Accelerated Design of Alumina-Forming, High-temperature Austenitic Alloys*
*Subtask 2B1 under the Powertrain Materials Core Program (PMCP)

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Overview: New exploratory effort from *subtask 2B1 in Powertrain Materials Core Program (PMCP)

<table>
<thead>
<tr>
<th>Timeline</th>
<th>Barriers</th>
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<tbody>
<tr>
<td>• Effort start: Mar 2020</td>
<td>• Changing internal combustion engine regimes requiring higher-temperature capable materials</td>
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<td>• Effort end: Sept 2020 (may be extended depending on results)</td>
<td>• Development time/cost of new materials</td>
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<td>• Percent complete: 20% (1 month)</td>
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<th>Budget</th>
<th>Collaboration</th>
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<tr>
<td>• Total effort funding: $75k in FY20</td>
<td>• Thermo-Calc Software</td>
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<td>• Internal ORNL Collaboration Among Computational Science and Materials Science Organizations</td>
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* Exploratory effort to demonstrate a novel machine learning alloy design approach in support of Subtask 2B1 Development of Cast, Higher Temperature Austenitic Alloys.
Increased temperatures and pressures to enable cleaner, more efficient engines with **alumina-forming austenitic (AFA)** alloys

- **Current exhaust component alloys:** lose oxidation resistance and strength ≥ ~800°C
- **Ni-base alloys:** meet these targets but are too costly (≥ 3 - 10x Fe-base)

**2B1 Objective:** Develop low-cost “Fe-base alloys” for “>900-950°C”
- **Improved oxidation resistance** by forming protective Al₂O₃ scale formation (→ AFA) instead of Cr₂O₃
- **Increased strength & creep** by nano-precipitates

**Objective:** Demonstrate machine learning accelerated design of AFA-type alloys
- Leverage 10 years of creep data over multiple wrought AFA alloys previously developed at ORNL
- Couple high-throughput computational thermodynamics with machine learning in the AFA dataset to train for **prediction of creep resistance**
- Predict creep resistance of millions of systematically generated hypothetical AFA alloys

Relevance

LMP* = (T(°C)+273) x (20+log $t_{rupture}$[h])

*Larson-Miller Parameter: time-temperature correlative approach based on the Arrhenius rate equation for creep rupture life prediction at a given stress*
Rapidly exploring high-dimensional multi-component alloy space with high-throughput data analytics approaches

Composition ranges from ORNL AFA experts

Design of Experiments (DOE)

2,000,000 hypothetical AFA alloy compositions

High-throughput Computational Thermodynamics

Pre-trained Machine Learning (ML) Model

Processing Conditions + Degree of Supersaturation*

Virtually validated new “recipes”

<table>
<thead>
<tr>
<th>wt.%</th>
<th>Max</th>
<th>Min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>Bal</td>
<td>Bal</td>
</tr>
<tr>
<td>Cr</td>
<td>25</td>
<td>12</td>
</tr>
<tr>
<td>Mn</td>
<td>7</td>
<td>0</td>
</tr>
<tr>
<td>Ni</td>
<td>40</td>
<td>15</td>
</tr>
<tr>
<td>Cu</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>Al</td>
<td>6</td>
<td>2.5</td>
</tr>
<tr>
<td>Si</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Nb</td>
<td>3.5</td>
<td>0.3</td>
</tr>
</tbody>
</table>

15 elements, 5 variants ~ \(5^{15}=30,517,578,125\) (!)

Impossible to search all these

800-1,200 calculations/min. with ~1,000 cores

Highly relevant features from computational thermodynamics have been used to train ML models.

Machine Learning (ML) Models
- LR: linear regression
- BR: Bayesian ridge
- NN: k-nearest neighbor
- RF: random forest
- SVM: support vector machines regression

Correlation Analysis Methods
- |PCC|: The absolute value of Pearson's Correlation Coefficient
- MIC: Maximal Information Coefficient
Linear regression-based models (BR and LR) outperform other ML models in predicting LMPs of new* AFA alloys

- The accuracy of trained models for non-linear regression based MLs are better than BR and LR.
- It is possible that non-linear ML models have been over fitted.

LR and BR models to predict LMPs of 2M hypothetical AFA alloys

* Alloys that are not in the training dataset
ML trained with key microstructure features identified a range of hypothetical AFA alloys with better & worse LMPs

Small subset of predicted hypothetical alloys will be experimentally validated to evaluate effectiveness of alloy design via ML methods
Collaboration and Coordination with Other Institutions

• **Thermo-Calc Software**: high throughput computational thermodynamic calculations of millions of hypothetical AFA alloys

• **ORNL CADES (Compute And Data Environment for Science)**
  – AWS-like cloud computing

• **ORNL Computational Sciences Division**: for Data Analytics Expertise

• ORNL Materials Science for computational thermodynamics and alloy design, alloy manufacture, and creep evaluation expertise
Proposed Future Research

• Experimental validation of alloys with predicted good LMPs
  – Manufacture 0.5 kg lab heats of select predicted alloys and creep test under an accelerated test conditions of 750°C and 130 MPa.
  – Success is defined by extent to which the predicted alloys exceeds creep resistance of best previously developed AFA alloys to date

• Uncertainty quantification to assign error bars of ML predicted LMPs by considering different number of features for a given ML model

Any proposed future work is subject to change based on funding levels.
Summary

• New high-fidelity machine learning models have been trained with ORNL AFA creep data and key microstructure features.

• Creep properties (i.e., LMP) of 2 million hypothetical AFA alloys have been predicted.

• Small subset of identified AFA alloys with improved creep will be experimentally validated.
Technical Back-Up Slides
Alumina-Forming Austenitic (AFA) Alloy Family: a Lower Cost, Fe-Base Alloys with Improved Oxidation Resistance + Strength

- **Wide composition range (wt.%)**: Fe-(10-25)Cr-(2-5)Al-(12-35)Ni-(0.6-3)Nb-(0.05-0.5)C+(B, Hf, Mn, Mo, Si, Ta, Ti, V, W, Y, Zr, ...)

- **AFA Strengthening**: *nano-carbides ± intermetallics*
  while forming protective alumina

- **Good oxidation resistance**

  
  - 800 °C, 10,000 h in air

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**Design better AFA alloys by machine learning?**

**Commercialization of 1st Generation AFA in progress**

Coupling physics into the machine learning predictions

**Data Collection/Population**
- Composition
- Creep (LMP)

Input:
- ORNL Alumina-forming Austenitic (AFA) alloys ~ a decade work

Output:
- High-throughput data population
- Microstructure related features
  - Degree of supersaturation

**Correlation Analysis**
- Compositions + Processing conditions
- R = 0.93
- R = 0.90
- R = 0.87
- R = 0.89

**Machine Learning**
- Random Forest
  - R = 0.93
  - R = 0.87

- Linear Regression
  - R = 0.93
  - R = 0.91

## Augment experimental data with scientific features

<table>
<thead>
<tr>
<th>Elements</th>
<th>Fe, Cr, Mn, Ni, Cu, Al, Si, Nb, V, Ti, Mo, W, Y, Zr, Hf, C, B</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1 (solutionizing, °C)</td>
<td>1100, 1150, 1200, 1250</td>
</tr>
<tr>
<td>T2 (creep test, °C)</td>
<td>650, 700, 750, 800</td>
</tr>
<tr>
<td>Stress (MPa)</td>
<td>70, 100, 130, 170, 200, 250, 300</td>
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**Experimental details**

<table>
<thead>
<tr>
<th>Phases</th>
<th>FCC</th>
<th>Austenite (FCC matrix), NbC, L12</th>
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<tbody>
<tr>
<td></td>
<td>BCC (B2, A2)</td>
<td>NiAl, FeCr</td>
</tr>
<tr>
<td></td>
<td>C14 Laves</td>
<td>NiZr, FeNb</td>
</tr>
<tr>
<td>others</td>
<td>M23C6, M7C3, M2B, M3B2, MB2_C32, Ni3Ta_D0A, Sigma</td>
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</tbody>
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**Phases determined by thermodynamic calculations**

- w[Elements]: Elemental composition in weight percent
- T1, T2, dT: Solutionizing, creep test temperatures, dT=T1-T2
- NB_C: Ratio between Nb and C
- Creep stress: Stress
- LMP: Larson–Miller parameter

**Features in the dataset**

- {Temperature}_VPV_{Phases}: Volume fractions of phases at T1 and T2
- {Temperature}_X_{Phases}_{Elements}: Concentrations of elements in phases at T1 and T2
- d{Phases}: Degree of supersaturation (volume fraction difference between T1 and T2)