

ICME DESIGN OF HIGH PERFORMANCE TURBINE ALLOYS

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**ABSTRACT**

Integrated Computational Materials Engineering (ICME) tools are being developed at QuesTek Innovations for the design of high-performance alloys for turbine blades, specifically (1) a highly castable yet creep-resistant low-Re Ni-based superalloy and (2) high entropy alloys (HEAs) for extreme environments. In this work, we detail progress in the design of these two classes of alloys for turbine blade applications.

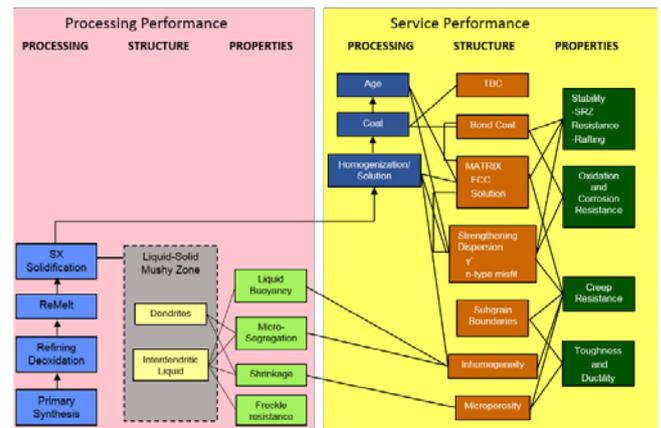
**INTRODUCTION**

Improvement to the efficiency of advanced power systems is a critical technological concern. Higher efficiencies can reduce harmful byproduct such as CO<sub>2</sub> and NO<sub>x</sub> emissions while utilizing coal resources more effectively. In order to improve the thermal efficiency of modern power plant technologies, the materials of the turbine blades and vanes must possess superior creep rupture resistance to endure the higher inlet temperatures associated with higher efficiencies.

Integrated Computational Materials Engineering (ICME) tools are being developed at QuesTek Innovations for the design of high-performance alloys for turbine blades, specifically (1) a highly castable yet creep-resistant low-Re Ni-based superalloy and (2) high entropy alloys (HEAs) for extreme environments. The integration of various calculations such as phase-equilibria, solidification pathways, diffusion fields, and precipitation behavior form an important foundation for designing high-performance alloys.

A key component of QuesTek’s ICME Materials by Design® methodology is the systems engineering approach [Olson97]. The interconnectivity of the processing-structure-properties-performance is shown schematically by the systems design chart in *Figure 1*. The properties are a function

of the microstructure, which can be achieved by the appropriate processing steps.



**Figure 1: Systems design chart developed by QuesTek for SX casting.**

Driven by the need for predictive computational tools for microstructural simulation of superalloys, a natural choice is CALPHAD-based (CALculation of PHAse Diagrams) simulation. Commercially available software tools and databases adopting the CALPHAD approach have been extensively used for the projects [Olson14, Jou04, Apelian04].

**METHODOLOGY**

**The CALPHAD Approach**

The CALPHAD (CALculation of PHAse Diagrams) approach utilizes phenomenological models to describe the thermodynamic properties of complex, multicomponent systems [Lukas07]. Thermodynamic and phase equilibria data are used to determine fitting parameters for the Gibbs free

energies of individual phases in the system. For a solid solution phase  $\alpha$ , the Gibbs free energy of that phase ( $G^\alpha$ ) is defined by

$$G^\alpha = \sum_i^c x_i G_i^\alpha - T S_{mix}^{ideal} + {}^{xs}G_m. \quad (1)$$

where  $x_i$  is the composition of component  $i$ ,  $G_i^\alpha$  is the Gibbs energy of component  $i$  in the  $\alpha$  structure,  $T$  is temperature,  $S_{mix}^{ideal}$  is the ideal configurational entropy (i.e.  $R \sum x_i \ln x_i$ ), and  ${}^{xs}G_m$  is the excess Gibbs energy. Databases containing these free energy descriptions can be used to predict phase equilibria and other properties for large, multicomponent alloys at arbitrary compositions by combining the functions of simpler subsystems.

For complex solid solutions (such as HEAs), the CALPHAD approach offers an accurate prediction of thermodynamic properties, such as stability. It is important to note that, although HEAs are typically considered to be stabilized by the ideal configuration entropy, the excess Gibbs energy must also be taken into account when modeling the stability of an HEA. The excess Gibbs energy ( ${}^{xs}G_m$ ) encompasses the non-ideal mixing enthalpy and entropy and is typically defined in CALPHAD databases by a Redlich-Kister polynomial:

$${}^{xs}G_m = \sum_{i=1}^{c-1} \sum_{j>i}^c x_i x_j \sum_{v=0}^n {}^v L_{ij} (x_i - x_j)^v + \sum_{i=1}^{c-2} \sum_{j>i}^{c-1} \sum_{k>j}^c x_i x_j x_k {}^0 L_{ijk}. \quad (2)$$

where  $x_i$  is the composition of component  $i$ ,  ${}^v L_{ij}$  are binary interaction parameters describing the non-ideality of the binary solid solution between components  $i$  and  $j$ ,  $v$  is the order of the interaction (e.g. 0 is regular, 1 is sub-regular, etc.), and  ${}^0 L_{ijk}$  is the regular ternary solution interaction parameter for elements  $i$ ,  $j$ , and  $k$ . The interaction parameters are fitted to experimental or theoretically-predicted thermodynamic data. For an arbitrarily large number of components, the solid solution thermodynamics are described by summing over all the subsystem interactions.

## RESULTS AND DISCUSSION

### Single Crystal Ni Superalloy for Turbine Blades

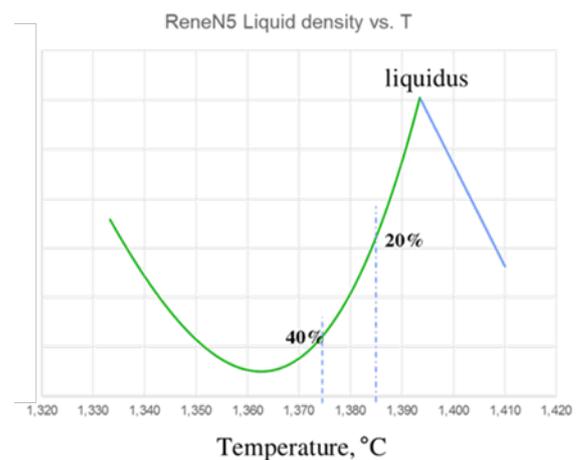
Ni-based single crystal (SX) superalloy blades, seen widely in aerospace engines, possess high creep strength in comparison with conventionally cast and directionally solidified (DS) blades. However, industrial gas turbines (IGTs) require much larger sized castings than aerospace engines, and the use of SX superalloys in IGTs is limited because of the low casting yield due to defects associated with large size and slow solidification, such as freckling, high/low angle boundary (HAB/LAB) formation, grain nucleation, and shrinkage/porosity. The first generation Re-free alloy, such as PWA1483, while castable as IGT blade toolings, lacks the creep-rupture and temperature capability properties of more recent SX alloys. On the other hand, 2<sup>nd</sup> gen Re-containing alloys, e.g. Rene N5, provide superior creep rupture properties but are difficult to cast as large IGT blades. Furthermore, the 2<sup>nd</sup> generation SX alloys and some more recent developed SX alloys usually possess more than 3wt% Re, which is one of the strategic elements whose price has been increased

significantly with the increasing use worldwide. Much effort has been made in both the academia and industry trying to reduce the use of Re while maintaining comparable thermomechanical properties of the high Re alloys. Some of the developments include the CMSX7/8 from Cannon-Muskegon [Wahl12] and ReneN515 from GE [Fink10]. Although these new developments do reduce the Re content, they are designed primarily for the field of aerospace and do not have the grain defect resistance for large-scale IGT casting as a criterion.

QuesTek Innovations LLC has developed an ICME framework and designed a highly-castable SX Ni-based superalloy with 1wt% Re, QTSX, that can be cast effectively as large, defect-free IGT components and provides creep performance comparable to state-of-the-art aeroturbine blades materials, thereby allowing higher gas temperatures and increased thermal efficiency. Single crystal casting trials were performed on physically large geometries with cross-section transitions that were representative of large IGT blades. Under nominally identical casting conditions, the QuesTek alloy “QTSX” was found to exhibit no evidence of casting defects (100% yield rate) and assessment of the microstructural stability, oxidation resistance, and creep performance of the newly developed QTSX alloy are exceptionally promising.

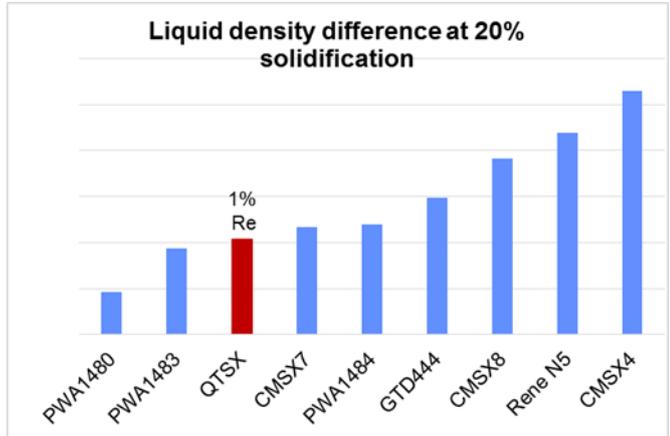
### Freckle Formation Modelling

One of the primary casting constraints for current SX alloys for IGT is freckle formation, which is usually initiated by convective instabilities developed in the mushy zone when the segregated interdendritic liquid is less dense than the overlying bulk liquid of original composition [Shah00]. The same thermosolutal convection processes that affect freckling formation also significantly influence the formation of misoriented grains or HABs [Pollock92]. Consequently, the same design parameters that reduce freckling tendency will also reduce the potency of defects such as misoriented grains.



**Figure 2. Predicted liquid density evolution with temperature during the solidification process. The blue line represents the liquid above liquidus and the green curve the remaining liquid during the solidification. The dashed lines indicate 20% and 40% solidification temperatures, respectively.**

A liquid buoyancy model based on CALPHAD was developed to identify components that reduce the density difference between the bulk liquid and the interdendritic liquid during, i.e. the primary driver for the freckling tendency. It is suggested that the alloy can be successfully cast under these processing conditions without freckle defects if this density difference can be consistently controlled under a critical Rayleigh number. Thus, it is important for the computational model to accurately predict this density difference during the solidification, and the critical Rayleigh value as the process parameters are changed. Figure 2 shows the calculated liquid density variation with temperature during the solidification process of ReneN5 alloy using a collection of ICME tools and databases. Above the liquidus, the liquid density increases with decreasing temperature as shown by the blue line. Below the liquidus, the compositional partitioning starts as part of the liquid solidifies so the density decreases with decreasing temperature. The dashed lines indicate 20% and 40% solidification temperatures, respectively. It is found that the critical liquid density difference that predicts freckling behavior is the one between liquidus and 20% solidification, i.e.  $\Delta\rho_{0.2}$ . The modeling results are shown in Figure 3 which listed the predicted liquid density difference at 20% solidification for a range of alloys of interest. The lower the normalized  $\Delta\rho_{0.2}$  value, the better castability for the alloy to be successfully cast into large IGT components. It is evident that QTSX (1% Re) has comparable castability to PWA1483 (non-Re) and CMSX7 (non-Re) alloy.

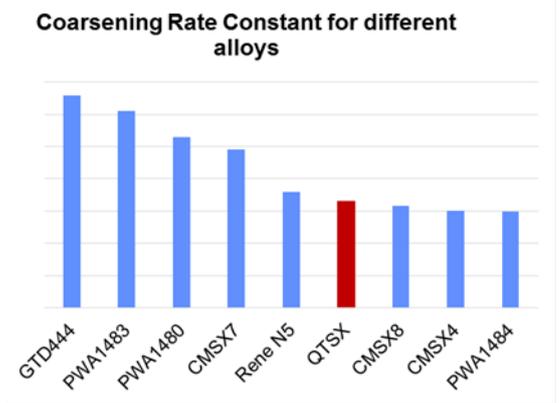


**Figure 3. Predicted liquid density difference at 20% solidification (the lower the better castability). QTSX (1% Re) has comparable value to PWA1483 (non-Re) and CMSX7 (non-Re) alloy.**

Creep Property Modelling

A complete mechanistic model for creep would require information one does not have at the alloy design stage, such as dislocation density, particle morphology and distribution, dislocation escape frequency, glide velocity etc. The creep properties of the alloy are strongly related to the coarsening resistance of the  $\gamma'$  precipitates and the vacancy diffusivity of the alloy. An approach for modeling creep strain as a function of time [Dyson09] indicates the primary relationship between composition and the strain is through the effective diffusivity

that is dependent upon mobility and vacancy binding energy. A multicomponent precipitate coarsening model was developed based on [Lee87], [Morral94], and [Kuehmann96] and built into QuesTek’s proprietary ICME design platform. The interfacial energy normalized coarsening rate constants at various high temperatures were calculated and compared between different alloys since they have similar  $\gamma$ - $\gamma'$  interfaces and the results can be used as an indicator for the creep performance of the alloys.



**Figure 4. Predicted coarsening rate of  $\gamma'$  precipitates at 1000°C (the lower the better creep performance). QTSX (1% Re) has comparable coarsening rate to CMSX-8 (1.5% Re) and ReneN5 (3% Re) alloy.**

The normalized coarsening rate constants at 1000°C for those alloys of interest are listed in Figure 4. It is evident that this value decreases from the 1st generation alloys to the 2nd generation. The low coarsening rate constant of the 2nd generation alloys explains their better strength and creep properties than the 1st generation alloys. On this design criterion, the coarsening rate constant of QTSX is in the range of the 2nd generation SX alloys, and even lower than that of ReneN5. It also has a comparable value with the new CMSX8 alloy. Considering QTSX uses less Re than CMSX8 to achieve this value, the advantage of QTSX is evident.

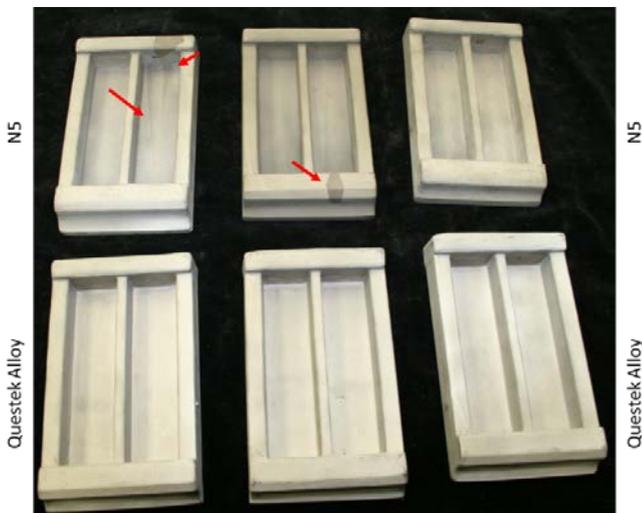
Other important design factors, such as phase equilibria, solidification temperature range, incipient melting, tcp phase formation, solution treatment window, precipitation events,  $\gamma'$  phase fraction at different temperature, lattice misfit between the  $\gamma$  matrix and  $\gamma'$  precipitates, and oxidation resistance, also contributes to the foundation for designing more castable, high-temperature-capability SX Ni-based superalloys for IGT components. Using the ICME models and both commercially available and internal databases, those design criteria were also calculated and used in the design of QTSX. For example, The  $\gamma'$  phase fraction at 1000 °C is designed to maintain optimal fraction about 60%  $\gamma'$  at service temperature to provide sufficient precipitate strengthening as it is found that there is an optimum phase fraction for creep properties and this optimum fraction depends on the temperature [Murakamo04]. Calculated results also show that for QTSX, a negative misfit about -0.3% is kept between the  $\gamma$  matrix and  $\gamma'$  precipitates at 1000°C. According quantitative regression

analysis of creep strengthening factors [Yokokawa12], lattice misfit is negatively correlated with creep-rupture life and is one of the most potent factors besides Re content. Therefore, it is predicted that QTSX will demonstrate similar, if not better, rafting resistance and creep-rupture life when compared with the 2<sup>nd</sup> generation SX Ni Superalloys.

Casting Trial Production

Through a partnership with a third party casting house, QTSX alloy has been cast into different test pieces with various extreme geometries as well as actual IGT blade castings to test the castability. The test cast pieces utilize thick corners and ridges to simulate the extreme casting parameters experienced in very large IGT blades that it is known for freckle-prone for existing alloys. ReneN5 was used as a baseline comparison alloy that was cast side by side with QTSX using the same molding and parameters.

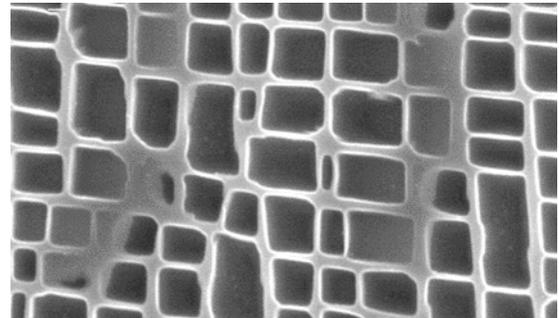
The casting results are best demonstrated by the pictures of the actual casting slabs, as shown in Figure 5 providing images of the front and backside of the as cast slabs made of both N5 and QTSX. It is very clear that the QuesTek alloy produces none freckles nor other grain defects while the slabs cast with N5 suffering from significant freckle formation – three out of three failed. The 100% yield rate of QTSX alloy is in sharp contrast to the 0% passing rate of Rene N5, for severe casting conditions of slow solidification rate and lower thermal gradient for large IGT component castings.



**Figure 5. As cast slabs made of QTSX and Rene N5. The red arrows indicate the casting defects, e.g. freckles**

A multistep homogenization treatment was identified that effectively avoids incipient melting, with the final step of the treatment occurring above the predicted  $\gamma'$  solvus. The as-homogenized microstructure shows some trace evidence of the original interdendritic phases and primary dendrite structure, which is typical of such highly-alloyed single crystal nickel alloys. EDX analysis indicates that these interdendritic features are primarily carbides and confirms that top phases have been successfully prohibited. Comparison of the alloy

design to baseline Rene N5 even suggests that a higher degree of homogeneity was achieved through this multistep homogenization schedule. Following homogenization, the alloys are further heat treated using double-step aging treatment to produce a fine, stable distribution of  $\gamma'$  precipitates. The resulting  $\gamma/\gamma'$  microstructure of fully heat-treated QTSX alloy is shown in Figure 6, which validates the aforementioned single crystal design using CALPHAD modeling.

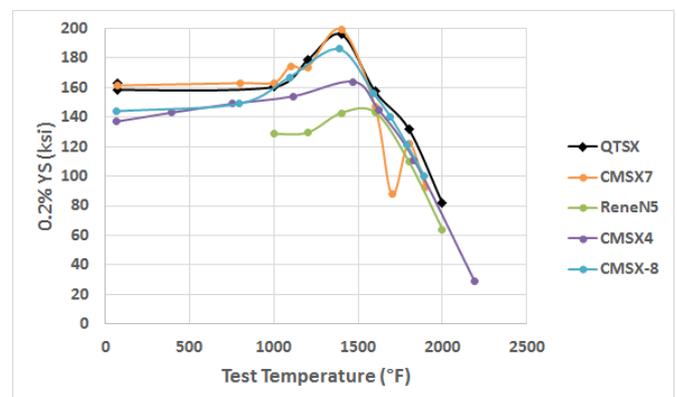


**Figure 6. Microstructure of fully heat-treated QTSX after double-step aging with  $\gamma/\gamma'$  structure revealed ( $\gamma'$  revealed as darkened phase).**

Properties and Performance

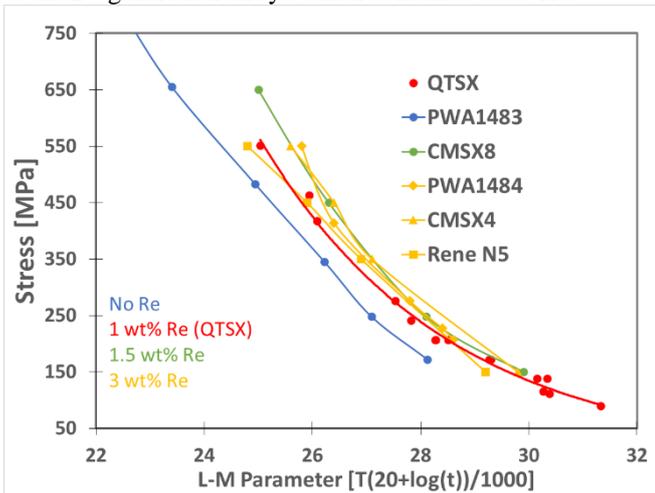
Mechanical properties of the alloy must be examined in a variety of conditions, but most importantly in high temperature and stressed environments to demonstrate the alloy’s application potential. Along with the blade castings, a couple dozen of test bars is also produced and heat-treated for various testing.

High-temperature tensile tests are performed to evaluate the yield strength of the alloy and can be compared to other commercial single crystal alloys. The 0.2% yield strength results of tensile tests across a wide range of temperatures from room temperature to as high as over 2000°F of the QTSX alloy are plotted against the published values of other SX alloys in Figure 7. It is clearly shown that the yield strength of QTSX stays near the top of the all the data points across the temperature range, indicating excellent tensile properties in various application scenarios.



**Figure 7. Comparison of yield strength tensile test results of QTSX with other SX alloys over temperature range from room temperature to over 2000°F.**

Stress rupture testing also serves as a critical test to compare the performance of the QTSX alloy with other commercially available single crystal alloys. The LMP stress rupture was determined experimentally for the QTSX alloy, and its result in comparison to different classes of Ni superalloys is shown in Figure 8. High Re-containing alloys, shown in yellow, usually have higher LMP, whereas the Re-free alloy PWA1483, shown in blue, tend to have lower LMP. It can be seen that the QTSX alloy, containing only 1 wt.% Re, has exceptionally good creep properties that is equivalent to the 2<sup>nd</sup> generation alloys which contain 3 wt.% Re.



**Figure 8. Comparison of Larson-Miller plot of stress rupture for QTSX and various benchmark alloys.**

The superiority of QTSX is more evident when comparing the Larson-Miller parameter at a low stress level of 150MPa, which is the usual performance-limiting case for the blade. Compared to the Larson-Miller parameters for the 2<sup>nd</sup> generation alloys, QTSX really exceeds all the other competition in the creep performance criterion for the higher temperature lower stress region.

Further testing is ongoing or arranged for the near future. The initial oxidation testing and long-term creep tests have also shown promising results and more validating is on the way waiting for those long-term experiments to complete.

### High Entropy Alloy CALPHAD Database Development

To some degree, engine operating temperature is limited by blade materials properties, particularly high temperature thermodynamic stability, strength, and creep resistance. While Ni-based superalloy technology development (e.g. single crystal growth) has steadily increased the operational limits of turbine blades, diminishing returns in Ni-alloy improvements are beginning to suggest an inherent limitation in these systems. Therefore, a radically different materials solution is necessary to produce a large increase in IGT operating temperature.

High entropy alloys (HEA) are a recently discovered class of materials that consist of a single-phase disordered solid solution of five or more components [Gao16]. HEAs are stabilized at high temperature by their configurational entropy and have exhibited high strength and low plasticity at elevated

temperatures. For these reasons, HEAs, particularly those containing refractory elements, have the potential to surpass Ni-based superalloy performance in turbine blade applications. Recent studies have reported several advancements towards HEA-based superalloys, including a  $\gamma/\gamma'$  system found in  $Al_{0.3}CoCrCu_{0.5}FeNi$  with an FCC Cu-rich HEA and  $(Ni,Cu)_3Al$   $L1_2$  [Tsai13], and the growth of a single crystal HEA [Ma13]. Although the possibility of HEAs serving as a superalloy component has been demonstrated, an exhaustive design search for HEAs using these stability criteria is necessary to predict suitable compositions worthy of future study. Since the composition space of HEAs is large, the complex phase equilibria surrounding HEAs (which determines the high-temperature stability of the alloy) is generally unexplored. Therefore, the use of modeling is required to design and optimize potential IGT HEA systems.

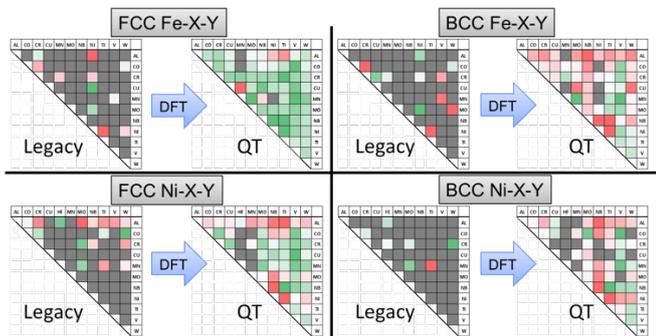
As HEAs are a significant departure from previously known materials, the underlying tools in the ICME framework need to be updated to describe these novel compositions and structures. Specifically, the processing-structure and structure-property models that QuesTek and the ICME community have developed for other materials systems (e.g. steels, superalloys, refractories, etc.) must be reevaluated to gauge their applicability to HEAs. Such activities embody the current efforts in HEA design for high-temperature turbine applications at QuesTek, in particular the thermodynamic foundation of ICME tools, CALPHAD databases.

The primary challenge in utilizing established CALPHAD techniques for ICME development of HEAs is the equiatomic nature of the HEA. CALPHAD databases are typically constructed with a focus on specific base elements (e.g. Fe for steels and Ni for superalloys), which places an emphasis on accuracy at corners of composition space. As such, specific parameters governing interactions between three or more components in FCC and BCC solid solutions are typically ignored. This is due to not only the application of the database to these narrow regions of composition space but also a lack of experimental thermochemical data for the other regions.

To resolve these issues, ICME efforts have focused on the construction and validation of a large CALPHAD thermodynamic database specifically designed for HEA compositions (QT-HEA), as current CALPHAD databases are not sufficiently accurate at equiatomic compositions. Such CALPHAD databases are an essential component of any materials design effort and QT-HEA offers quantitative predictions of HEA phase stability. The QT-HEA CALPHAD database is based on experimental data as well as exhaustive high-throughput density functional theory (DFT) calculations of the mixing enthalpies for all combinatorially possible FCC and BCC ternary solid solutions. These DFT predictions are from first-principles, employing quantum mechanical physics to simulate energetics of crystals at the electronic level without any experimental data needed, and they were calculated on high-performance computing resources at the University of Illinois at Urbana-Champaign National Center for Supercomputing Applications. Novel HEA compositions

have been identified using QT-HEA and experimentally verified by lab-scale alloy synthesis and characterization.

Due to a lack of experimental data, ternary interactions ( ${}^0L_{ijk}$  in Equation 2) in CALPHAD databases are typically ignored. The large sparsity of ternary interaction parameters is illustrated in Figure 9 for the ThermoTech Ni 7 (TTNI7) database. It is assumed that this is an adequate approximation for two reasons. (1) Binary interactions have the largest influence over the total free energy due to the compositional prefactor (i.e. ternary interactions have smaller effects since they are preceded by three mole fractions). (2) Typical alloys are largely composed of a single element (e.g. Fe in steels and Ni in superalloys), so the compositional prefactors are particularly small. However, these assumptions fail for equiatomic HEAs, where the effects of mixing are maximized and ternary interactions can have a large influence on solid solution stability.



**Figure 9: Values of Fe- and Ni-containing ternary interaction parameters for the FCC and BCC phases in the ThermoTech Ni 7 database before and after fits to DFT ternary mixing enthalpies. Green and red boxes indicate favourable and unfavourable interactions, respectively. Grey boxes indicate no interaction is modelled.**

The  ${}^0L_{ijk}$  ternary interaction parameters are the largest source of uncertainty in the CALPHAD database for the purposes of HEA stability prediction. Therefore, the high-throughput DFT  $\Delta H_{ABC}^{mix}$  ternary mixing enthalpies were used to fit their corresponding  ${}^0L_{ijk}$  ternary interaction parameters for the FCC and BCC phases. As binary systems are typically well described in commercial CALPHAD databases, the  ${}^0L_{ij}$  binary interaction parameters were left unchanged. The ThermoTech Ni 7 database was used as the starting point for the modified ternary interaction parameters since this database contains all twelve of the considered elements. All ternary interaction parameters were fit to reproduce the predicted ternary mixing enthalpies. The result of the fits for Fe- and Ni-containing ternary systems is shown in Figure 9.

All known HEA-forming compositions consisting of the current 12-component system were compiled from the literature, totaling 67 compositions. For each of these compositions, the phase equilibrium at 1000°C was calculated using the legacy Thermo-Calc FE 6 (TCFE6) and ThermoTech Ni 7 (TTNI7) databases and the new QT-HEA database. If a single phase was predicted to be stable (with at least 0.9 phase fraction), then the database was considered to be in agreement

with experiment. The accuracy of these databases to predict HEA stability is summarized in Table 1. The QT-HEA database is superior in predicting single phase equilibria for these observed HEA-forming compositions, demonstrating the improvements by integrating high-throughput DFT.

**Table 1: Agreement of HEA stability between CALPHAD predictions and experiment for the legacy CALPHAD databases (TCFE6 and TTNI7) and the new QT-HEA database.**

Database	Agreement with Exp.
TCFE6	24%
TTNI7	24%
QT-HEA	55%

Having demonstrated the improved accuracy of the QT-HEA CALPHAD database to predict HEA formation, the database was used to predict the phase equilibria of all 5-component equiatomic compositions in the 12-component Al-Co-Cr-Cu-Fe-Mn-Mo-Nb-Ni-Ti-V-W system, 792 compositions. Assuming, again, a minimum 0.9 phase fraction for the majority phase constitutes a HEA, 104 HEA-forming compositions are predicted. Several of these compositions have been used for experimental verification.

Ongoing work includes the use of the QT-HEA database to develop the necessary HEA structure-property models to design HEAs for IGT application, with a focus on strength, creep, and oxidation. QuesTek is leveraging prior internal work on structure-property modeling of Ni-, Fe-, Co-, and refractory-based alloys, emphasizing high-temperature creep resistance, minimizing vacancy diffusivity, maximizing high temperature solution hardening, and maximizing high temperature oxidation resistance. These models are founded on CALPHAD calculations of phase-equilibria, diffusion, and precipitation form an important foundation for designing high-temperature-capability HEAs for IGT components. The design process ultimately involves balancing all these properties against the property requirements (as defined by OEM partners), targeting IGT blade and vane applications.

## CONCLUSIONS

Integrated Computational Materials Engineering (ICME) tools are being developed at QuesTek Innovations for the design of high-performance alloys for turbine blades, specifically (1) a highly castable yet creep-resistant low-Re Ni-based superalloy and (2) high entropy alloys (HEAs) for extreme environments. In this work, we detail progress in the design of these two classes of alloys for turbine blade applications.

## ACKNOWLEDGMENTS

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