Multi-Scale Computational Design and Synthesis of Protective Smart Coatings for Refractory Metal Alloys

John H. Perepezko (PI), Otto J. Lu-Steffes
Dept. Materials Science & Engineering, University of Wisconsin-Madison
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Outline

• Background

• Research Accomplishments
  – Mo-Si-B coating development for Nb alloys
  – Zr additions to Mo-Si-B coating

• Future Work

• Concluding Remarks
Background

- Ni-based superalloys provide the necessary structural strength while at the same time remaining oxidation resistant in combustion systems.
- Other high temperature materials must be explored because Ni-based superalloys are reaching their limit in operational temperature.
- Protective coatings necessary for increased oxidation performance.

Background

- Refractory metals are have higher melting points than current nickel-base superalloys.
- Refractory metals readily oxidize at high temperatures forming metal oxides that drastically reduce the integrity of the metal.
- Prospective alloys are Nb-based alloys consisting of Hf, Ti, Cr, Ge, Al, B, and Si additions.

<table>
<thead>
<tr>
<th>Main alloying addition</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>Aid in creep and oxidation resistance resistance (Nb₅Si₃, Nb₂Si)</td>
</tr>
<tr>
<td>Ti (~25 at.%)</td>
<td>Formation of Nb₂O₃-TiO₂ to reduce oxidation rate of alloy [2]</td>
</tr>
<tr>
<td>Cr (~8 at.%)</td>
<td>Formation of Cr₂Nb Laves phase, aiding in oxidation resistance (CrNbO₄ during oxidation) [3][4]</td>
</tr>
<tr>
<td>Ge/B</td>
<td>Reduce viscosity of silica glass</td>
</tr>
</tbody>
</table>

Oxidation of Nb alloys in air at 1100°C [5]

Background

• While alloy additions to Nb have shown increased oxidation resistance the long term goal of these alloys is still out of reach
  – Long term goal operational temperature of 1315°C with <25µm material loss in 100 hrs
• Nb produces 3 different oxides: Nb₂O₅, NbO₂, NbO [6]
• Protective coatings necessary for increased oxidation performance
  – Retain ductility in Nb alloys

Background: Current Protection Methods

- Current protection methods are based on silicide coatings that produce a protective silica layer
  - Due to selective oxidation of Si
- A study by Kurokawa et al. showed that disilicides would either form a protective SiO$_2$ scale or a non protective mixed oxide scale depending on the temperature during oxidation (Table 1) [8]
  - Refractory metal disilicides (MoSi$_2$, WSi$_2$) also form trioxide, which is either stable or volatile depending on temperature and partial pressure of O$_2$

<table>
<thead>
<tr>
<th>Oxide scale structure</th>
<th>Silicide</th>
<th>Oxid. resistance</th>
<th>Temp. region</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$ scale</td>
<td>FeSi$_2$</td>
<td>⊙</td>
<td>&lt;1273K</td>
</tr>
<tr>
<td></td>
<td>CoSi$_2$</td>
<td>⊙</td>
<td>&lt;1273K</td>
</tr>
<tr>
<td></td>
<td>MoSi$_2$</td>
<td>⊙</td>
<td>&gt;1073K</td>
</tr>
<tr>
<td></td>
<td>WSi$_2$</td>
<td>⊙</td>
<td>&gt;1573K</td>
</tr>
<tr>
<td></td>
<td>VSi$_2$</td>
<td>⊙</td>
<td>&gt;1173K</td>
</tr>
<tr>
<td></td>
<td>ReSi$_{1.75}$</td>
<td>△</td>
<td>&gt;1273K</td>
</tr>
<tr>
<td></td>
<td>ReSi$_{1.75}$</td>
<td>✗</td>
<td>&lt;1273K</td>
</tr>
<tr>
<td>Double layer scale</td>
<td>CrSi$_2$</td>
<td>⊙</td>
<td>&lt;1373K</td>
</tr>
<tr>
<td></td>
<td>x</td>
<td>&gt;1473K</td>
<td></td>
</tr>
<tr>
<td>Mixed oxide scale</td>
<td>NbSi$_2$</td>
<td>x</td>
<td>&lt;1773K</td>
</tr>
<tr>
<td></td>
<td>TaSi$_2$</td>
<td>x</td>
<td>&lt;1773K</td>
</tr>
<tr>
<td></td>
<td>MoSi$_2$</td>
<td>x</td>
<td>773–1073K</td>
</tr>
<tr>
<td></td>
<td>WSi$_2$</td>
<td>x</td>
<td>1073–1573K</td>
</tr>
<tr>
<td></td>
<td>VSi$_2$</td>
<td>⊙</td>
<td>&lt;1173K</td>
</tr>
<tr>
<td></td>
<td>TiSi$_2$</td>
<td>x</td>
<td>&gt;773K</td>
</tr>
<tr>
<td></td>
<td>Ti$_6$Si$_3$</td>
<td>x</td>
<td>&gt;773K</td>
</tr>
</tbody>
</table>

Mo-Si-B Coatings for Oxidation Protection

• The Mo-Si-B system has been proven to provide oxidation protection in a temperature range of 1200-2000°C
  – $T_1$ phase serving as a source of Si and B to enable a self-healing capability [10].
  – $T_2$ phase serving as a diffusion barrier to inhibit dissolution of the coating into the substrate
• Mo-Si-B coatings have been applied to other refractory metal substrates and cermets to provide enhanced oxidation protection [11,12]
  – Suggests coatings can be extended to Nb base systems

Synthesis of Mo-Si-B Coatings on RM Substrates

• 2 step processes:
  1. Mo deposition onto substrate for < 5 minutes at 300°C using Mo(CO)$_6$ decomposition process

\[ \text{Mo(CO)}_6 \rightarrow \text{Mo} + 6\text{CO(g)} \]

Plan view SEM image of Mo particles on Nb sample

Cross section SEM image of Mo particles on Nb sample
Synthesis of Mo-Si-B Coatings on RM Substrates

- 2 step processes:
  2. Co-deposition of Si+B into Mo deposit and substrate ~ 50 hours at 1000°C.
- CVD technique that uses halide vapor species to diffuse to the substrate and decompose, depositing the desired elements

\[
\begin{align*}
3\text{NaF} + 2\text{Si(s)} & \leftrightarrow 2\text{SiF(g)} + 3\text{Na(g)} + ½\text{F}_2(g) \\
3\text{NaF} + 2\text{B(s)} & \leftrightarrow 2\text{BF(g)} + 3\text{Na(g)} + ½\text{F}_2(g) \\
3\text{SiF}(g) & \leftrightarrow \text{SiF}_2(g) + \text{Si(s)} \downarrow \\
3\text{SiF}_2(g) & \leftrightarrow 2\text{SiF}_3(g) + \text{Si(s)} \downarrow \\
4\text{SiF}_3(g) & \leftrightarrow 3\text{SiF}_4(g) + \text{Si(s)} \downarrow \\
3\text{BF}(g) & \leftrightarrow \text{BF}_2(g) + \text{B(s)} \downarrow \\
3\text{BF}_2(g) & \leftrightarrow 2\text{BF}_3(g) + \text{B(s)} \downarrow 
\end{align*}
\]

- To optimize coating structure, vary the proportion of Si and B in the pack powder

Activity of Si and B fluorides (NaF activator, 35:1 Si: B ratio)
Smart Coating: Benefits of TBC

- Thermal barrier coatings (TBC) are used to limit thermal exposure
  - reduce the substrate temperature of a superalloy by as much as 300°C [13]
- CTE mismatch between the TBC and underlying layers can result in failure of the coating [14]

Extended coating system

- Mo-Si-B coatings provide robust oxidation resistance
- Thermal barrier coatings (TBC) are used to limit thermal exposure
- Research is focused on trying to create a coating that contains advantageous properties of both Mo-Si-B and TBC coatings
  - Divide research into two categories
    - Mo-Si-B coating for Nb alloys
    - Zr additions to Mo-Si-B on Mo
Performance of Mo-Si-B Coatings

• Oxidation tests show that for a given Si:B ratio, Mo-Si-B coatings offer better protection compared to Si-B coatings
• However, the cross section of the coating reveals large amounts of Nb borides that can interfere with the formation of a protective oxidation coating
Boride Formation in Coatings

- Study by Cockeram and Rapp produced a boron modified disilicide coating on Ti alloys and also noticed significant boride formation [15]—proposed following reaction for the formation of TiB₂:
  \[ 2B + TiSi₂ \rightarrow TiB₂ + 2Si \] (1) 1000°C, \( \Delta G = -95.64 \text{ kJ/mol} \)

- \( \Delta G \) values calculated using HSC Chemistry for NbSi₂ and MoSi₂:
  \[ 2B + NbSi₂ \rightarrow NbB₂ + 2Si \] (2) 1000°C, \( \Delta G = -44.33 \text{ kJ/mol} \)
  \[ 2B + MoSi₂ \rightarrow MoB₂ + 2Si \] (3) 1000°C, \( \Delta G = +37.50 \text{ kJ/mol} \)

- Explains why borides are less prominent in Mo-Si-B coatings
- Boron additions in Si-B coatings all resulted in substantial boride formation

Activities of Halides

- Optimize coating structure by varying both Si:B$_{\text{source}}$ ratio as well as B source

Plots have a Si: B$_{\text{source}}$ ratio of 10:1
Optimize Mo-Si-B coating for Nb

- Substitution of TiB$_2$ in place of B in pack cementation treatment lowers the partial pressures of the boron fluorides [16]
  - TiB$_2$ substitution in pack completely suppresses boride formation in the coating structure
- CrB$_2$ source shows slightly more boride formation

\[
\begin{array}{|c|c|c|}
\hline
 & \Delta G^\circ (\text{J/mol}) & \text{Boron activity} \\
\hline
\text{SiB}_3 & -4,990 & 0.656 \\
\text{CrB}_2 & -41,100 & 3.10 \times 10^{-2} \\
\text{TaB}_2 & -98,100 & 2.51 \times 10^{-4} \\
\text{TiB}_2 & -149,700 & 3.20 \times 10^{-6} \\
\hline
\end{array}
\]

MoB substitution

- Si:MoB ratio of 10:1 used in pack cementation process at 1000°C for 25 hours
- Heat treatment at 1200°C at 25 hours in Ar flow
- TGA results show minimal mass change (0.96 mg/cm²)
- SEM of coating shows continuous glass formed on top of the sample, but boride phase is still present at the MoSi₂/NbSi₂ interface
- Suggests conditioning samples in Ar atmosphere prior to oxidation testing a preferred option for Nb samples
Mo-Si-B coating for Nb alloy

• Substrate: Nb-24Ti-15Si-4Cr-2Al-2Hf (at.%)  
  – Two phases: Nb$_{ss}$ (light regions) Nb$_5$Si$_3$ (dark regions)

• The samples were coated with Mo-Si-B in a two-step process. (1) molybdenum was deposited through the decomposition of Mo(CO)$_6$. (2) The silicon and boron/boride were deposited through a pack cementation process.

• B, CrB$_2$, TiB$_2$, MoB

• Pack condition: 1000 °C /25h
Coating structure (as-pack) for Nb alloy

Si:B 35:1

Si : MoB 10:1

Si : CrB₂ 20:1

Si : TiB₂ 10:1

<table>
<thead>
<tr>
<th>B source</th>
<th>NbSi₂</th>
<th>Boron Zone</th>
<th>MoSi₂ + glass</th>
<th>Total (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si:B</td>
<td>~30</td>
<td>~15</td>
<td>~30</td>
<td>~75</td>
</tr>
<tr>
<td>MoB</td>
<td>~40</td>
<td>~10</td>
<td>~30</td>
<td>~80</td>
</tr>
<tr>
<td>CrB₂</td>
<td>~13</td>
<td>~7</td>
<td>~30</td>
<td>~50</td>
</tr>
<tr>
<td>TiB₂</td>
<td>~20</td>
<td>0</td>
<td>~30</td>
<td>~50</td>
</tr>
</tbody>
</table>

- Boride layer reduced with TiB₂ and CrB₂ substitutions
Coating structure (as-pack) for Nb alloy

- XRD of the sample is composed of MoSi$_2$
- EPMA shows that the boron in the “boride region” is the (Nb,Ti)$_5$SiB$_2$ phase
  - T$_2$ phase of Nb-Si-B system

**EPMA**

<table>
<thead>
<tr>
<th>Phase</th>
<th>B (at.%)</th>
<th>Si (at.%)</th>
<th>Mo (at.%)</th>
<th>Nb (at.%)</th>
<th>Ti (at.%)</th>
<th>Na (at.%)</th>
<th>Al (at.%)</th>
<th>O (at.%)</th>
<th>Hf (at.%)</th>
<th>Cr (at.%)</th>
<th>Elemental total</th>
</tr>
</thead>
<tbody>
<tr>
<td>NbSi$_2$</td>
<td>0</td>
<td>56.94</td>
<td>0.00</td>
<td>25.12</td>
<td>12.56</td>
<td>0.05</td>
<td>0.63</td>
<td>1.41</td>
<td>0.56</td>
<td>2.36</td>
<td>99.63</td>
</tr>
<tr>
<td>(Nb,Ti)$_5$SiB$_2$</td>
<td>31.88</td>
<td>14.10</td>
<td>0.34</td>
<td>29.90</td>
<td>14.99</td>
<td>0.04</td>
<td>1.22</td>
<td>4.35</td>
<td>0.62</td>
<td>2.54</td>
<td>99.11</td>
</tr>
<tr>
<td>MoSi$_2$</td>
<td>0</td>
<td>62.84</td>
<td>29.28</td>
<td>0.01</td>
<td>0.02</td>
<td>0.89</td>
<td>0.63</td>
<td>6.97</td>
<td>0.00</td>
<td>0.10</td>
<td>98.41</td>
</tr>
</tbody>
</table>
**Oxidation: 1300°C 2 hours in air**

<table>
<thead>
<tr>
<th>B source: Si ratio</th>
<th>Weight gain(mg/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MoB:Si (1:10)</td>
<td>1.46</td>
</tr>
<tr>
<td>CrB₂:Si (1:20)</td>
<td>0.81, 0.82</td>
</tr>
<tr>
<td>TiB₂:Si (1:10)</td>
<td>1.03, 2.37</td>
</tr>
<tr>
<td>B:Si (1:20)</td>
<td>1.52</td>
</tr>
</tbody>
</table>

- CrB₂ B source shows the lowest mass gain
- Surface of sample is composed of borosilica and MoSi₂
- NbSi₂ converted to Nb₅Si₃ during oxidation

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Oxidation in air 1300 °C /2h CrB₂ sample
Prior to TGA testing, sample was first annealed in Ar atmosphere at 1200°C for 25 hours, then oxidized in air at 1300°C for 2 hours.

- Annealing step allows for NbSi$_2$ to convert to Nb$_5$Si$_3$ for better coating compatibility.

TGA testing was conducted on coated Nb alloy (CrB$_2$ B source) at 1300°C for 24 hours.

- Mass change: -0.83 mg/cm$^2$
- Initial transient stage (volatilization of MoO$_3$) followed by steady state oxidation.
- TGA curve shows a coating that prevents catastrophic oxidation of the alloy.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Crystal Structure</th>
<th>CTE in a direction (C$^{-1}$)</th>
<th>CTE in c direction (if applicable) (C$^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MoSi$_2$</td>
<td>Tetragonal I4/mmm</td>
<td>1.04E-05</td>
<td></td>
</tr>
<tr>
<td>MoSi$_3$</td>
<td>Tetragonal I4/mcm</td>
<td>6.14x10$^{-6}$</td>
<td>1.10x10$^{-5}$</td>
</tr>
<tr>
<td>NbSi$_2$</td>
<td>Hexagonal P62 22</td>
<td>9.81E-06</td>
<td>1.13E10$^{-5}$</td>
</tr>
<tr>
<td>NbSi$_3$</td>
<td>Tetragonal I4/mcm</td>
<td>~9.0E-06</td>
<td></td>
</tr>
</tbody>
</table>
Zr additions to Mo-Si-B coating

• Zr additions were made first by arc melting Mo-Zr alloys (1, 2at. %Zr)
  — Apply Si-B pack cementation treatment
• Static oxidation tests and TGA testing of coated Mo-Zr alloys
  — Samples conditioned at 1500°C for 2 hours prior to testing
• Mo-1at.% Zr 1500°C 50 hours mass change: -0.047 mg/cm²
• Zr rich particles seen in cross section and plan view of silica

<table>
<thead>
<tr>
<th>Element</th>
<th>Atom %</th>
<th>Atom % Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>O K</td>
<td>45.94</td>
<td>+/- 0.43</td>
</tr>
<tr>
<td>Na K</td>
<td>0.62</td>
<td>+/- 0.05</td>
</tr>
<tr>
<td>Al K</td>
<td>0.56</td>
<td>+/- 0.04</td>
</tr>
<tr>
<td>Si K</td>
<td>33.34</td>
<td>+/- 0.13</td>
</tr>
<tr>
<td>Zr L</td>
<td>19.54</td>
<td>+/- 0.09</td>
</tr>
<tr>
<td>Total</td>
<td>100.00</td>
<td></td>
</tr>
</tbody>
</table>
TGA Results

- TGA Results of Si-B coated Mo-2atZr show reasonable oxidation
  - Zr additions do not inhibit the formation of an oxidation resistant coating
- Mass change less than those obtained from Mo-Si-B-Zr alloy [17]
  - Oxidation protection not obtained at temperatures above 1300°C

Calculated oxidation rates for MoSiB and MoSiB-1Zr alloys [13]

SEM: Mo-2Zr (1500°C, 24 hr)

- Mass change: 1.40 mg/cm²
- Zr-O phase seen within the substrate (~250 μm from the surface)
  - Zr additions increasing oxygen diffusion through coating
Zr additions to Mo-Si-B coating

- Zr additions should be focused within the coating as opposed to the substrate
  - Could produce unwanted phases compromising the properties of the substrate
- Pack cementation treatment to infuse Zr into the Mo-Si-B coating
  - Check vapor pressures to assess feasibility
- ZrCl$_x$ much more active compared to ZrF$_x$

**Graphs:**

- NaF activator
- NH$_4$Cl activator
Zr additions to Mo-Si-B coating

• Si-B pack cementation applied to polished Mo coins (2mm thickness)
  – 1000°C 50 hrs
• Samples were ultrasonic cleaned and packed in Zr pack
  – 5 wt.% Zr
  – 2.5 wt.% NH₄Cl
  – Bal. Al₂O₃
  – 1000°C 10 hr
• Samples oxidized at 1500°C
Zr additions to Mo-Si-B coating

- As- Packed
  - Coating composed of MoSi$_2$ and Mo boride (standard as-pack structure)
- EDS on the surface suggest formation of Zr silicides
Zr additions to Mo-Si-B coating: oxidation (1500°C 2hr)

- Mass change: 0.25 mg/cm²
- Cross section after oxidation shows Mo-Si-B coating has converted some of the MoSi₂ into T₁ and T₂
- Surface of sample appears to be completely covered by Zr-O particles
  - EDS detects regions containing both ZrO₂ as well as Zircon (ZrSiO₄)

<table>
<thead>
<tr>
<th>Element Line</th>
<th>Atom %</th>
</tr>
</thead>
<tbody>
<tr>
<td>O K</td>
<td>62.69</td>
</tr>
<tr>
<td>Al K</td>
<td>0.37</td>
</tr>
<tr>
<td>Si K</td>
<td>19.87</td>
</tr>
<tr>
<td>Si L</td>
<td>---</td>
</tr>
<tr>
<td>Zr L</td>
<td>17.07</td>
</tr>
<tr>
<td>Zr M</td>
<td>---</td>
</tr>
<tr>
<td>Total</td>
<td>100.00</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample</th>
<th>O-K</th>
<th>Na-K</th>
<th>Al-K</th>
<th>Si-K</th>
<th>Zr-L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base(209)_pt1</td>
<td>55.56</td>
<td>0.73</td>
<td>0.47</td>
<td>26.77</td>
<td>16.47</td>
</tr>
<tr>
<td>Base(209)_pt2</td>
<td>54.93</td>
<td></td>
<td>9.42</td>
<td></td>
<td>35.65</td>
</tr>
<tr>
<td>Base(209)_pt3</td>
<td>56.17</td>
<td></td>
<td>6.98</td>
<td></td>
<td>36.85</td>
</tr>
</tbody>
</table>
Zr additions to Mo-Si-B coating: oxidation (1500°C 10hr)

- 1500 C 10 hr, mass change: 1.30 mg/cm²
- XRD shows peaks corresponding to Zircon, MoSi₂ and T₁
- SEM shows a layer of Zircon formation on top of the borosilica
Future Work: Utilize plasma spray deposition

- Thermal spray process combined with Si-B pack cementation technique is an effective process to produce Mo-Si-B coatings on larger samples [18]
  - Plasma spray deposition of Mo allows for samples to be scaled up in size and complexity
- Formation of a larger coating allowing for more aggressive oxidation testing for Nb and Nb alloys

Future Work: Formation of Smart Coating

- Optimization of parameters for the CVD coating processes
- Integration of the smart coating into an expanded range of other TM-based alloys with emphasis on Nb-based alloys.
- Coatings with a compositional gradient decrease residual stresses [19]
  - Sharp changes in stresses may result in delamination of the coating
- Object Oriented FEA (OOF2) is public domain finite element analysis (FEA) software created by NIST to investigate the properties of microstructures
  - utilizes actual microstructure images as the basis for the finite element mesh
  - Obtain a coating structure that minimizes the residual stresses due to CTE mismatch of the coating and substrate

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Temperatures of the TBC, BC, and Nb-Silicide as Measured during the JETS Test; the BC Temperature Was Calculated Using Measurements of the Temperatures of the TBC and Nb-Silicide Substrate, Together with Thermal Conductivity Measurements [20]
OOF-2: Boundary Conditions

- **Bottom**
  - $Y=0$ (substrate)
  - $X$ free to move (infinite plane)
- **Left side**
  - $X=0$
  - $Y$ free to move (no constraints on top of coating)
- **Right side**
  - $X =$ allow for free expansion such that $\sum \sigma_x = 0$
  - Want non-free surfaces to expand in a planar fashion
    - Run simulation first under no constraints to provide estimate of expansion taking place
OOF-2: Materials and BC

- Isotropic values
- Assume at 1000°C the coating is in a relaxed state (temperature at which coating forms).
- Boundary Conditions
  - $T_{hot} = 300$
  - $T_{cold} = 0$
  - $Y_{bottom} = 0$
  - $X_{left} = 0$
  - $X_{right} = 2.23 \times 10^{-8} \text{ m}$

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (kg/m$^3$)</th>
<th>Young's Modulus (GPa)</th>
<th>Poisson Ratio</th>
<th>Thermal Cond (W/mK)</th>
<th>CTE (C$^{-3}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mo</td>
<td>10200</td>
<td>324.8</td>
<td>0.293</td>
<td>138</td>
<td>6.50x10$^{-6}$</td>
</tr>
<tr>
<td>MoSi2</td>
<td>6300</td>
<td>432</td>
<td>0.151</td>
<td>28.6</td>
<td>1.04x10$^{-5}$</td>
</tr>
<tr>
<td>Mo5Si3 (T1)</td>
<td>8190</td>
<td>363</td>
<td>0.268</td>
<td>19</td>
<td>8.35x10$^{-6}$</td>
</tr>
<tr>
<td>Mo5SiB2 (T2)</td>
<td>8800</td>
<td>383</td>
<td>0.27</td>
<td>28</td>
<td>8.5x10$^{-6}$</td>
</tr>
<tr>
<td>borosilicate glass</td>
<td>2400</td>
<td>64</td>
<td>0.19</td>
<td>1.1</td>
<td>4.00x10$^{-6}$</td>
</tr>
</tbody>
</table>

FEM mesh generated in OOF-2
OOF-2: Stresses

- Top portion of coating is in compression while bottom (substrate) is in tension
- Compressive yield stress for MoSi$_2$: 769 MPa
  - Suggests that coating will be intact under these conditions
Concluding Remarks

• Mo-Si-B coating applied to Nb alloys to provide oxidation resistance
  – Can be synthesized first by applying a Mo layer via decomposition of molybdenum hexacarbonyl followed by a Si-B co-pack cementation process
  – Boride formation during pack cementation process within Nb produces undesirable coating structure, but can be optimize using different boride source
  – Enhanced oxidation protection by reducing the formation of Nb oxides in favor for a protective borosilica coating
• Coated Mo-Zr alloys show promise that the transition metal oxide can develop within the Mo-Si-B coating to provide further environmental protection.
  – Zr can be added to Mo-Si-B coating via pack cementation technique
    • Upon oxidation, oxidation resistant silica and zircon/zirconia form
  – Coating provides the necessary components for the development of a smart coating for refractory metal alloys.
Thank You!
List of papers published, conference presentations, students supported under grant FE0007377

• Publications:

• Conferences
  – Research results were presented at the 2012 Materials Science and Technology conference in the symposium “Beyond Nickel Based Superalloys-II.” The title of the presentation was “Transition Metal Doped Mo-Si-B Coatings.”
  – Research results were presented at the 2012 First ACEEES International Forum. The title of the presentation was “Design and Synthesis of Zr Doped Mo-Si-B Coatings”
  – Research results were presented at the 2013 Materials Science and Technology conference in the symposium “High Temperature Corrosion and Oxidation of Materials.” The title of the presentation is “Mo-Si-B Coatings on Niobium Base Systems for Enhanced Oxidation Protection”
  – Research results were presented at the 2013 Second ACEEES International Forum. The title of the presentation was “Enhanced Oxidation Protection for Niobium Base Alloys Utilizing a Mo-Si-B Coating”
  – Research Accomplishments from this project were presented at the 2012 University Coal Research/Historically Black Colleges and Universities and Other Minority Institutions Contractors Review that took place on May 31, 2012.
  – Research Accomplishments from this project were presented at the 2013 University Coal Research/Historically Black Colleges and Universities and Other Minority Institutions Contractors Review that took place on June 11-13, 2013.

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  – John Perepezko; PI
  – Otto Lu-Steffes; graduate student
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