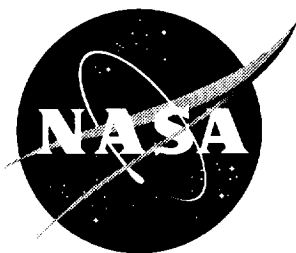NASA Contractor Report 201753

Oxidation and Emittance Studies of Coated Mo-Re

David E. Glass
Analytical Services and Materials, Inc., Hampton, Virginia

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David E. Glass
Analytical Services & Materials, Inc.
Hampton, VA 23666

Abstract

A commercially available Fe-Cr-Si coating (R512E) and a silicide coating were evaluated regarding their ability to protect Mo-Re from oxidation. The R512E coating provided very good oxidation protection at 1260°C in atmospheric air. Oxidation tests were also performed at Mach 4 in the HYMETS facility at NASA Langley Research Center where again the R512E provided good oxidation protection but for much shorter times. Emittance measurements as a function of wavelength were also obtained for the R512E coating on Mo-Re after exposure to the Mach 4 environment at 1150°C and 1230°C.

Introduction

Mo-Re oxidizes rapidly in air at elevated temperatures (incipient oxidation of Mo occurs at 327°C [1]). The oxides that are formed by the oxidation of Mo-Re are non protective, and thus significant mass loss occurs during relatively short times at elevated temperatures. Silicide coatings have been used with success to protect Mo from oxidation. The oxidation protection of Mo-Re, however, appears to be less characterized than that of pure Mo.

The phase diagram of Mo and Si shows three intermetallic compounds, Mo_3Si, Mo_5Si_3, and $MoSi_2$. $MoSi_2$ has the highest Si content, is the most oxidation resistant, and has a metallic conductivity [2]. The oxidation resistance of $MoSi_2$ is due to the formation of silica when the $MoSi_2$ is exposed to an oxidizing environment at high temperatures [3]. The oxidation rate of $MoSi_2$ is controlled by the diffusion of oxygen through a SiO_2 layer. An interlayer of Mo_5Si_3 is sometimes present between the $MoSi_2$ and the SiO_2 layers. At temperatures below 800°C the oxidation of $MoSi_2$ is governed by the reaction

$$2MoSi_2 + 7O_2 \text{ --> } 2\,MoO_3 + 4SiO_2$$
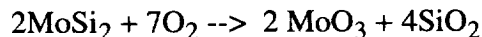
and above 800°C by the reaction

$$5MoSi_2 + 7O_2 \text{ --> } Mo_5Si_3 + 7SiO_2$$
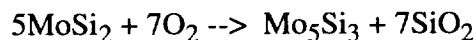
Above 800°C, the formation of Mo_5Si_3 occurs between the SiO_2 and the $MoSi_2$ layers. The metal rich interlayers, such as the Mo_5Si_3, act as a diffusion barrier for the silicon diffusing into the base metal [4]. It is the diffusion of the silicon into the base metal that limits the life of the coating at high temperatures.

Below 800°C, $MoSi_2$ oxidizes to form SiO_2 and MoO_3 and often will disintegrate in an oxidizing atmosphere [3]. At these temperatures, the oxidation product does not form a protective silica layer as it does at higher temperatures, and "pesting" occurs. The

Mo oxides form a solid powder-like oxide on the surface, preventing the amorphous silicon oxide from forming a continuous glassy layer. Several approaches to reducing pesting are possible. If the $MoSi_2$ is preoxidized at temperatures above 1300°C in air, the "pest" does not form at lower temperatures. Pesting has been linked to the presence of impurities and microcracks, believed to be introduced during processing [5]. Recent results indicate that a fully dense (> 95 percent), crack-free $MoSi_2$ coating does not pest. The addition of Ge to the $MoSi_2$ appears to suppress low temperature oxidation by the formation of a lower viscosity glass phase [6]. This lower viscosity glass enhances oxidation protection at low temperatures and seems to help prevent pesting and eliminate spallation, increasing the life of the coating by an order of magnitude.

Pesting is not a problem at high temperatures because $MoSi_2$ grows pure SiO_2 protective scales which are volatile at low temperatures and evaporate during the oxidation process. [6] In addition to pesting, $MoSi_2$ coatings undergo both passive and active oxidation in the presence of oxygen. Passive oxidation of the $MoSi_2$ coating occurs if sufficient oxygen is available at the surface and the $MoSi_2$ forms a protective film of SiO_2. Once the coating is sufficiently depleted of Si, a lower silicide (Mo_5Si_3) remains which does not form protective silica scales and oxidizes rapidly. In most cases, the diffusion of Si through the $MoSi_2$ is faster than the diffusion of oxygen through the SiO_2 layer, and no Mo_5Si_3 interlayer results. Under these conditions, the protective SiO_2 layer is formed, and the oxidation is referred to as passive oxidation. During the initial oxidation of $MoSi_2$, the oxygen partial pressure is high, and the volatile MoO_3 is formed. However, after the SiO_2 layer is formed, the oxygen partial pressure at the $MoSi_2$ and SiO_2 interface is too low for the oxidation of the Mo, and only the Si is oxidized due to its higher oxygen affinity (called passive oxidation).

Active oxidation is another aspect of $MoSi_2$ oxidation. At low oxygen pressures (due to a low total pressure of O_2 or to a dilute concentration of oxygen in an inert carrier gas) the oxygen will not be sufficient to support a protective SiO_2 film and SiO (v) becomes the stable specie. Active oxidation is referred to as the condition where Mo oxides are formed in addition to the SiO_2. The large Mo oxides can cause the SiO_2 layer to be porous. Mo is known to absorb oxygen from the environment and become brittle when exposed to a high temperature inert gas containing a small amount of oxygen [7].

Tuominen and Dahl [8] studied oxidation coatings for use on Mo and the Mo alloy TZM since at high temperatures the oxide formed, MoO_3, is volatile. $MoSi_2$ was found to be a good coating with an effective life longer than the time required for the $MoSi_2$ to be converted to Mo_5Si_3, which is a non-protective phase. There is a difference in the coefficient of thermal expansion (CTE) between Mo (5.6 μm/m-°C) and $MoSi_2$ (8.6 μm/m-°C) as measured from -18°C to 1200°C. At temperatures above 1205°C, interdiffusion between the Mo and Si limits the coating life, while the presence of cracks in the coating may limit the coating life at temperatures below 980°C. $MoSi_2$ is suitable for applications in still air for at least 100 hours under low cycle conditions up to 1315°C. $MoSi_2$, SiC, and Al_2O_3 can improve oxidation resistance of Mo only within a time range of hours above 1000°C [9]. Rexer [10], however, states that a 125-μm-thick Ir coating will last for at least 600 hours on Mo at 1700°C.

Clark and Wallace [11] studied the static and dynamic oxidation of Pt-clad Mo-47Re. A platinum foil was diffusion bonded to Mo-Re disks for the oxidation testing. They determined that the Pt foil provided good protection from dynamic oxidation for up to approximately 12.5 hours at 1260°C. No separation of the Pt cladding from the substrate was experienced during the 12.5 hour dynamic oxidation in the HYMETS facility at NASA Langley Research Center.

Results of oxidation studies on Mo-Re are presented in this report. Oxidation tests were performed on arc cast and powder metallurgy Mo-Re to evaluate a commercial coating (R512E) and a silicide coating. The two coatings were tested in still air and at Mach 4 in the HYMETS facility at NASA Langley. The primary means of evaluation was weight change. Emittance measurements on the coated samples were performed at various times after exposure in the HYMETS facility.

Experimental Approach

The objective of this study was to evaluate the effectiveness of two coatings for the reduction of oxidation on Mo-Re at elevated temperatures. The study was performed on both arc cast and powder metallurgy Mo-Re using two different coatings. The specimens and coatings used for the study are briefly discussed here along with the test methods, which included oxidation in an atmospheric pressure furnace and in a Mach 4 arc jet facility. Emittance measurements were also conducted on one of the coatings.

Specimens

Three different Mo-Re alloys were used for the oxidation studies. Tests were performed with powder metallurgy (PM) Mo-49Re circular tubes supplied by Sandvik Rhenium Alloys and arc cast Mo-41Re tubes. The Mo-41Re tubes, with a "D-shaped" cross section, were drawn from an arc cast billet fabricated by Amax Research and Development Center, CO. The "D-shaped" tubes had an inner radius of 0.76 cm and a wall thickness of 0.0254 cm. Various lengths and sections (some flat) of the "D-shaped" tubing were cut and used for the oxidation tests.
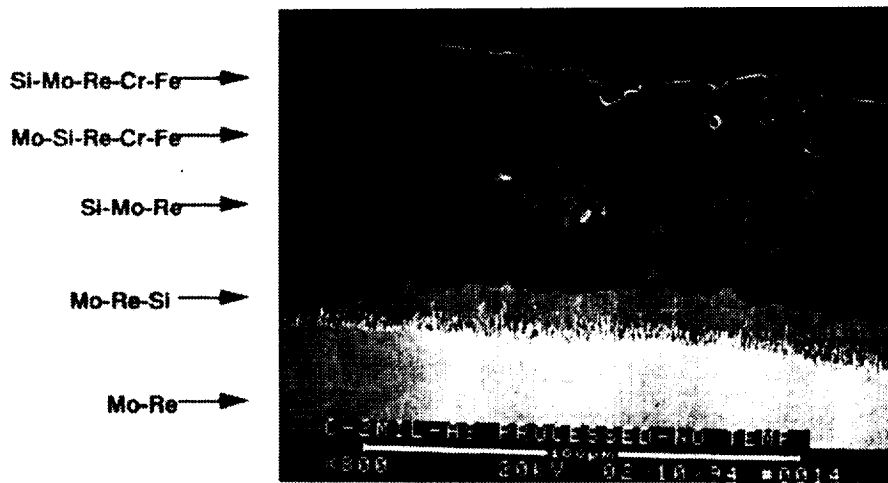

Figure 1: Photomicrograph of an as coated (R512E) Mo-41Re sample during electron microprobe characterization.

Flat, circular coupons, with a 2.03-cm diameter were used for the dynamic oxidation and emittance tests. The coupons were produced from a Mo-47Re sheet 0.051-cm thick. The Mo-47Re sheet was obtained from Climax Specialty Metals in a stress relieved condition.

Coatings

The R512E coating is a Fe-Cr-Si coating applied by Hitemco, Bethpage, NY. The

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coating is applied via a slurry process at 1427°C for approximately 1 hour. The process also includes fusing, spraying, dipping, and firing. Approximately half of the coating thickness is converted from the substrate and half is added to the outer surface during the coating process. Figure 1 shows a cross section of an as coated Mo-41Re tube evaluated with a scanning electron microscope (SEM). The composition of each layer was determined using an electron probe micro analyzer and is identified in the figure.

The Mo-Re alloys were also coated with a silicide coating, $Mo(Si_2, Ge)$. The coating was applied by a pack cementation process by Dr. Bob Rapp at Ohio State University (OSU). The tube sections were ultrasonically cleaned prior to coating. Coating thicknesses varied from 25 to 75 μm. In several cases, the coated specimens were heated in a vacuum prior to oxidation to determine if oxidation protection was improved as a result.

Oxidation

The oxidation protection of the coatings was determined by performing both static and dynamic oxidation tests. The specimens were either circular tubes, "D-shaped" tubes, or flat disks.

Range of test conditions:

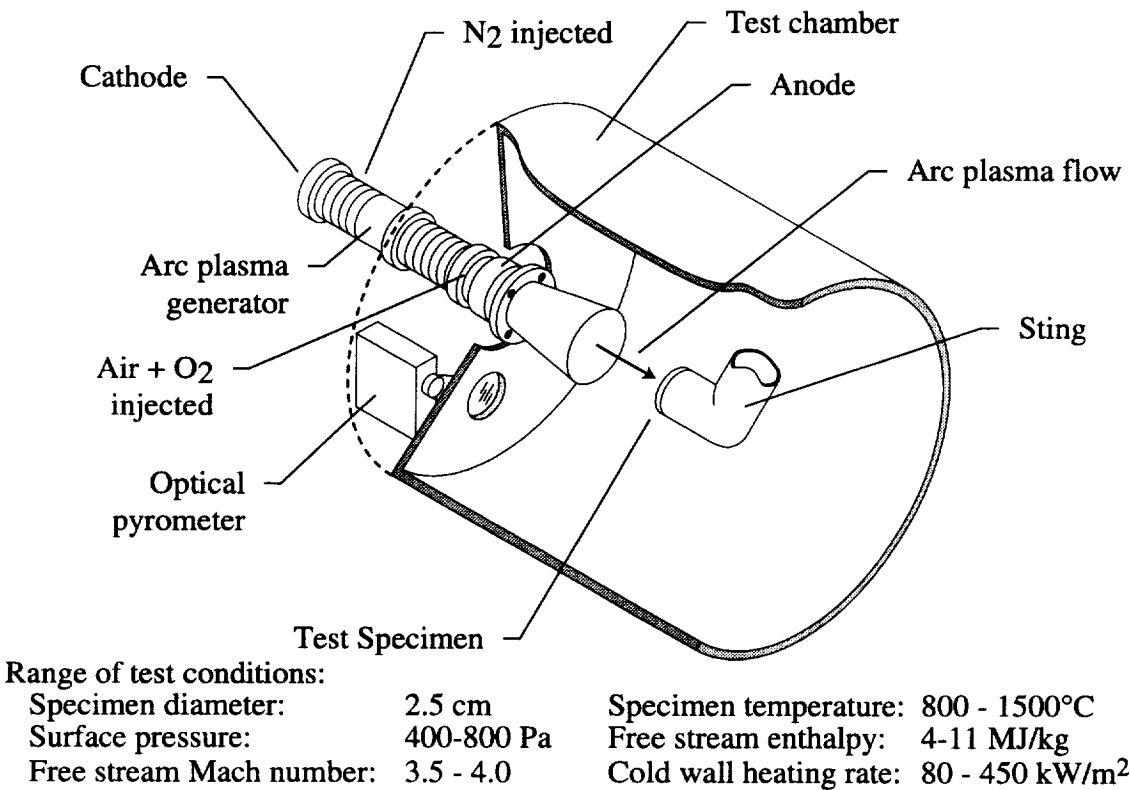|			
Specimen diameter:	2.5 cm	Specimen temperature:	800 - 1500°C
Surface pressure:	400-800 Pa	Free stream enthalpy:	4-11 MJ/kg
Free stream Mach number:	3.5 - 4.0	Cold wall heating rate:	80 - 450 kW/m^2

Figure 2: Schematic drawing of the HYMETS facility at NASA Langley Research Center.

After the tubes were prepared for oxidation, they were oxidized in a furnace in air at 1260°C at 1 atmosphere. The specimens were placed on an alumina block and placed in the furnace for specified times. After the desired time had expired, the samples were removed, allowed to cool, and weighed. Depending on the condition of the specimens, they were then either reinserted in the furnace for further oxidation or removed from the testing sequence.

Oxidation measurements were also performed at Mach 4 using a NASA Langley arc jet wind tunnel with sufficiently high enthalpies to simulate the dissociated airflow encountered in hypersonic flight. A schematic diagram of the arc-jet wind tunnel used for dynamic oxidation is shown in Figure 2. The range of test conditions available in the HYMETS facility are also shown in the figure.

The HYMETS is an electric arc-heated wind tunnel that uses air plus nitrogen and oxygen ratios required to produce the test environment. The flow of gases is normal to the sample and the sample temperature is monitored using a radiometer focused on its front surface. The power to the arc is adjusted to control the sample surface temperature. [11]

Emittance

Emittance is an optical property influencing radiative heat transfer. High emittances are required for efficient dissipation of heat. Emittance is important because of its impact on the integrated thermal loads experienced by the substrate. Emittance of coated samples was measured using a government-furnished heated-cavity reflectometer. A detailed description of the emittance measurement experimental procedure is described in ref. 12.

Reflection measurements as a function of wavelength can be used to calculate the total emittance of the surface using Kirchhoff's rule and integrating the spectral emittance against the blackbody radiation curve for a given temperature. The total hemispherical emittance can be calculated from measurements of the spectral emittance using the following expression:

$$\varepsilon_{TN} = \frac{\int \varepsilon(\lambda) \, M_b(\lambda, T) \, d\lambda}{\int M_b(\lambda, T) \, d\lambda}$$

The total hemispherical emittance is a function of temperature since the emittance is a function of wavelength.

Discussion of Results

Oxidation studies were performed using both static and dynamic oxidation from 1150°C to 1260°C. The R512E coating provided far superior oxidation protection, protecting Mo-41Re for hours compared to only minutes for the $Mo(Si_2, Ge)$ coating. Emittance measurements were also conducted on the R512E coating.

$Mo(Si_2, Ge)$

Several samples of arc-cast Mo-41Re specimens were coated with the silicide coating at OSU. Prior to the coating process, the specimens were tumbled for several days in a tumbler (similar to that used by jewelers to polish rocks) to round the edges. After tumbling, the specimens were cleaned and sent to OSU for coating.

A flat Mo-41Re specimen (cut from a "D-shaped" tube) with the $Mo(Si_2, Ge)$ coating was heated at 1260°C for two 15-minute intervals, and weighed between each heating. The initial weight was 0.58029 g. After the first heating for 15 minutes at 1260°C, the weight was 0.48731 g. After the second heating, the weight was 0.42493 g.

Due to the significant weight loss, the testing was stopped. Next, a 0.64-cm-long section of the coated tubing was heated in air at 1260°C. When the section of tube was removed from the furnace after 420 minutes of heating, a small hole could be seen in the tube and the tube seemed to have "delaminated" in certain areas. As the tube cooled to room temperature, it fell apart. Another sample was heated for multiple cycles in air at 1260°C. After a total heating time of 105 minutes, the sample began to deteriorate. A small hole was observed through the Mo-Re. As in previous cases, the sides of the specimen experienced the most oxidation.
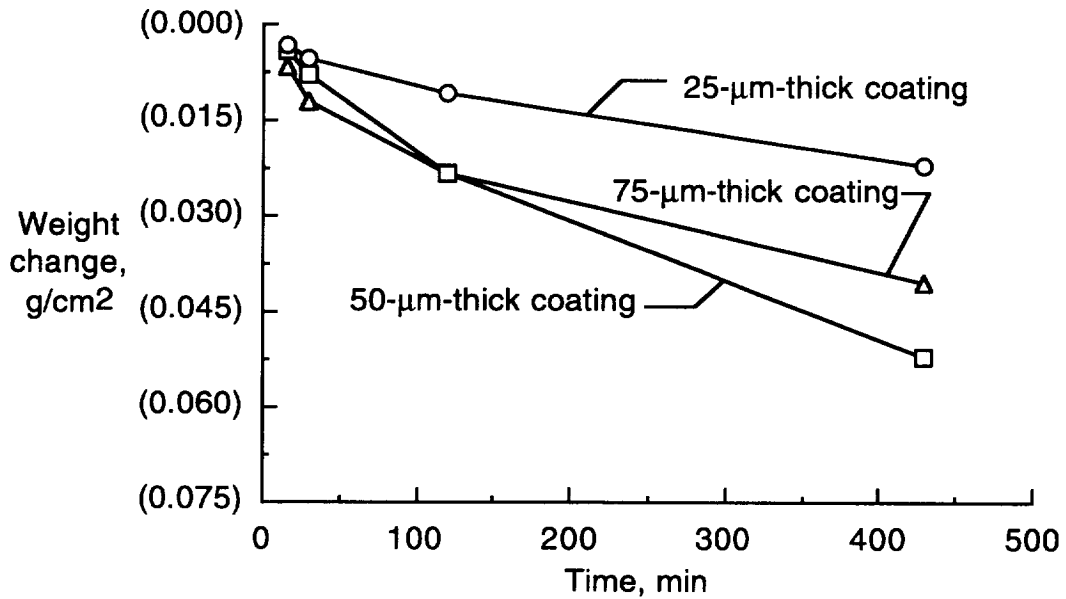

Figure 3: Weight of coated Mo-49Re tubes after cyclic oxidation as a function of coating thickness.

Several 1-in-long samples of the Mo-49Re PM tubes coated with 25, 50, and 75 μm of a Mo(Si$_2$, Ge) coating at OSU were heated in an air furnace at 1260°C. The samples were periodically removed from the furnace, allowed to cool to room temperature, and weighed. The weight of the samples as a function of heating time is shown in Figure 3. The sample with the thinnest coating, 25 μm, had the smallest weight loss at the end of the heating cycles. On all three samples, the weight loss appeared to be from oxidation, not from spallation. After the final heating cycle, the tubes with the 50 and 75-μm-thick coating both had cracks visible to the naked eye. In the 50-μm-thick coating, the crack went through the entire thickness of the tube. The crack in the 75-μm-thick coating did not penetrate the tube. The tube with the 25-μm-thick coating did not appear to have any large cracks in the coating. The tube with the 75-μm-thick coating appeared to have more of a matte finish on it than the 25 and 50-μm-thick coatings, which appeared shinier. Due to the greater problems with the thicker coatings, it appeared that the thicker coating may have a greater problem with the CTE mismatch with the Mo-Re substrate than the thinner 50-μm-thick coating. On all three tubes, it appears that the oxidation of the tube started at the ends of the tube where the tubes were cut, and worked its way in toward the center of the tube. After the final heating, both ends of the tube with the 50-μm-thick coating broke off of the tube, and a section of the tube with the 75-μm-thick coating broke off of one end of the tube. A small piece of one end of the tube with the 50-μm-thick coating chipped off.
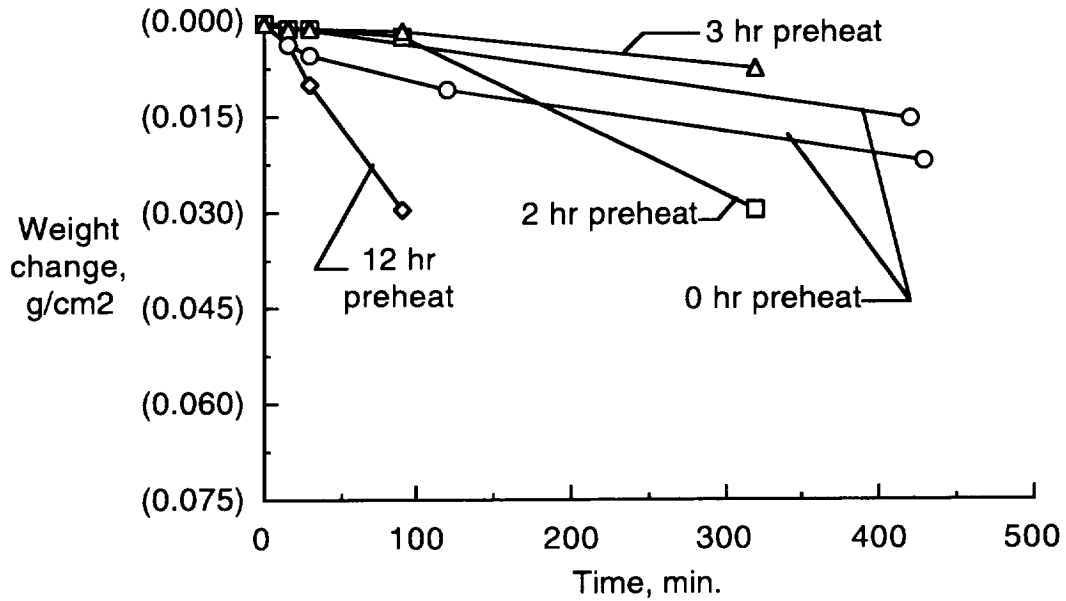
Figure 4: Weight change of Mo-49Re specimens with 25-μm-thick coating and various length preheats at 1250°C.

An attempt was made to build up the diffusion layer between the coating and the substrate to evaluate its effect on the oxidation. Toward this end, the coated tubes were heated in a vacuum at 1250°C for 3 hours. The tubes were then cycled at 1260°C in a 1-atmosphere environment.
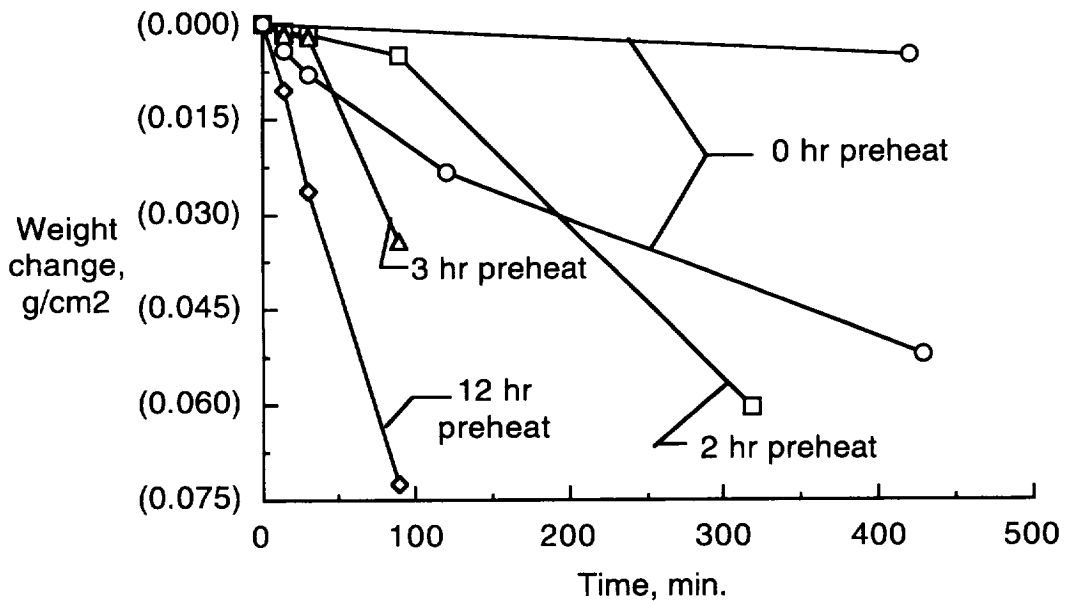

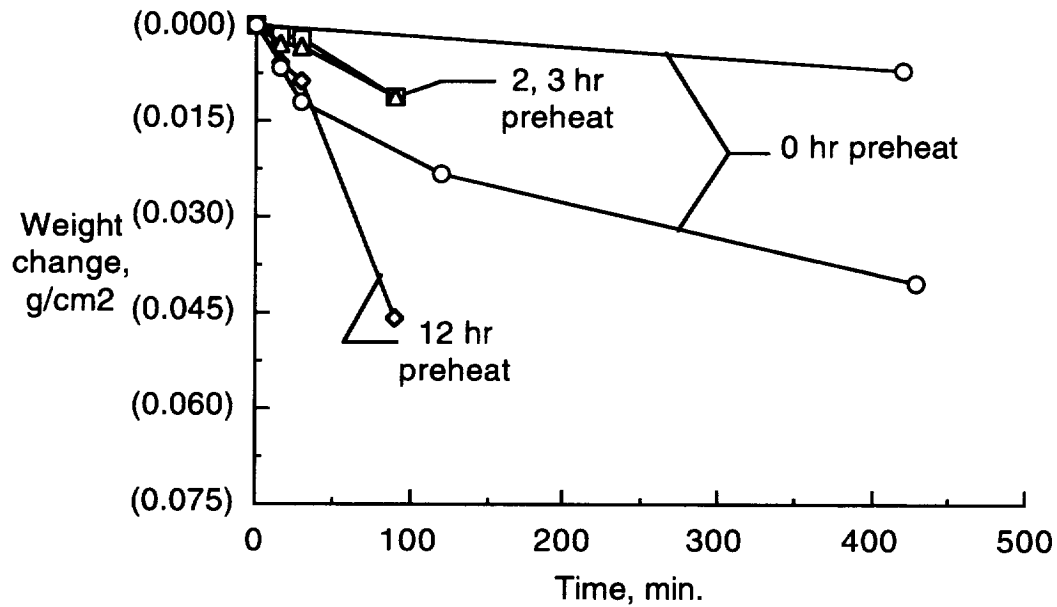Figure 5: Weight change of Mo-49Re specimens with 50-μm-thick coating and various length preheats at 1250°C.

Figure 6:   Weight change of Mo-49Re specimens with 75-μm-thick coating and various length preheats at 1250°C.

A thicker diffusion layer was also created by heating tubes with 25, 50, and 75-μm-thick coatings at 1250°C in a vacuum for 12 hours. The tubes were then exposed to the same thermal cycling. At the end of the thermal cycling, the tubes with the 50 and 75 μm thick coating completely crumbled when an attempt was made to pick them up for weighing. Both edges of the tube with the 25-μm-thick coating also crumbled, but the middle 1 cm of the tube remained rigid. No weight values were obtained at the end of the thermal cycling.

An estimate of the pre-heating time necessary to give a minimum weight loss was performed. The weight loss after two 15 minute cycles for the tubes with 25-μm-thick coatings were plotted versus the square root of the time for 0, 3, and 12 hours of preheating. The data were then curve fit, and the minimum of the curve occurred at a time of 2 hours. The final three tubes were then pre-heated for 2 hours at 1250°C in a vacuum.

Figure 4 - Figure 6 show the change in mass of the heated specimens as a function of preheat time for coatings 25, 50, and 75-μm thick, respectively. For each coating thickness, two tests were performed with no preheat. One was cyclic and the other was continuous. In all cases, the mass loss for the continuous heating was significantly less than for the cyclic heating. For short times, preheating the specimens in a vacuum prior to oxidation reduced the weight loss, but in general, the protection provided with pre-heating did not provide a significant advantage and in some cases was a disadvantage.

R512E Coating

Two 0.64-cm-long sections of a Mo-41Re "D-shaped" tube were coated by Hitemco with their standard R512E coating. One tube was coated with 25 μm and the second tube was coated with 50 μm of the coating. The tube sections were oxidized at 1 atmosphere in a furnace at 1260°C. The change in weight per unit area as a function of heating time is shown in Figure 7. The surface area of the tube section was approximately 5.262 cm^2.
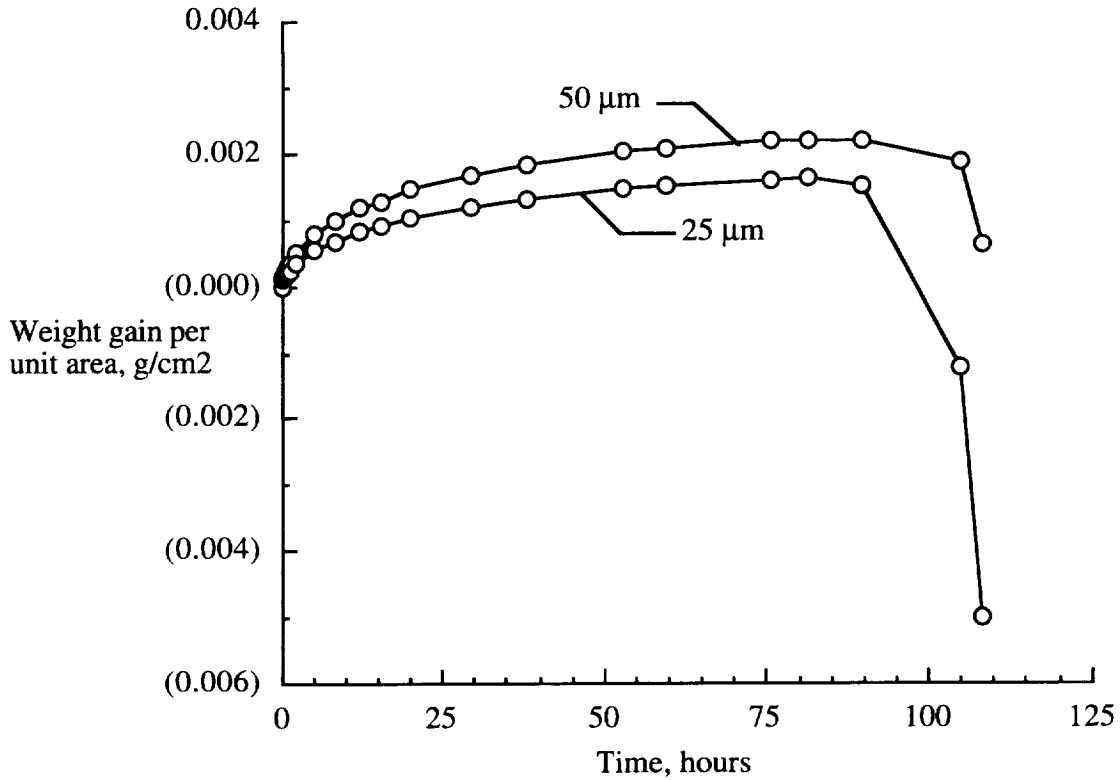
Figure 7:   Weight gain of R512E coated Mo-41Re as a function of time when heated in
air at 1 atm and 1260°C.

The tests were conducted for over 100 hours.   The sample with the 50-μm-thick
coating performed slightly better than the 25-μm-thick coating, but the performance of
the two was similar.   In both cases, a weight gain was observed for nearly 100 hours until
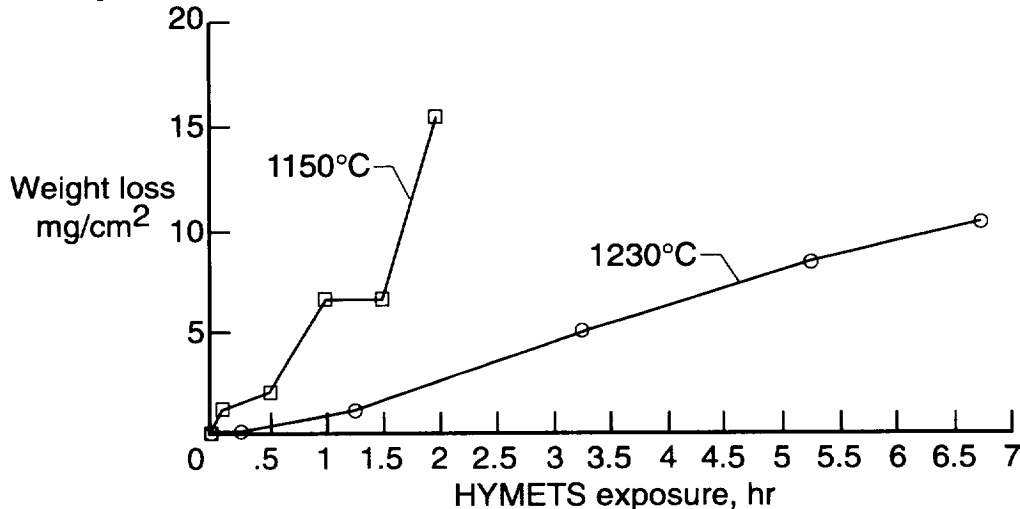a rather sharp decrease in the weight occurred.

Figure 8:   Weight loss of R512E coated Mo-47Re as a function of exposure time in the
HYMETS facility at Mach 4.

The R512E coating was also tested on Mo-47Re.   Several 1-in-diameter coupons
were coated with the R512E coating and tested in the NASA Langley HYMETS facility.

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The R512E coated Mo-47Re coupons were tested at 1150°C and 1230°C under a Mach 4 air environment. Figure 8 shows the weight loss at 1150°C and 1230°C as a function of exposure time in the HYMETS facility. The weight loss during the 1150°C test occurred sooner and was greater than during the 1230°C test.
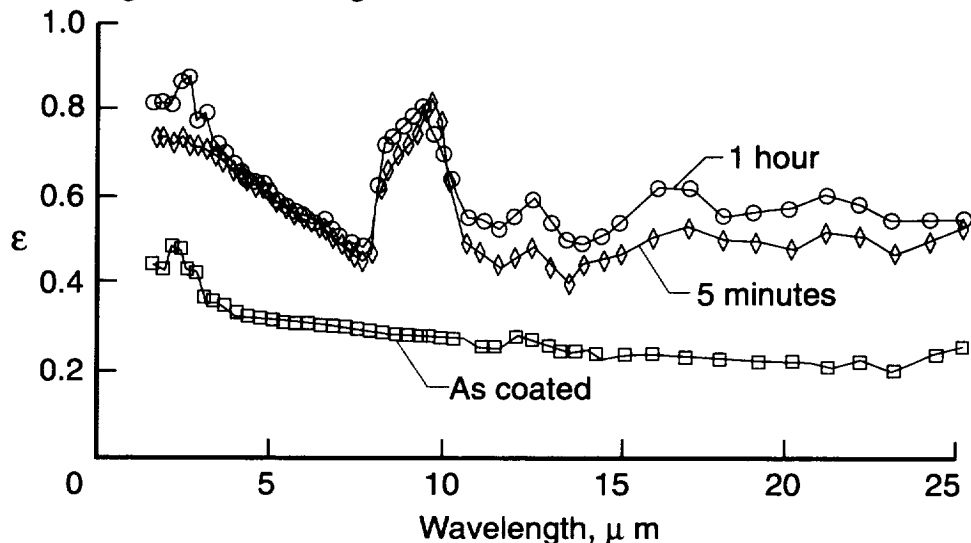
Figure 9: Spectral emittance for R512E coated Mo-47Re after exposure in the HYMETS facility at 1150°C.

The spectral emittance as a function of wavelength was obtained on the coupons tested in the HYMETS facility. On several occasions when the coupons were removed from the facility for weighing, the spectral emittance was measured. Figure 9 shows the spectral emittance for the 1150°C heated coupon in the as coated condition, after 5 min of exposure, and after 1 hour of exposure. The emittance increased after heating to 1150°C, but most of the increase in emittance occurred within the first 5 min of exposure.
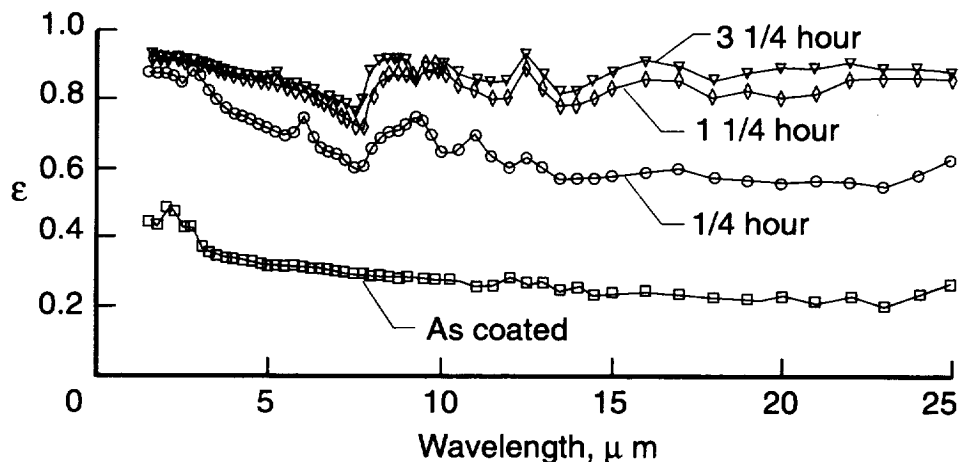
Figure 10: Spectral emittance for R512E coated Mo-47Re after exposure in the HYMETS facility at 1230°C.

The spectral emittance of the R512E coated Mo-47Re coupon heated at 1230°C in the HYMETS is shown in Figure 10. Emittance values are shown in the as coated condition, after 0.25 hours, 1.25 hours, and 3.25 hours of exposure. As with the 1150°C exposure, a significant increase in the emittance occurred after minimal exposure time. The emittance did continue to increase with increased exposure time, but at a much

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slower rate. The emittance values were also significantly higher after the 1230°C exposure than after th 1150°C exposure. Part of this difference may be due to the longer heating time at 1230°C than at 1150°C

Conclusions

Oxidation and emittance tests were preformed on arc cast and powder metallurgical Mo-Re using both a Fe-Cr-Si (R512E) coating and a $Mo(Si_2, Ge)$ coating. The R512E coating provided far superior oxidation protection on the Mo-Re than the $Mo(Si_2, Ge)$. Emittance measurements of the R512E coated Mo-47Re as a function of wavelength were performed and indicated a rapid increase in emittance after a short exposure (~15 min.) in the HYMETS facility at Mach 4.

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