/ml	-0.010	
Flux set by EMFV.			C.T.,ug/ml	0.000	
			RXN T,ug/ml	-0.001	
			subtotal, ug	-11.750	
			(+/-) ug	0.228	
			Blk sub, ug	-8.400	
"IF" statement compares Total			(+/-) ug	3.742	
with 3*SD(ug), and all "FX" & "Component"			Total, ug	-3.350	
measurements with 3*SD(ug/ml).			(+/-) ug of Sum	3.749	
Total is selected if any of the					
"FX",QW,CT or RXN T measurements are			DL (3*SD),ug/ml	0.015	
greater than 3*SD(ug/ml).			DL (3*SD), ug	1.500	
		Detection Limit (EMFV)	g/m2-h	6.04E-05	
		Total correct for DL (IF statement), ug		1.500	
		Calculated from Total	Mass Flux, g/m2-h	-1.35E-04	
		Calc. from 'IF" statement	Mass Flux, g/m2-h	6.04E-05	
			± g/m2-h	1.51E-04	
			± %	249.91	

TZM15: Tested at 500°C for 24 hours (1000 sccm flow)

Mo Oxidation Test: TZM15 ---- 500C for 24 h at 1 liter/min.							
ICP Results	202.0	Volume	Mass Mo				
sample	Mo µg/mL	sample(mL)	ug				
TZM15G1	0.010	100	1.04				
TZM15G2	0.011	100	1.1				
TZM15G3	0.012	100	1.2				
TZM15G4	0.018	100	1.82				
TZM15G5	0.013	100	1.28				
TZM15G6	0.004	100	0.38				
TZM15G7	0.001	100	0.1				
TZM15G8	0.000	100	0				
TZM15G9	0.004	100	0.35				
TZM15G10	0.003	100	0.25				
TZM15G11	0.003	100	0.34				
TZM15QW	0.013	100	1.25				
TZM15 CT	0.020	100	1.96				
TZM15 RX	0.334	250	83.475				
		Total (ug)	94.545				
		Total (mg)	0.09				
	Amount excluding RXN and Col tube (		9.11				
			Mass Mo (ug)	April,29			
	Position	Mass Mo	w Col tube	Temp.,	Temp.,		
	in.	ug	redistributed	C	x 0.01		
TZM15G1	2	1.04	1.3	494	4.94	Specimen (cm^2)	
TZM15G2	5	1.1	1.3	539	5.39	10.369	
TZM15G3	8	1.2	1.5	508	5.08		
TZM15G4	11	1.82	2.2	456	4.56		
TZM15G5	14	1.28	1.6	349	3.49		
TZM15G6	17	0.38	0.5	274	2.74	Rate (g/m^2-h):	
TZM15G7	20	0.1	0.1	238	2.38	3.8E-03	
TZM15G8	23	0	0.0	211	2.11		
TZM15G9	26	0.35	0.4	187	1.87		
TZM15G10	29	0.25	0.3				
TZM15G11	32	0.34	0.4				
TZM15QW	35	1.25	1.5				
TZM15 CT		1.96					

C-2

TZM15: Tested at 500°C for 24 hours (1000 sccm flow)

Test TZM15		ICP-AES measurements for 100 ml aloquits, except RXN Tube is 250 ml.					
				Mo 202.0		Mass Mo ug	Percent in Component
Calculated per EDF SA-17, i.e., 2 sigma (S.D.) for uncertainities				============			
and 3 sigma (S.D.) for detection limits.							
For Data analyzed		% SD of Cal. Stds	sigma %	5.100			
on 7-06-99		SD of Blk Std	sigma prime ug/ml	0.004	**RXN CHMBR**	83.475	88.81
					CT&INSRTS	9.27	9.86
TZM15	500C/24h		G1, ug/ml	0.010	**QW FLTR**	1.25	1.33
Tested 6-8-99	air		G2, ug/ml	0.011			
time	24.000		G3, ug/ml	0.012	**Total**	**93.995**	
			G4, ug/ml	0.018			
			G5, ug/ml	0.013			
			G6, ug/ml	0.004			
area,cm2	10.36900		G7, ug/ml	0.001			
			G8,ug/ml	-0.006			
			G9,ug/ml	0.004			
			G10,ug/ml	0.003			
			G11,ug/ml	0.003			
			QW,ug/ml	0.013			
Flux set by EMFV.			C.T.,ug/ml	0.020			
			RXN T,ug/ml	0.334			
			subtotal, ug	93.995			
			(+/-) ug	8.524			
			Blk sub, ug	-8.400			
"IF" statement compares Total			(+/-) ug	2.993			
with 3*SD(ug), and all "IS" & "DB"			Total, ug	102.395			
measurements with 3*SD(ug/ml).			(+/-) ug of Sum	9.034			
Total is selected if any of the							
"GX",QW,CT or RXN T measurements are			DL (3*SD),ug/ml	0.012			
greater than 3*SD(ug/ml).			DL (3*SD), ug	1.200			
		Detection Limit (EMFV)	g/m2-h	4.82E-05			
		Total correct for DL (IF statement), ug		102.395			
		Calculated from Total	Mass Flux, g/m2-h	4.11E-03			
		Calc. from 'IF" statement	Mass Flux, g/m2-h	4.11E-03			
			± g/m2-h	3.63E-04			
			± %	8.82			
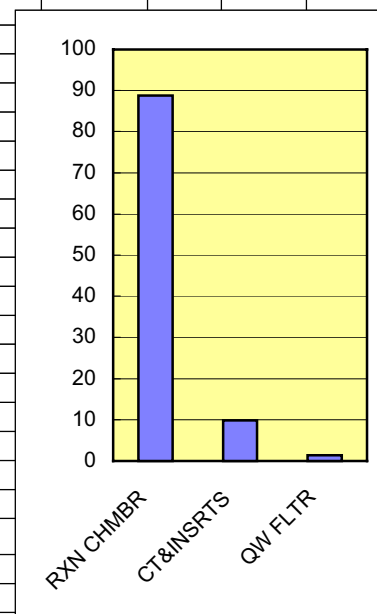
TZM1: Tested at 500°C for 24 hours (1000 sccm flow)

Test TZM1		ICP-AES measurements for 100 ml aloquits, except CT (w stuck I1) is 250 ml.		
				Mo 202.0
Calculated per EDF SA-17, i.e., 2 sigma (S.D.) for uncertainities				=========
and 3 sigma (S.D.) for detection limits.				
For Data analyzed		% SD of Cal. Stds	sigma %	2.600
on 12-03-99		SD of Blk Std	sigma prime ug/ml	0.007
TZM1	500C/24h			
Tested 11-15-99	air		I2, ug/ml	0.020
time	24.000		I3, ug/ml	0.027
			I4, ug/ml	0.016
			I5, ug/ml	0.001
			I6, ug/ml	-0.003
area,cm2	9.1010		I7, ug/ml	0.007
			I8,ug/ml	0.003
			I9,ug/ml	-0.003
			I10,ug/ml	-0.007
			I11,ug/ml	-0.004
			QW,ug/ml	0.022
Flux set by EMFV.			(C.T+I1).,ug/ml	0.014
			RXN T,ug/ml	0.405
			subtotal, ug	51.900
			(+/-) ug	2.127
			Blk sub, ug	-7.800
"IF" statement compares Total			(+/-) ug	5.048
with 3*SD(ug), and all "IX" & "Component"			Total, ug	59.700
measurements with 3*SD(ug/ml).			(+/-) ug of Sum	5.477
Total is selected if any of the				
"IX",QW,CT or RXN T measurements are			DL (3*SD),ug/ml	0.021
greater than 3*SD(ug/ml).			DL (3*SD), ug	2.100
		Detection Limit (EMFV)	g/m2-h	9.61E-05
		Total corrected for DL (IF statement), ug		59.700
		Calculated from Total	Mass Flux, g/m2-h	2.73E-03
		Calc. from 'IF" statement	Mass Flux, g/m2-h	2.73E-03
			± g/m2-h	2.51E-04
			± %	9.18

TZM1: Tested at 500°C for 24 hours (1000 sccm flow)

							Mass Mo ug	Percent in Component	
Mo Oxidation Test: TZM1 ---- 500C for 24 h at 1 liter/min.						**RXN CHMBR**	40.500	78.03	
ICP Results	**202.0**	**Volume**	**Mass Mo**			**CT&INSRTS**	9.200	17.73	
sample	**Mo µg/m**	**sample(mL)**	**ug**			**QW FLTR**	2.2	4.24	
						Total	**51.9**		
TZM1I2	0.020	100	2.000						
TZM1I3	0.027	100	2.700						
TZM1I4	0.016	100	1.600						
TZM1I5	0.001	100	0.100						
TZM1I6	-0.003	100	-0.300						
TZM1I7	0.007	100	0.700						
TZM1I8	0.003	100	0.300						
TZM1I9	-0.003	100	-0.300						
TZM1I10	-0.007	100	-0.700						
TZM1I11	-0.004	100	-0.400						
TZM1QW	0.022	100	2.200						
TZM1(CT+I1	0.014	250	3.500						
TZM1 RXN	0.405	100	40.500						
		Total (ug)	**51.9**						
		Total (mg)	**0.052**						
		Amount excluding RXN and Col tube (		**7.9**					

	Position in.	Mass Mo ug	Mass Mo (ug) w Col tube redistributed			Nov.8,99 Temp., C	Temp., x 0.01	
	2					503	5.03	Specimen (cm²)
TZM1I2	5	2.000	2.9	4.2		517	5.17	9.101
TZM1I3	8	2.700	3.9	5.6		491	4.91	
TZM1I4	11	1.600	2.3	3.3		435	4.35	
TZM1I5	14	0.100	0.1	0.2		350	3.5	
TZM1I6	17	-0.300	-0.4	0.0		301	3.01	Rate (g/m²-h):
TZM1I7	20	0.700	1.0	1.5		263	2.63	**2.38E-03**
TZM1I8	23	0.300	0.4	0.6		232	2.32	
TZM1I9	26	-0.300	-0.4	0.0		205	2.05	
TZM1I10	29	-0.700	-1.0	0.0				
TZM1I11	32	-0.400	-0.6	0.0				
TZM1QW	35	2.200	3.2	4.6				
TZM1(CT+I1)		3.500						

TZM16: Tested at 550°C for 24 hours (1000 sccm flow)

									Mass Mo ug	Percent in Component	
Mo Oxidation Test: TZM16 ---- 550C for 24 h at 1 liter/min.								**RXN CHMBR**	559.700	61.90	
ICP Results	**202.0**	**Volume**	**Mass Mo**					**CT&INSRTS**	334.100	36.95	
sample	**Mo µg/m**	**sample(mL)**	**ug**					**QW FLTR**	10.4	1.15	
TZM16E1	0.025	100	2.500					**Total**	**904.2**		
TZM16E2	0.020	100	2.000								
TZM16E3	0.024	100	2.400								
TZM16E4	1.045	100	104.500								
TZM16E5	1.071	100	107.100								
TZM16E6	0.511	100	51.100								
TZM16E7	0.280	100	28.000								
TZM16E8	0.134	100	13.400								
TZM16E9	0.100	100	10.000								
TZM16E10	0.059	100	5.900								
TZM16E11	0.032	100	3.200								
TZM16QW	0.104	100	10.400								
TZM16CT	0.040	100	4.000								
TZM16 RXN	5.597	100	559.700								
		Total (ug)	**904.200**								
		Total (mg)	**0.904**								
		Amount excluding RXN and Col tube (		**340.500**							

	Position in.	Mass Mo ug	Mass Mo (ug) w Col tube redistributed								
TZM16E1	3	2.5	2.529				Specimen (cm^2)				
TZM16E2	5	2.000	2.023				10.402				
TZM16E3	8	2.400	2.428								
TZM16E4	11	104.500	105.728								
TZM16E5	14	107.100	108.358								
TZM16E6	17	51.100	51.700				Rate (g/m^2-h):				
TZM16E7	20	28.000	28.329				**3.62E-02**				
TZM16E8	23	13.400	13.557								
TZM16E9	26	10.000	10.117								
TZM16E10	29	5.900	5.969								
TZM16E11	32	3.200	3.238								
TZM16QW	35	10.400	10.522								
TZM16CT		4.000									

TZM11: Tested at 600°C for 8 hours (1000 sccm flow)

Mo Oxidation Test: TZM11 ---- 600C for 8 h at 1 liter/min.						Mass Mo	Fraction in		
ICP Results	202.0	Volume	Mass Mo			ug	Component		
sample	Mo µg/mL	sample(mL)	ug						
					RXN CHMBR	261.75	8.07		
TZM 11-B1	0.055	100	5.5		CT&INSRTS	2621.2	80.77		
TZM 11-B2	0.024	100	2.4		QW FLTR	362.45	11.17		
TZM 11-B3	0.042	100	4.2						
TZM 11-B4	0.486	100	48.6		Total	3245.4			
TZM 11-B5	6.956	100	695.6						
TZM 11-B6	7.339	100	733.9						
TZM 11-B7	4.077	100	407.7						
TZM 11-B8	2.830	100	283						
TZM 11-B9	1.698	100	169.8						
TZM 11-B10	1.206	100	120.6						
TZM 11-B11	0.469	100	46.9						
TZM 11 GW(avg.)	3.625	100	362.45						
TZM 11 CT-B	0.412	250	103						
TZM 11 RXN	1.047	250	261.75						
		Total (ug)	3245.4						
		Total (mg)	3.25						
	Amount excluding RXN and Col tube (ug		2880.65						

Fraction in Component bar chart: RXN CHMBR, CT&INSRTS, QW FLTR

	Position in.	Mass Mo ug	Mass Mo (ug) w Col tube redistributed	April,28 Temp.(2), C				
TZM 11-B1	2	5.5	5.7	593	Specimen (cm²)			
TZM 11-B2	5	2.4	2.5	620	10.235			April,7
TZM 11-B3	8	4.2	4.4	585				Temp.(1)
TZM 11-B4	11	48.6	50.3	520		15	0	602
TZM 11-B5	14	695.6	720.5	422		16	1	634
TZM 11-B6	17	733.9	760.1	354	Rate (g/m²-h):	17	2	647
TZM 11-B7	20	407.7	422.3	311	0.40	18	3	650
TZM 11-B8	23	283	293.1	274		19	4	652
TZM 11-B9	26	169.8	175.9	240		20	5	653
TZM 11-B10	29	120.6	124.9			21	6	646
TZM 11-B11	32	46.9	48.6			22	7	632
TZM 11 GW(avg.)	35	362.45	375.4			23	8	617
TZM 11 CT-B		103				24	9	607
						25	10	590
						26	11	500
						30	15	407
						34	19	354
						38	23	339

Mass, Temp.(2), Temp.(1) vs. Distance along collection tube, in.

TZM7: Tested at 600°C for 24 hours (1000 sccm flow)

Mo Oxidation Test: TZM7 ---- 600C for 24 h at 1 liter/min.					Percent	
					Mass Mo	in
ICP Results	202.0	Volume	Mass Mo		ug	Component
sample	Mo µg/mL	sample(mL)	ug			
				RXN CHMBR	392.7	11.03
TZM 7 E1	0.049	100	4.94	CT&INSRTS	2800.71	78.65
TZM 7 E2	0.062	100	6.21	QW FLTR	367.6	10.32
TZM 7 E3	0.061	100	6.07			
TZM 7 E4	12.730	100	1273	Total	3561.01	
TZM 7 E5	7.416	100	741.6			
TZM 7 E6	4.041	100	404.1			
TZM 7 E7	1.758	100	175.8			
TZM 7 E8	0.524	100	52.42			
TZM 7 E9	0.204	100	20.37			
TZM 7 E10	0.155	100	15.54			
TZM 7 E11	0.683	100	68.25			
TZM 7 QW	3.676	100	367.6			
TZM 7 Col Tube	0.324	100	32.41			
TZM 7 RXN	3.927	100	392.7			
		Total (ug)	3561.01			
		Total (mg)	3.56			
		Amount excluding RXN and Col tube (ug)	3135.9			

			Mass Mo (ug)	04/28/1999		
	Position	Mass Mo	w Col tube	Temp (1).,		
	in.	ug	redistributed	C		
TZM 7 E1	2	4.94	5.0	593	Specimen (cm²)	
TZM 7 E2	5	6.21	6.3	620	9.136	
TZM 7 E3	8	6.07	6.1	585		
TZM 7 E4	11	1273	1286.2	520		
TZM 7 E5	14	741.6	749.3	422		
TZM 7 E6	17	404.1	408.3	354	Rate (g/m²-h):	
TZM 7 E7	20	175.8	177.6	311	0.16	
TZM 7 E8	23	52.42	53.0	274		
TZM 7 E9	26	20.37	20.6	240		
TZM 7 E10	29	15.54	15.7			
TZM 7 E11	32	68.25	69.0			
TZM 7 QW	35	367.6	371.4			
TZM 7 Col Tube		32.41				
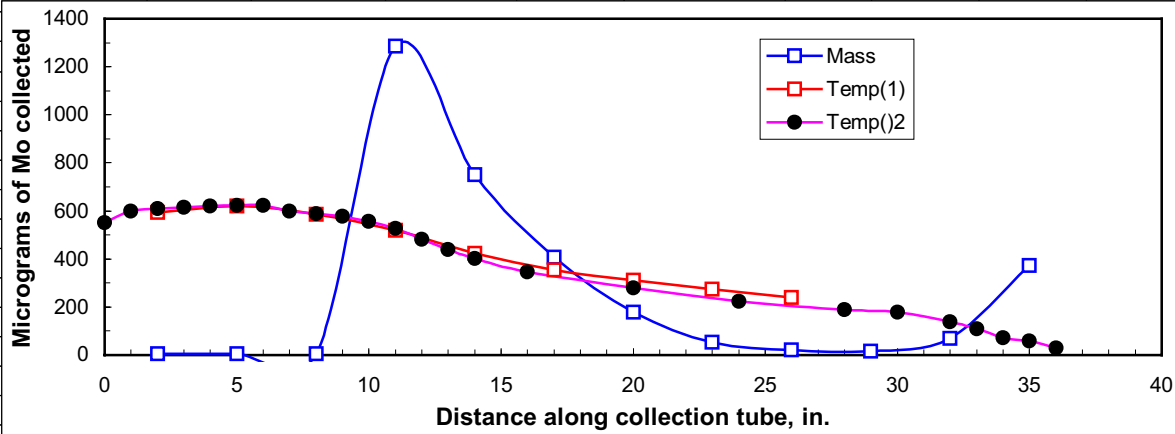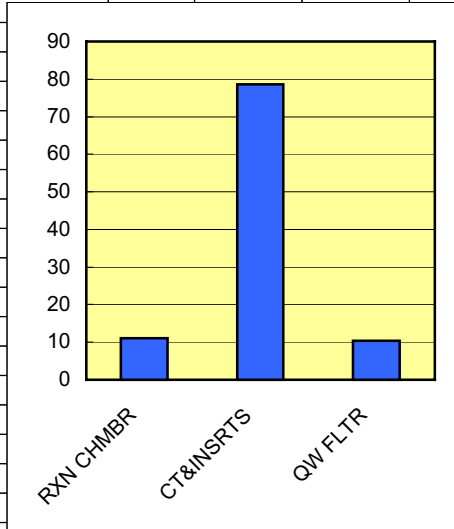
TZM8: Tested at 650°C for 8 hours (1000 sccm flow)

Mo Oxidation Test TZM8 ---- 650C for 8 h at 1.0 liters/minute						Mass Mo ug	Percent in Component
ICP Results	**202.0**	**Volume**	**Mass Mo**				
sample	Mo µg/mL	Sample(mL)	ug				
					RXN CHMBR	2757	11.01
TZM 8 B1	0.337	100	33.7		CT&INSRTS	18595.8	74.23
TZM 8 B2	0.439	100	43.9		QW FLTR	3698	14.76
TZM 8 B3	7.132	100	713.2				
TZM 8 B4	104.100	100	10410		Total	25050.8	
TZM 8 B5	40.940	100	4094				
TZM 8 B6	9.321	100	932.1				
TZM 8 B7	2.387	100	238.7				
TZM 8 B8	1.641	100	164.1				
TZM 8 B9	1.447	100	144.7				
TZM 8 B10	1.942	100	194.2				
TZM 8 B11	3.922	100	392.2				
TZM 8 QW	36.980	100	3698				
TZM 8 Col Tube	12.350	100	1235				
TZM 8 RXN	27.570	100	2757				
		Total (ug)	25051				
		Total (mg)	25.1				
	Amount excluding RXN and Col tube (mg)		21059				

	Position in.	Mass Mo ug	Mass Mo,ug w Col tube redistributed	Mass Mo,mg w Col tube redistributed
TZM 8 B1	2	33.7	35.7	0.0357
TZM 8 B2	5	43.9	46.5	0.0465
TZM 8 B3	8	713.2	755.0	0.7550
TZM 8 B4	11	10410.0	11020.5	11.0205
TZM 8 B5	14	4094.0	4334.1	4.3341
TZM 8 B6	17	932.1	986.8	0.9868
TZM 8 B7	20	238.7	252.7	0.2527
TZM 8 B8	23	164.1	173.7	0.1737
TZM 8 B9	26	144.7	153.2	0.1532
TZM 8 B10	29	194.2	205.6	0.2056
TZM 8 B11	32	392.2	415.2	0.4152
TZM 8 QW	35	3698.0	3914.9	3.9149
TZM 8 Col Tube		1235.0		

Specimen (cm²) 9.123

Rate (g/m²-h): **3.43**

Aug.2,99

	Front of C.T.	Temp., C	Temp., x 10
16	0	588	5880
17	1	633	6330
18	2	643	6430
19	3	649	6490
20	4	657	6570
21	5	663	6630
22	6	660	6600
23	7	647	6470
24	8	640	6400
25	9	627	6270
26	10	615	6150
27	11	586	5860
28	12	550	5500
29	13	499	4990
30	14	466	4660
31	15	430	4300
32	16	397	3970
33	17	375	3750
34	18	361	3610
35	19	341	3410
36	20	326	3260
40	24	269	2690
44	28	228	2280
46	30	205	2050
48	32	166	1660
49	33	128	1280
50	34	84	840
51	35	68	680
52	36	30	300

TZM4: Tested at 700°C for 2 hours (10 sccm flow)

		Mass Mo ug	Percent in Component
Mo Oxidation Test TZM4 ---- 700C for 2 h at 0.01 liter/min.			
	RXN CHMBR	4208	93.55
ICP Results 202.0	CT&INSRTS	282.8	6.29
sample Mo µg/mL	QW FLTR	7.2	0.16
	Total	**4498**	

sample	Mo µg/mL	Volume sample(mL)	Mass Mo ug
TZM 4 H1	0.072	100	7.2
TZM 4 H2	0.044	100	4.4
TZM 4 H3	0.038	100	3.8
TZM 4 H4	1.035	100	103.5
TZM 4 H5	0.147	100	14.7
TZM 4 H6	0.011	100	1.1
TZM 4 H7	-0.004	100	-0.4
TZM 4 H8	-0.002	100	-0.2
TZM 4 H9	0.009	100	0.9
TZM 4 H10	0.034	100	3.4
TZM 4 H11	0.001	100	0.1
TZM 4 QW	0.072	100	7.2
TZM 4 Col Tube	1.443	100	144.3
TZM 4 RXN	42.08	100	4208
		Total (ug)	**4498**
		Total (mg)	**4.5**
		Amount excluding RXN and Col tube (ug):	**145.7**

	Position in.	Mass Mo ug	Mass Mo (ug) w Col tube redistributed	Temp. x 10	April,28 Temp.(1), C				Summer'99 Temp.(2),C
TZM 4 H1	2	7.2	14.3	6720	672	Specimen (cm^2)	16	0	659
TZM 4 H2	5	4.4	8.8	7060	706	8.994	17	1	676
TZM 4 H3	8	3.8	7.6	6790	679		18	2	683
TZM 4 H4	11	103.5	206.0	5990	599		19	3	690
TZM 4 H5	14	14.7	29.3	4750	475		20	4	699
TZM 4 H6	17	1.1	2.2	3850	385	Rate (g/m^2-h):	21	5	708
TZM 4 H7	20	-0.4	-0.8	3270	327	**2.50**	22	6	707
TZM 4 H8	23	-0.2	-0.4	2820	282		23	7	696
TZM 4 H9	26	0.9	1.8				24	8	685
TZM 4 H10	29	3.4	6.8				25	9	671
TZM 4 H11	32	0.1	0.2				26	10	648
TZM 4 QW	35	7.2	14.3				27	11	609
TZM 4 Col Tube		144.3					28	12	563
							29	13	518
							30	14	476
							32	16	415
							36	20	319
							40	24	255
							44	28	220
							46	30	174
							48	32	96
							49	33	63
							50	34	39
							51	35	30
							52	36	20

TZM17: Tested at 700°C for 8 hours (10 sccm flow)

									Mass Mo	Percent in	
									ug	Component	
Mo Oxidation Test: TZM17 ---- 700C for 8 h at 0.01 liter/min.											
								RXN CHMBR	12810.000	66.95	
ICP Results	**202.0**	**Volume**	**Mass Mo**					**CT&INSRTS**	6321.400	33.04	
sample	**Mo µg/m**	**sample(mL)**	**ug**					**QW FLTR**	1.7	0.01	
TZM17J1	5.234	100	523.400					**Total**	**19133.1**		
TZM17J2	2.062	100	206.200								
TZM17J3	5.346	100	534.600								
TZM17J4	16.670	100	1667.000								
TZM17J5	1.233	100	123.300								
TZM17J6	0.033	100	3.300								
TZM17J7	0.002	100	0.200								
TZM17J8	-0.006	100	-0.600								
TZM17J9	-0.007	100	-0.700								
TZM17J10	-0.003	100	-0.300								
TZM17J11	0.010	100	1.000								
TZM17QW	0.017	100	1.700								
TZM17CT	32.640	100	3264.000								
TZM17 RXN	128.100	100	12810.000								
		Total (ug)	**19133.100**								
		Total (mg)	**19.133**								
		Amount excluding RXN and Col tube (	**3059.100**								
			Mass Mo (ug)								
	Position	**Mass Mo**	**w Col tube**								
	in.	**ug**	**redistributed**								
TZM16E1	3	523.4	1081.858	1081.86				Specimen (cm^2)			
TZM16E2	5	206.200	426.211	426.21				10.311			
TZM16E3	8	534.600	1105.008	1105.01							
TZM16E4	11	1667.000	3445.656	3445.66							
TZM16E5	14	123.300	254.859	254.86							
TZM16E6	17	3.300	6.821	6.82				Rate (g/m^2-h):			
TZM16E7	20	0.200	0.413	0.41				**2.32E+00**			
TZM16E8	23	-0.600	-1.240	0.00							
TZM16E9	26	-0.700	-1.447	0.00							
TZM16E10	29	-0.300	-0.620	0.00							
TZM16E11	32	1.000	2.067	2.07							
TZM16QW	35	1.700	3.514	3.51							
TZM16CT		3264.000									

TZM5: Tested at 700°C for 2 hours (50 sccm flow)

				Mo Oxidation Test TZM5 ---- 700C for 2 h at 0.05 liters/minute				Percent	
							Mass Mo	in	
ICP Results	202.0	Volume	Mass Mo				ug	Component	
sample	Mo µg/mL	Sample(mL)	ug						
					RXN CHMBR		3750	82.46	
TZM 5 F1	0.179	100	17.9		CT&INSRTS		793.7	17.45	
TZM 5 F2	0.098	100	9.8		QW FLTR		4.2	0.09	
TZM 5 F3	0.118	100	11.8						
TZM 5 F4	6.181	100	618.1		Total		4547.9		
TZM 5 F5	0.368	100	36.8						
TZM 5 F6	0.153	100	15.3						
TZM 5 F7	0.009	100	0.9						
TZM 5 F8	0.009	100	0.9						
TZM 5 F9	0.079	100	7.9						
TZM 5 F10	0.104	100	10.4						
TZM 5 F11	0.000	100	0						
TZM5 QW	0.042	100	4.2						
TZM 5 FG	0.158	100	15.8						
TZM 5 Col Tube	0.481	100	48.1						
TZM 5 RXN	37.500	100	3750						
		Total (ug)	4548						
		Total (mg)	4.5						
	Amount excluding RXN and Col tube (mg)		749.8						

			Mass Mo,ug	Mass Mo,mg					
	Position	Mass Mo	w Col tube	w Col tube					
	in.	ug	redistributed	redistributed					
TZM 5 F1	2	17.9	19.0	0.0190					
TZM 5 F2	5	9.8	10.4	0.0104	Specimen (cm²)				Summer'99
TZM 5 F3	8	11.8	12.6	0.0126	9.111				Temp., C
TZM 5 F4	11	618.1	657.8	0.6578			16	0	659
TZM 5 F5	14	36.8	39.2	0.0392			17	1	676
TZM 5 F6	17	15.3	16.3	0.0163			18	2	683
TZM 5 F7	20	0.9	1.0	0.0010	Rate (g/m²-h):		19	3	690
TZM 5 F8	23	0.9	1.0	0.0010	2.50		20	4	699
TZM 5 F9	26	7.9	8.4	0.0084			21	5	708
TZM 5 F10	29	10.4	11.1	0.0111			22	6	707
TZM 5 F11	31	0.0	0.0	0.0000			23	7	696
TZM5 QW	35	4.2	4.5	0.0045			24	8	685
TZM 5 Col Tube		48.1					25	9	671
							26	10	648
							27	11	609
							28	12	563
							29	13	518
							30	14	476
							32	16	415
							36	20	319
							40	24	255
							44	28	220
							46	30	174
							48	32	96
							49	33	63
							50	34	39
							51	35	30
							52	36	20

TZM18: Tested at 700°C for 8 hours (50 sccm flow)

							Mass Mo ug	Percent in Component
Mo Oxidation Test: TZM18 ---- 700C for 8 h at 0.05 liter/min.						RXN CHMBR	3001.0	5.22
ICP Results	202.0	Volume	Mass Mo			CT&INSRTS	53302.3	92.78
sample	Mo µg/mL	sample(mL)	ug			QW FLTR	1148.0	2.00
TZM18 H1, ug/ml	0.190	100	19.000			Total	57451.3	
TZM18 H2, ug/ml	3.359	100	335.900					
TZM18 H3, ug/ml	140.600	100	14060.000					
TZM18 H4, ug/ml	237.400	100	23740.000					
TZM18 H5, ug/ml	6.535	100	653.500					
TZM18 H6, ug/ml	0.481	100	48.100					
TZM18 H7, ug/ml	0.961	100	96.100					
TZM18 H8,ug/ml	1.547	100	154.700					
TZM18 H9,ug/ml	12.830	100	1283.000					
TZM18 H10,ug/ml	38.120	100	3812.000					
TZM18 H11,ug/ml	81.680	100	8168.000					
TZM18 QW,ug/ml	11.480	100	1148.000					
TZM18 (C.T.).,ug/ml	9.320	100	932.000					
TZM18 RXN T,ug/ml	30.010	100	3001.000					
		Total (ug)	57451.300					
		Total (mg)	57.451					
	Amount excluding RXN and Col tube (ug):		53518.300					
			Mass Mo (ug)					
	Position	Mass Mo	w Col tube					
	in.	ug	redistributed					
TZM18H1	3	19	19.331			Specimen (cm^2)		
TZM18H2	5	335.900	341.750			10.326		
TZM18H3	8	14060.000	14304.849					
TZM18H4	11	23740.000	24153.423					
TZM18H5	14	653.500	664.880					
TZM18H6	17	48.100	48.938			Rate (g/m^2-h):		
TZM18H7	20	96.100	97.774			6.95E+00		
TZM18H8	23	154.700	157.394					
TZM18H9	26	1283.000	1305.343					
TZM18H10	29	3812.000	3878.384					
TZM18H11	32	8168.000	8310.242					
TZM18QW	35	1148.000	1167.992					
TZM18CT		932.000						

TZM10: Tested at 700°C for 2 hours (100 sccm flow)

Mo Oxidation Test TZM10 ---- 700C for 2 h at 0.1 liter/min.								Mass Mo ug	Percent in Component
ICP Results	202.0	336.1	339.1	Volume	Mass Mo		RXN CHMBR	6940	22.53
sample	Mo µg/m	Ti µg/mL	Zr µg/mL	sample(mL)	ug		CT&INSRTS	23118	75.04
							QW FLTR	751.2	2.44
TZM 10 E1	1.941	0.001	-0.002	100	194.1				
TZM 10 E2	0.424	-0.002	-0.004	100	42.4		Total	30809.2	
TZM 10 E3	21.705	0.000	0.016	100	2170.5				
TZM 10 E4	139.900	0.012	0.085	100	13990				
TZM 10 E5	3.068	-0.005	-0.006	100	306.8				
TZM 10 E6	16.260	0.000	0.003	100	1626				
TZM 10 E7	6.315	0.001	0.000	100	631.5				
TZM 10 E8	14.420	-0.001	0.000	100	1442				
TZM 10 E9	3.164	0.001	0.002	100	316.4				
TZM 10 E10	5.311	-0.003	-0.001	100	531.1				
TZM 10 E11	9.064	0.012	0.041	100	906.4				
TZM 10 QW	7.512	0.003	0.003	100	751.2				
TZM 10 CT	9.608	-0.001	0.004	100	960.75				
TZM 10 RXN	27.760	0.001	0.017	250	6940				
				Total (ug)	30809				
				Total (mg)	30.8				
			Amount excluding RXN and Col tube (		22908.4				

Bar chart — Percent in Component:
RXN CHMBR, CT&INSRTS, QW FLTR (y-axis 0–100)

	Position in.	Mass Mo ug	Mass Mo (ug) w Col tube redistributed	Temp. x 10	April,28 Temp., C
TZM 10 E1	2	194.1	202.2	6720	672
TZM 10 E2	5	42.4	44.2	7060	706
TZM 10 E3	8	2170.5	2261.5	6790	679
TZM 10 E4	11	13990	14576.7	5990	599
TZM 10 E5	14	306.8	319.7	4750	475
TZM 10 E6	17	1626	1694.2	3850	385
TZM 10 E7	20	631.5	658.0	3270	327
TZM 10 E8	23	1442	1502.5	2820	282
TZM 10 E9	26	316.4	329.7		
TZM 10 E10	29	531.1	553.4		
TZM 10 E11	32	906.4	944.4		
TZM 10 QW	35	751.2	782.7		
TZM 10 CT			960.75		

Specimen (cm^2): 9.112

Rate (g/m^2-h): **16.91**

			Summer'99 Temp.(2),C	Temp. x 10
16	0		659	6590
17	1		676	6760
18	2		683	6830
19	3		690	6900
20	4		699	6990
21	5		708	7080
22	6		707	7070
23	7		696	6960
24	8		685	6850
25	9		671	6710
26	10		648	6480
27	11		609	6090
28	12		563	5630
29	13		518	5180
30	14		476	4760
32	16		415	4150
36	20		319	3190
40	24		255	2550
44	28		220	2200
46	30		174	1740
48	32		96	960
49	33		63	630
50	34		39	390
51	35		30	300
52	36		20	200

Line chart — Micrograms of Mo collected vs. Distance along collection tube, in.
Legend: Mass; Temp.(1)x10; Temp.(2)x10

TZM6: Tested at 700°C for 2 hours (500 sccm flow)

ICP Results sample	202.0 Mo µg/mL	Volume Sample(mL)	Mass Mo ug			Mass Mo ug	Percent in Component
					RXN CHMBR	12560	19.36
TZM 6 D1	3.204	100	320.4		CT&INSRTS	44242	68.19
TZM 6 D2	0.460	100	46		QW FLTR	8080	12.45
TZM 6 D3	0.266	100	26.6				
TZM 6 D4	0.147	100	14.7		Total	64882	
TZM 6 D5	0.120	100	12				
TZM 6 D6	0.102	100	10.2				
TZM 6 D7	0.167	100	16.7				
TZM 6 D8	0.425	100	42.5				
TZM 6 D9	39.470	100	3947				
TZM 6 D10	124.700	100	12470				
TZM 6 D11	92.540	100	9254				
TZM 6 D12	63.440	100	6344				
TZM 6 D13	40.130	100	4013				
TZM 6 D14	22.030	100	2203				
TZM 6 D16	3.568	100	356.8				
TZM 6 D17	2.217	100	221.7				
TZM 6 D18	2.251	100	225.1				
TZM 6 D19	1.680	100	168				
TZM 6 D20	1.423	100	142.3				
TZM 6 D21	1.339	100	133.9				
TZM 6 D22	1.379	100	137.9				
TZM 6 D23	1.428	100	142.8				
TZM 6 D24	1.183	100	118.3				
TZM 6 D25	0.948	100	94.8				
TZM 6 D26	1.080	100	108				
TZM 6 D27	0.836	100	83.6				
TZM 6 D28	0.888	100	88.8				
TZM 6 D29	1.430	100	143				
TZM 6 D30	2.259	100	225.9				
TZM 6 D31	2.759	100	275.9				
TZM 6 D32	4.910	100	491				
TZM 6 D33	5.436	100	543.6				
TZM 6 QW	80.800	100	8080				
TZM 6 Col Tube	18.205	100	1820.5				
TZM 6 RXN	125.6	100	12560				
		Total (ug)	64882				
		Total (mg)	64.9				
	Amount excluding RXN and Col tube (mg)		50502				

C-15
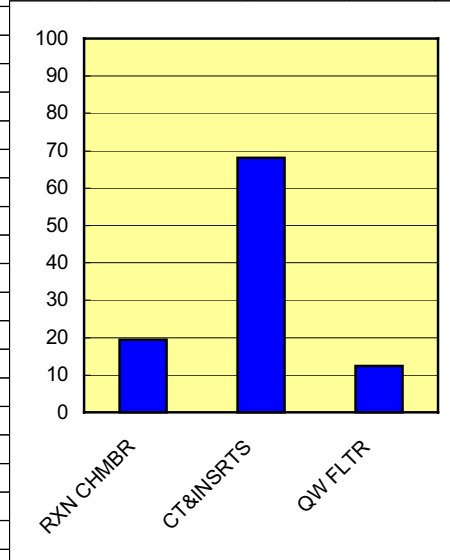
TZM6: Tested at 700°C for 2 hours (500 sccm flow)

	Position in.	Mass Mo ug	Mass Mo,ug w Col tube redistributed	Mass Mo,mg w Col tube redistributed	Temp., C				
TZM 6 D1	1.5	320.4	331.9	0.3319					
TZM 6 D2	2.5	46.0	47.7	0.0477		Specimen (cm^2)			
TZM 6 D3	3.5	26.6	27.6	0.0276		9.075			Summer'99
TZM 6 D4	4.5	14.7	15.2	0.0152					
TZM 6 D5	5.5	12.0	12.4	0.0124			16	0	662
TZM 6 D6	6.5	10.2	10.6	0.0106			17	1	676
TZM 6 D7	7.5	16.7	17.3	0.0173		Rate (g/m^2-h):	18	2	683
TZM 6 D8	8.5	42.5	44.0	0.0440		**35.75**	19	3	690
TZM 6 D9	9.5	3947.0	4089.3	4.0893			20	4	697
TZM 6 D10	10.5	12470.0	12919.5	12.9195			21	5	705
TZM 6 D11	11.5	9254.0	9587.6	9.5876			22	6	704
TZM 6 D12	12.5	6344.0	6572.7	6.5727			23	7	693
TZM 6 D13	13.5	4013	4157.7	4.1577			24	8	683
TZM 6 D14	14.5	2203	2282.4	2.2824			25	9	669
TZM 6 D16	15.5	356.8	369.7	0.3697			26	10	647
TZM 6 D17	16.5	221.7	229.7	0.2297			27	11	609
TZM 6 D18	17.5	225.1	233.2	0.2332			28	12	564
TZM 6 D19	18.5	168	174.1	0.1741			29	13	522
TZM 6 D20	19.5	142.3	147.4	0.1474			30	14	483
TZM 6 D21	20.5	133.9	138.7	0.1387			32	16	418
TZM 6 D22	21.5	137.9	142.9	0.1429			34	18	371
TZM 6 D23	22.5	142.8	147.9	0.1479			36	20	330
TZM 6 D24	23.5	118.3	122.6	0.1226			40	24	265
TZM 6 D25	24.5	94.8	98.2	0.0982			44	28	225
TZM 6 D26	25.5	108	111.9	0.1119			48	32	132
TZM 6 D27	26.5	83.6	86.6	0.0866			49	33	104
TZM 6 D28	27.5	88.8	92.0	0.0920			50	34	76
TZM 6 D29	28.5	143	148.2	0.1482			51	35	60
TZM 6 D30	29.5	225.9	234.0	0.2340			52	36	30
TZM 6 D31	30.5	275.9	285.8	0.2858					
TZM 6 D32	31.5	491	508.7	0.5087					
TZM 6 D33	32.5	543.6	563.2	0.5632					
TZM 6 QW	34	8080	8371.3	8.3713					
TZM 6 Col Tube		1820.5							
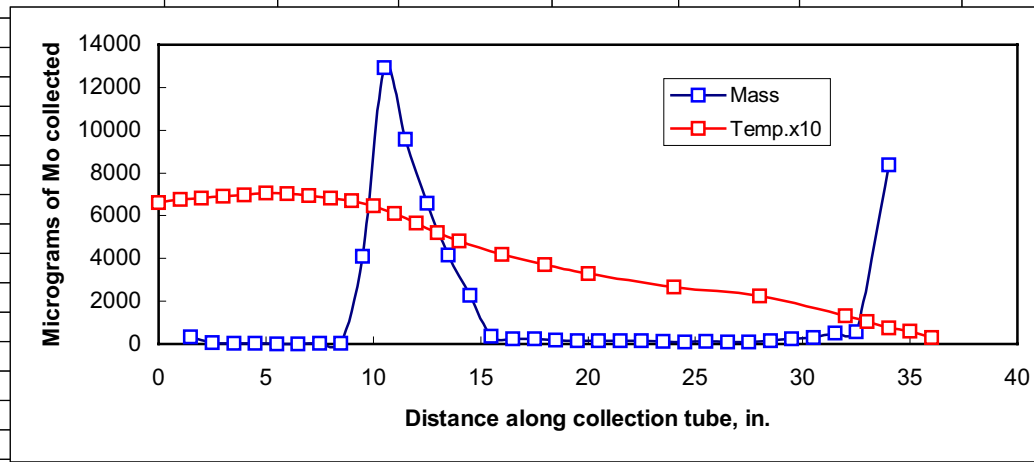
TZM12: Tested at 700°C for 2 hours (1000 sccm flow)

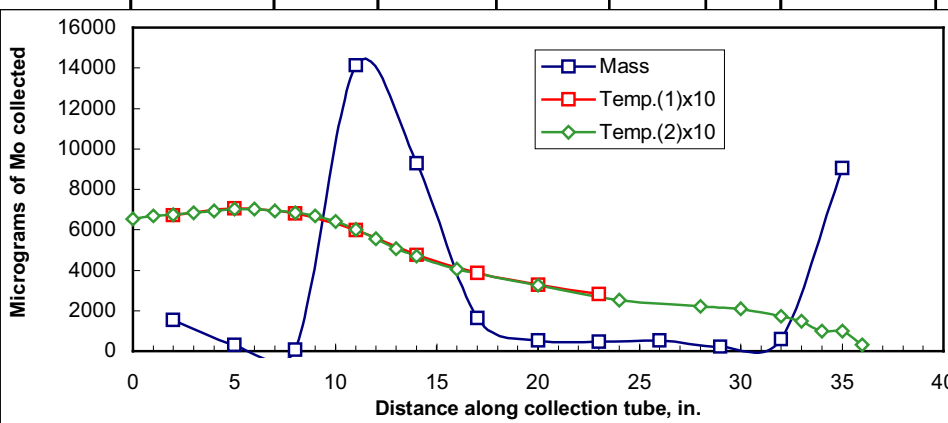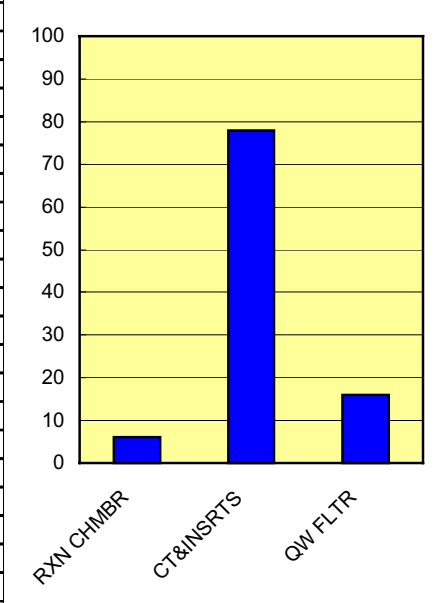|	Mo Oxidation Test TZM12 ---- 700C for 2 h at 1.0 liters/minute										Percent	
						Mass Mo (mg)	April,28			Mass Mo	in	
ICP Results	202.0	Volume	Mass Mo	Position	Mass Mo	w Col tube	Temp.x10	Temp(1).		ug	Component	
sample	Mo µg/mL	Sample(mL)	ug	in.	ug	redistributed	C	C				
									RXN CHMBR	975.25	1.80	
D1	0.957	100	95.7	1.5	95.7	98.7	6720	672	CT&INSRTS	42197.7	77.77	
D2	0.476	100	47.6	2.5	47.6	49.1	6860	686	QW FLTR	11090	20.44	
D3	0.424	100	42.4	3.5	42.4	43.7	6970	697				
D4	0.415	100	41.5	4.5	41.5	42.8	7060	706	Total	54262.95		
D5	0.270	100	27	5.5	27	27.8	7040	704				
D6	0.255	100	25.5	6.5	25.5	26.3	6920	692				
D7	0.422	100	42.2	7.5	42.2	43.5	6790	679				
D8	0.513	100	51.3	8.5	51.3	52.9	6620	662				
D9	14.96	100	1496	9.5	1496	1542.9	6380	638				
D10	115.000	100	11500	10.5	11500	11860.8	5990	599				
D11(avg.)	78.590	100	7859	11.5	7859	8105.6	5550	555				
D12	60.370	100	6037	12.5	6037	6226.4	5100	510				
D13	45.970	100	4597	13.5	4597	4741.2	4750	475				
D14	34.580	100	3458	14.5	3458	3566.5	4400	440				
D16	17.760	100	1776	15.5	1776	1831.7	4130	413				
D17	1.435	100	143.5	16.5	143.5	148.0	3850	385				
D18	0.937	100	93.7	17.5	93.7	96.6	3660	366				
D19	0.977	100	97.7	18.5	97.7	100.8	3470	347				
D20	1.030	100	103	19.5	103	106.2	3270	327				
D21	1.108	100	110.8	20.5	110.8	114.3	3080	308				
D22	1.237	100	123.7	21.5	123.7	127.6	2950	295				
D23	1.271	100	127.1	22.5	127.1	131.1	2820	282				
D24	1.184	100	118.4	23.5	118.4	122.1	2690	269				
D25	1.077	100	107.7	24.5	107.7	111.1	2560	256				
D26	1.221	100	122.1	25.5	122.1	125.9						
D27	0.981	100	98.1	26.5	98.1	101.2						
D28	1.035	100	103.5	27.5	103.5	106.7						Temp.(2)
D29	1.651	100	165.1	28.5	165.1	170.3					Summer'99	x10
D30	2.672	100	267.2	29.5	267.2	275.6			16	0	662	6620
D31	3.994	100	399.4	30.5	399.4	411.9			17	1	676	6760
D32	4.802	100	480.2	31.5	480.2	495.3			18	2	683	6830
D33	8.193	100	819.3	32.5	819.3	845.0			19	3	690	6900
Quartz Wool	110.900	100	11090	34	11090	11437.9			20	4	697	6970
ZM12 CT-D	16.210	100	1621						21	5	705	7050
ZM12 RXN	3.901	250	975.25						22	6	704	7040
						Specimen (cm²)			23	7	693	6930
		Total (ug)	54263.0			10.343			24	8	683	6830
		Total (mg)	54.263						25	9	669	6690
						Rate (g/m²-h):			26	10	647	6470
	Amount excluding RXN and Col tube (u		51666.7			26.23			27	11	609	6090
									28	12	564	5640
									29	13	522	5220
									30	14	483	4830
									32	16	418	4180
									34	18	371	3710
									36	20	330	3300
									40	24	265	2650
									44	28	225	2250
									48	32	132	1320
									49	33	104	1040
									50	34	76	760
									51	35	60	600
									52	36	30	300

Bar chart: RXN CHMBR, CT&INSRTS, QW FLTR. Line chart: Micrograms of Mo collected vs Distance along collection tube, in. Legend: Mass, Temp.(1)x10, Temp.(2)x10.
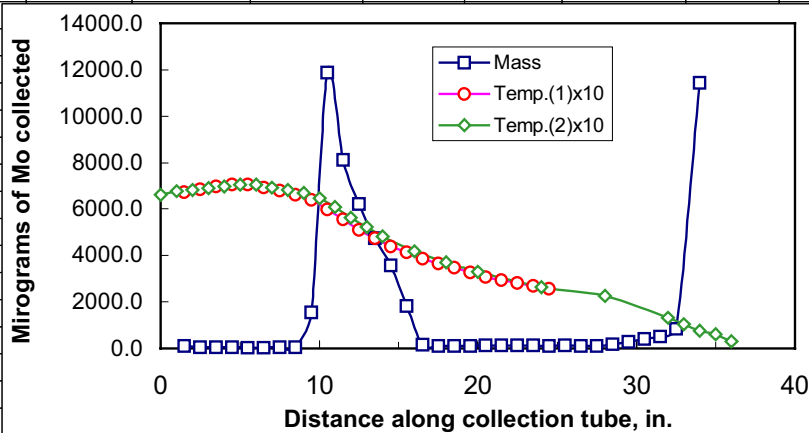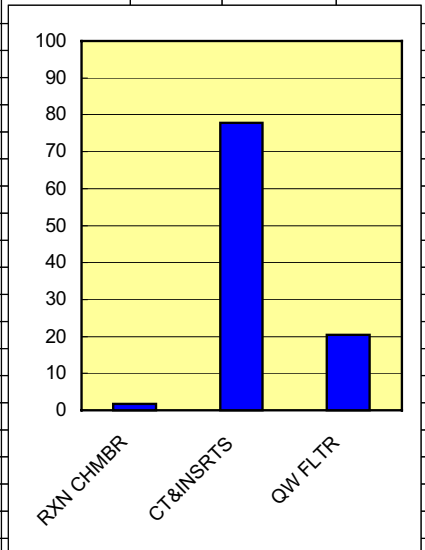
TZM9: Tested at 700°C for 2 hours (2500 sccm flow)

								Mass Mo ug	Percent in Component	
	Mo Oxidation Test TZM9 ---- 700 for 2 h at 2.5 liters/minute									
ICP Results	202.0	336.1	339.1	Volume	Mass Mo					
sample	Mo µg/mL	Ti µg/mL	Zr µg/mL	sample(mL)	ug	RXN CHMBR		3442.5	6.07	
						CT&INSRTS		44212.8	77.97	
TZM 9 H1	15.28	-0.007	0.004	100	1528	QW FLTR		9050	15.96	
TZM 9 H2	3.031	-0.008	-0.002	100	303.1					
TZM 9 H3	0.683	-0.004	-0.002	100	68.3	Total		56705.3		
TZM 9 H4	141.400	0.001	0.062	100	14140					
TZM 9 H5	92.780	0.000	0.039	100	9278					
TZM 9 H6	16.260	-0.004	0.005	100	1626					
TZM 9 H7	5.268	-0.004	-0.001	100	526.8					
TZM 9 H8	4.558	-0.007	-0.002	100	455.8					
TZM 9 H9	5.224	-0.006	-0.002	100	522.4					
TZM 9 H10	2.233	-0.006	-0.002	100	223.3					
TZM 9 H11	5.811	-0.005	-0.001	100	581.1					
TZM 9 QW	90.500	0.001	0.039	100	9050					
TZM 9 CT	149.600	0.006	0.068	100	14960					
TZM 9 RXN	13.770	-0.002	0.005	250	3442.5					
			Total (ug)	56705						
			Total (mg)	56.7						
			Amount excluding RXN and Col tube	38302.8						

	Position in.	Mass Mo ug	Mass Mo (ug) w Col tube redistributed	Temp. x 10	April,28 Temp., C					
TZM 9 H1	2	1528	2124.8	6720	672					
TZM 9 H2	5	303.1	421.5	7060	706					
TZM 9 H3	8	68.3	95.0	6790	679					Temp.(2)
TZM 9 H4	11	14140	19662.7	5990	599	Specimen (cm²)			Summer'99	x10
TZM 9 H5	14	9278	12901.7	4750	475	9.069	16	0	652	6520
TZM 9 H6	17	1626	2261.1	3850	385		17	1	668	6680
TZM 9 H7	20	526.8	732.6	3270	327		18	2	675	6750
TZM 9 H8	23	455.8	633.8	2820	282		19	3	683	6830
TZM 9 H9	26	522.4	726.4			Rate (g/m²-h):	20	4	692	6920
TZM 9 H10	29	223.3	310.5			31.26	21	5	701	7010
TZM 9 H11	32	581.1	808.1				22	6	701	7010
TZM 9 QW	35	9050	12584.7				23	7	692	6920
TZM 9 CT		14960					24	8	682	6820
							25	9	667	6670
							26	10	641	6410
							27	11	601	6010
							28	12	554	5540
							29	13	505	5050
							30	14	470	4700
							32	16	404	4040
							36	20	326	3260
							40	24	250	2500
							44	28	222	2220
							46	30	207	2070
							48	32	172	1720
							49	33	148	1480
							50	34	99	990
							51	35	98	980
							52	36	30	300

TZM3: Tested at 750°C for 1 hour (1000 sccm flow)

These are the analyses associated with Post-test TZM3 (750C/1 h)							Percent	
						Mass Mo	in	
Mo Oxidation Test TZM3 ---- 750C for 1 h at 1.0 liters/minute						ug	Component	
ICP Results	202.0	Volume	Mass Mo	RXN CHMBR		46775	13.52	
sample	Mo µg/mL	Sample(mL)	ug	CT&INSRTS		245628.1	70.98	
				QW FLTR		53640	15.50	
TZM3 G1	4.589	100	458.9					
TZM3 G2	1.331	100	133.1	Total		346043.1		
TZM3 G3	3.981	100	398.1					
TZM3 G4	113.500	100	11350					
TZM3 G5	126.400	100	12640					
TZM3 G6	69.250	100	6925					
TZM3 G7	60.700	100	6070					
TZM3 G8	55.420	100	5542					
TZM3 G9	65.510	100	6551					
TZM3 G10	219.300	100	21930					
TZM3 G11	1617.000	100	161700					
TZM3 QW	536.400	100	53640					
TZM3 Col Tube	119.300	100	11930					
TZM3 RXN	187.100	250	46775					
		Total (ug)	346043					
		Total (mg)	346.0					
Amount excluding RXN and Col tube (ug):			287338.1					

	Position	Mass Mo	Mass Mo (ug) w Col tube redistributed	Mass Mo (mg) w Col tube redistributed				
	in.	ug						
TZM3 G1	2	459	478.0	0.4780		Specimen (cm^2)		
TZM3 G2	5	133	138.6	0.1386		9.056		
TZM3 G3	8	398	414.6	0.4146				
TZM3 G4	11	11350	11821.2	11.8212				
TZM3 G5	14	12640	13164.8	13.1648				
TZM3 G6	17	6925	7212.5	7.2125		Rate (g/m^2-h):		
TZM3 G7	20	6070	6322.0	6.3220		382.11		
TZM3 G8	23	5542	5772.1	5.7721				
TZM3 G9	26	6551	6823.0	6.8230				
TZM3 G10	29	21930	22840.5	22.8405				
TZM3 G11	32	161700	168413.6	168.4136				
TZM3 QW	35	53640	55867.1	55.8671				
TZM3 Col Tube		11930						

Right-side temperature data:

		12-Jul	Temp.,
		Temp.	x 0.2
16	0	706	141.2
17	1	720	144
18	2	727	145.4
19	3	735	147
20	4	744	148.8
21	5	753	150.6
22	6	752	150.4
23	7	741	148.2
24	8	732	146.4
25	9	717	143.4
26	10	695	139
27	11	661	132.2
28	12	616	123.2
29	13	566	113.2
30	14	529	105.8
32	16	455	91
34	18	408	81.6
36	20	361	72.2
40	24	271	54.2
44	28	248	49.6
48	32	145	29
49	33	106	21.2
50	34	80	16
51	35	64	12.8
52	36	30	6
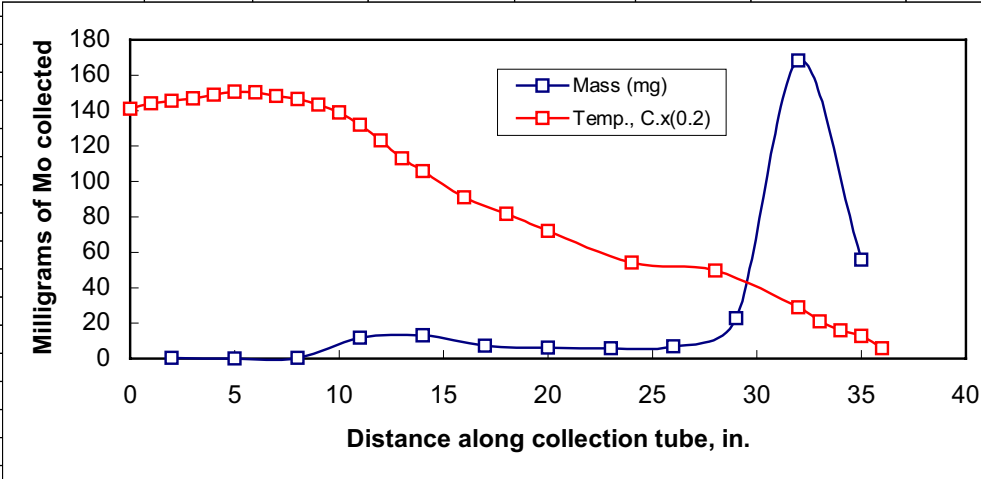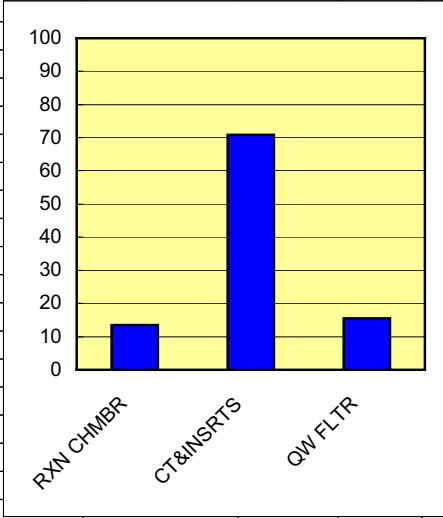
TZM2: Tested at 800°C for 1 hour (1000 sccm flow)

Mo Oxidation Test TZM2 ---- 800C for 1 h at 1.0 liters/minute						Mass Mo ug	Percent in Component
ICP Results sample	202.0 Mo µg/mL	Volume Sample(mL)	Mass Mo ug				
				RXN CHMBR		518000	23.35
TZM 2 E1	6.67	100	667	CT&INSRTS		1604391	72.32
TZM 2 E2	53.78	100	5378	QW FLTR		95960	4.33
TZM 2 E3	121.5	100	12150				
TZM 2 E4	13030	100	1303000	Total		2218351	
TZM 2 E5	433.6	100	43360				
TZM 2 E6	522.4	100	52240				
TZM 2 E7	67.48	100	6748				
TZM 2 E8	46.74	100	4674				
TZM 2 E9	55.94	100	5594				
TZM 2 E10	1435	100	143500				
TZM 2 QW	959.6	100	95960				
TZM 2 Col Tube	270.8	100	27080				
TZM 2 RXN	2072	250	518000				
		Total (ug)	2218351				
		Total (mg)	2218.4				
	Amount excluding RXN and Col tube (mg)		1673271				

	Position in.	Mass Mo ug	Mass Mo,ug w Col tube redistributed	Mass Mo,mg w Col tube redistributed
TZM 2 E1	2	667.0	677.8	0.6778
TZM 2 E2	5	5378.0	5465.0	5.4650
TZM 2 E3	8	12150.0	12346.6	12.3466
TZM 2 E4	11	1303000.0	1324087.6	1324.0876
TZM 2 E5	14	43360.0	44061.7	44.0617
TZM 2 E6	17	52240.0	53085.4	53.0854
TZM 2 E7	20	6748.0	6857.2	6.8572
TZM 2 E8	23	4674.0	4749.6	4.7496
TZM 2 E9	26	5594.0	5684.5	5.6845
TZM 2 E10	29	143500.0	145822.4	145.8224
TZM 2 QW	35	95960.0	97513.0	97.5130
TZM 2 Col Tube		27080.0		

Specimen (cm^2): 9.15

Rate (g/m^2-h): **2424.43**

		July,12 Temp.	Temp. x2
16	0	777	1554
17	1	790	1580
18	2	793	1586
19	3	801	1602
20	4	807	1614
21	5	815	1630
22	6	811	1622
23	7	797	1594
24	8	785	1570
25	9	770	1540
26	10	748	1496
27	11	713	1426
28	12	666	1332
29	13	624	1248
30	14	587	1174
32	16	522	1044
34	18	466	932
36	20	417	834
40	24	344	688
44	28	296	592
48	32	175	350
49	33	130	260
50	34	90	180
51	35	72	144
52	36	30	60