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Genomic materials design: The ferrous frontier

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Abstract

Our unique depth of scientific knowledge has allowed ferrous metallurgy to lead the development of a successful computational materials design methodology grounded in a system of fundamental databases. Expansion of this approach to embrace acceleration of the full development and qualification cycle has resulted in the flight of the first fully computationally designed and qualified material. The example of ferrous alloys provides a template for a general methodology of computational design for all materials.

Keywords: Materials design; Ferrous metallurgy; Integrated computational materials engineering; Materials genome

1. Introduction

Several recent US national academy studies of materials and manufacturing [1–5] have emphasized the unique opportunities of computational materials engineering. While the breadth of the best known 2008 Integrated Computational Material Engineering (ICME) study [2] created an impression of a field in its infancy, the 2004 Accelerated Technology Transition (ATT) study [3], uniquely charged with identifying best industrial practices to accelerate the transition of materials and process technology, elucidated practices which are now mature and ready for broader dissemination. The achievements of these practices have now been highlighted in a recent lightweighting technology study [1], and the importance of the underlying fundamental databases, now known as the Materials Genome, have been heralded in a recent report of the US President's Office of Science and Technology Policy [6].

Ferrous metallurgy has played a leading role in the creation of this technology [7]. While virtually every microstructural phenomenon occurring in structural materials can be found in ferrous alloys, ferrous systems enjoy the unique position that these phenomena have been studied long enough and deeply enough that theories for them exist, with quantitative validation by fundamental experiments. We here review the development of this technology with an emphasis on the leading examples of computationally designed "cybersteels". European efforts have made significant contributions to this technology, notably including computational thermodynamics and their application [8], computational quantum mechanics applications [9], microstructural simulation tools [10], computational materials design [11], and through process modeling [12,13]. This overview focuses on developments in the USA, which remain unique in their scope of integration.

2. Background: emergence of a design discipline

Founded in 1985 as the Steel Research Group, a multi-institutional materials design consortium has undertaken a quarter century of development of an interdisciplinary systems approach to the science-based computational design of hierarchically structured multiphase, multicomponent materials [7,14,15], grounded in the system of fundamental databases now known as the Materials Genome [6]. Integrating materials science, continuum mechanics and quantum physics, the approach has featured a suite of validated

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computational tools enabled by an iterative interaction of theory, simulation and experiment, spanning the full range of process/structure/property/performance relations. An initial focus on high performance steels, exploiting not only their unique depth of predictive scientific understanding but also their level of fundamental database development, has demonstrated the capabilities of the approach, providing a range of ultrahigh strength ferrous alloys [16–20] now in use in commercial applications. The founding of QuesTek in 1997 as the first computational materials design company has provided both commercial materials design services as well as successful licensing of the designed alloys [21–23], with broadened application of the approach to Al-, Ni-, Ti- and Cu-based alloys. Recognizing the central role of chemical thermodynamics in the science of all materials (as reflected in the structure of undergraduate materials curricula) a fundamental genomic database system centered on CALPHAD thermodynamics [24] has provided a framework for computational materials design with broad applicability, as attested by university projects in the 1990s demonstrating its application to polymers, ceramics and composites [25–27].

The successes in computational materials design in the 1990s helped to bring about the seminal DARPA-AIM (US Defense Advanced Research Projects Agency: Accelerated Insertion of Materials) initiative of the 2000s, expanding the scope of science-based computational materials engineering to embrace the full component level process optimization and material qualification scheme in order to significantly compress the full materials development cycle [28]. Under this initiative the PrecipiCalc precipitation simulator [29,30], grounded in the CALPHAD thermodynamics and mobility database and software system, was efficiently linked to macroscopic component level process simulation tools to successfully accelerate process optimization and accurately forecast manufacturing variation with calibration using minimal datasets for the test case of aeroturbine disc manufacturing. Designed concurrently with the development of the AIM process, the historic first flight of QuesTek's Ferrium S53 landing gear steel in December 2010 represented the first fully computationally designed and qualified material [31], fully exploiting the AIM methodology. As the principal follow-on to the DARPA-AIM successes, the recent 5 year "D3D" Digital Structure consortium program [32], jointly supported by the US Office of Naval Research (ONR) and DARPA, extended the fidelity of AIM modeling tools by integrating a multiscale suite of three-dimensional (3-D) tomographic microstructural characterization tools with a new generation of 3-D microstructural simulators. Combining local electrode atom probe (LEAP), focused ion beam (FIB) and metallographic serial sectioning techniques to span the nanometer, micron and millimeter microstructural scales, respectively, computer-aided tomographic reconstructions have provided a new level of spatial characterization informing novel numerical simulators addressing strength, toughness and fatigue resistance, using advanced steels as the test cases. This has notably included several generations of multiscale ductile fracture simulators [33–36], as well as the direct micromechanical simulation of fatigue nucleation processes at observed nucleants [37]. Extending the 3-D approach to bond topological analysis of quantum mechanical calculations has also provided new heuristics to guide materials design component selection [38,39]. Seeking to accelerate the development of fundamental databases supporting computational materials engineering, the design of niobium-based alloys under the MEANS (Materials Engineering for Affordable New Systems) initiative of the US Air Force Office of Sponsored Research (AFOSR) demonstrated efficient integration of first principles predictions with rapid experimental validation of phase relations in small scale samples [40–42].

3. The predictive science foundation

Early design-driven research to establish a predictive science of precipitation strengthening began with a thorough coordinated multitechnique evaluation of the precipitation hardening behavior of the commercial secondary hardening martensitic steel AF1410, as summarized in Fig. 1 [43]. Field ion microscopy (FIM), transmission electron microscopy (TEM), small angle neutron scattering (SANS), atom probe field ion microanalysis (APFIM), and scanning transmission electron microanalysis (STEM) were combined to quantify the detailed temporal evolution of the M_2C alloy carbide strengthening dispersion in terms of particle size, shape, number density, phase fraction, lattice parameter and chemical composition, correlated with the evolution of alloy hardness. The evolution of size validates the prediction of the Langer–Schwartz theory of precipitation at high supersaturation [44], where the system evolves along a trajectory of unstable equilibrium, enabling space–time separation in its description and control. Particle size can then be controlled through the thermodynamic driving force governing the initial critical nucleus size, while the timescale of precipitation can be independently controlled through a multicomponent coarsening rate constant [45,46] predictable from mobility databases. Observed shifts in lattice parameters and composition of the M_2C phase are consistent with initial coherent precipitation, and X-ray line broadening measurements in related model alloys give supporting evidence of associated matrix coherency strains [47]. The prediction of thermodynamic driving forces controlling particle size has required extension of the CALPHAD chemical thermodynamic databases to incorporate the elastic energy of the coherent state [48,49]. Calibrating analytical strength models to the measurements in AF1410 and related model alloys [50], parametric design controlling the length scales and timescales through the fundamental databases to place the carbide size at an optimal diameter of 3 nm has allowed a 50% strength increase in secondary hardening martensitic steels over previous steels of a given carbon content [51].

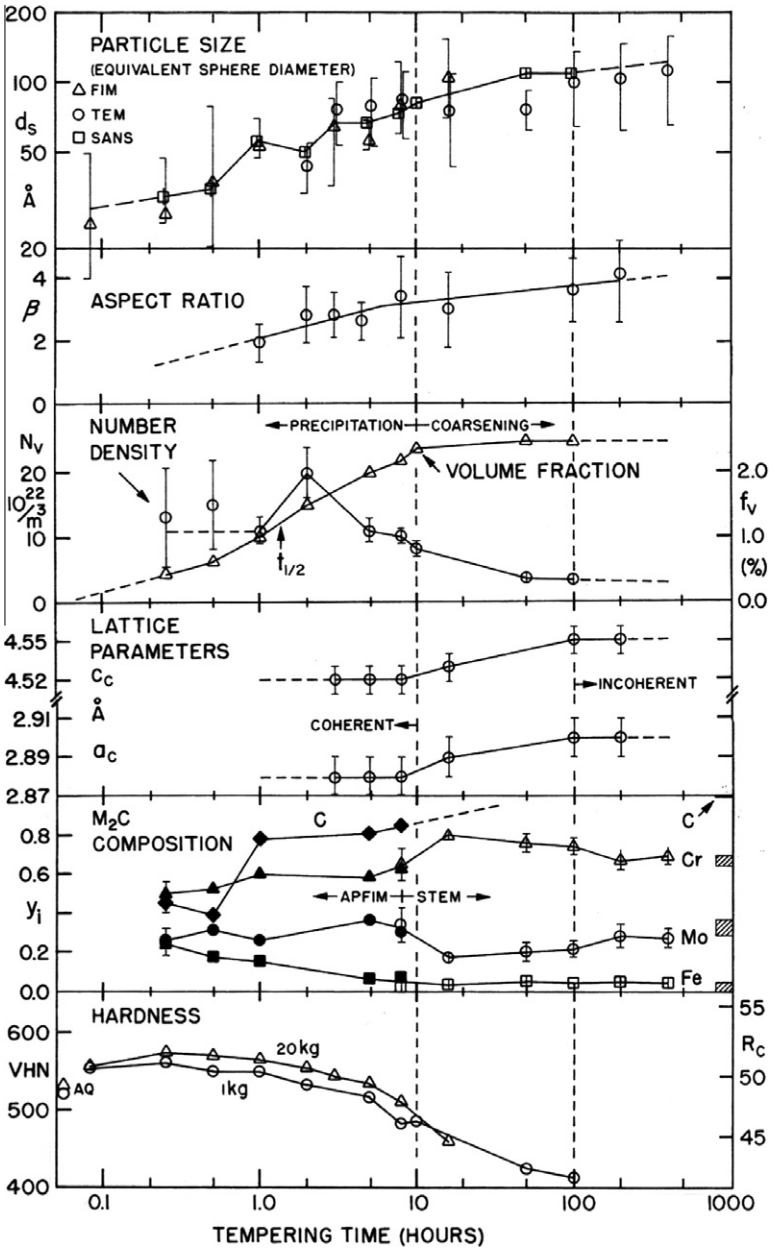


Fig. 1. Multi-technique quantification of the M_2C carbide precipitation hardening evolution in a commercial secondary hardening martensitic AF1410 Co–Ni steel at the standard tempering temperature of 510 °C [43].

Constraining the alloy processability at the ingot stage employs multicomponent diffusion simulation of solidification microsegregation and the subsequent response to homogenization heat treatment [52]. Control of the martensitic transformation temperatures employs the Olson–Cohen nucleation theory [53] supported by a kinetic database describing solution hardening effects in martensitic interfacial mobility [54–56]. Prediction of alloy hardenability employs a related model of bainitic transformation as a coupled displacive/diffusional mechanism [57]. Predictive control of austenite grain refinement employs a calibrated Zener pinning model [58]. Control of cleavage fracture resistance uses a "master curve" model of the ductile–brittle transition in which the transition temperature is expressed as a function of matrix composition, grain size, and alloy hardness. Improved resistance to ductile fracture employs insights from microvoid softening-induced plastic shear localization simulation [35] to optimize the grain refining dispersion, including phase selection for enhanced interfacial adhesion [38]. Further enhancement of the ductile fracture toughness has been demonstrated using transformation plasticity theory [59] to optimize the mechanical stability of dispersed austenite precipitated during secondary hardening, aided by two step tempering treatments [14].

A major barrier to the advance of ultrahigh strength steels has historically been their extreme sensitivity to segregation-induced intergranular embrittlement, as exacerbated by the effect of internally and externally induced

hydrogen. Perhaps the most challenging science problem addressed, an early significant achievement of the SRG design consortium, was the Rice–Wang thermodynamic theory of intergranular embrittlement, predicting the potency of a segregant from the difference in its segregation energy in the grain boundary and free surface environments [60]. While this theory is of universal applicability to grain boundaries in all materials, a unique feature of ferrous alloys is that the embrittlement potency of segregants has been measured along with the segregation energies in the two environments to validate the theory. With such limited thermodynamic data available, and recognizing the relative difficulty of surface thermodynamic measurements, the capability of predicting the underlying thermodynamics was developed through application of the highly precise all electron density functional theory full potential augmented plane wave (FLAPW) quantum mechanical method [61]. Fig. 2 summarizes the correlation of measured embrittlement potency (as a shift in the intergranular ductile–brittle transition temperature with amount of boundary segregant) with the calculated boundary–surface segregation energy difference. The two curves depict predictions with and without the prior segregation of Mn at the boundary, as Mn is a common alloying element in many of the steels for which the embrittlement potencies were measured. The first principles values generally agree with the measured thermodynamic quantities within 0.1 eV atom^{-1}, and show a strong correlation with the relative embrittling potencies of P and S, and the relative cohesion enhancing potencies of B and C. With the predictive capability thus validated attention turned to the less well characterized effects of substitutional alloying elements. After rigorous FLAPW calculations for Mn, Mo and Pd a simplified model was developed using boundary parameters (such as site size) from the FLAPW calculations together with handbook quantities such as elemental cohesive energies to make the prediction of potencies represented in Fig. 3 [62]. After screening for strongly negative values to identify potent cohesion enhancers, these estimates were followed by rigorous FLAPW calculations confirming the high potency of elements such as W and Re. In

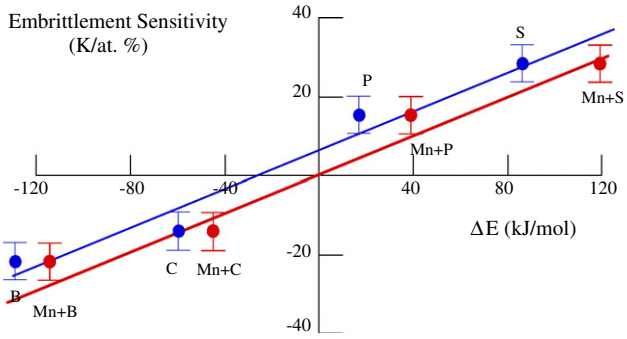


Fig. 2. Measured grain boundary embrittlement potency of B, C, P and S in steels vs. the FLAPW-computed boundary/surface segregation energy difference, with and without prior segregation of Mn [14].

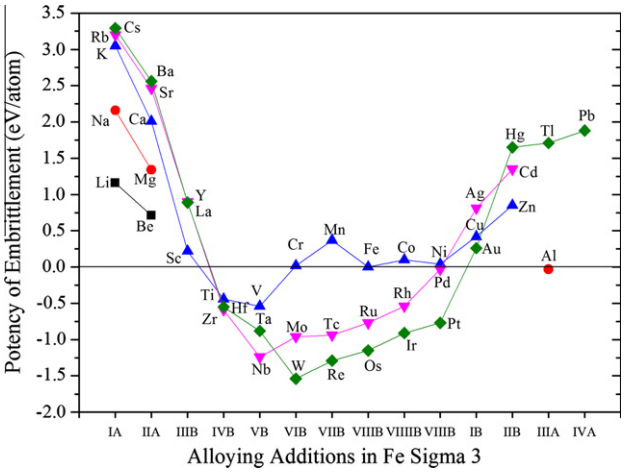


Fig. 3. FLAPW-calibrated model prediction of the embrittlement potency of substitutional elements in Fe grain boundaries [62].

this way a genomic fundamental surface thermodynamic database was assembled to enable design of the boundary composition to offset the effect of H [63]. Integration into alloy design led to the first "quantum steels" in which new directions in alloy composition led by first principles calculations allowed complete elimination of the intergranular mode of stress corrosion cracking in ultrahigh strength steels [14,64].

The resulting suite of computational models supporting materials design is summarized in Fig. 4, ordered according to length scale. At the electronic level quantum mechanical calculations predict surface thermodynamics, at the nanoscale materials science continuum models control precipitation strengthening, at the submicron scale numerical mechanics addresses unit processes of ductile fracture, while at higher scales allotropic transformation and solidification models address processability. Acronyms on the right denote the design models and their software platforms, while acronyms on the left denote the equally important advanced instrumentation allowing calibration and validation for quantified predictive accuracy, which are described in detail elsewhere [14].

4. Computational design successes: flying cybersteel

The first example of a commercial alloy created by computational design using these tools is Ferrium C61 (AMS6517) high durability gear steel, now performing well in off-road racing applications [22,51]. The first such designer alloy to reach flight qualification is Ferrium® S53 (AMS 5922), a corrosion-resistant landing gear steel allowing drop-in replacement for current non-stainless landing gear steels, eliminating the need for cadmium plating [23,31,65]. S53 is a secondary hardening martensitic steel strengthened by efficient M_2C carbide precipitates which contains sufficient Cr to provide passivation against general corrosion. Employing a systems approach to materials design integration [14] the full structural hierarchy of

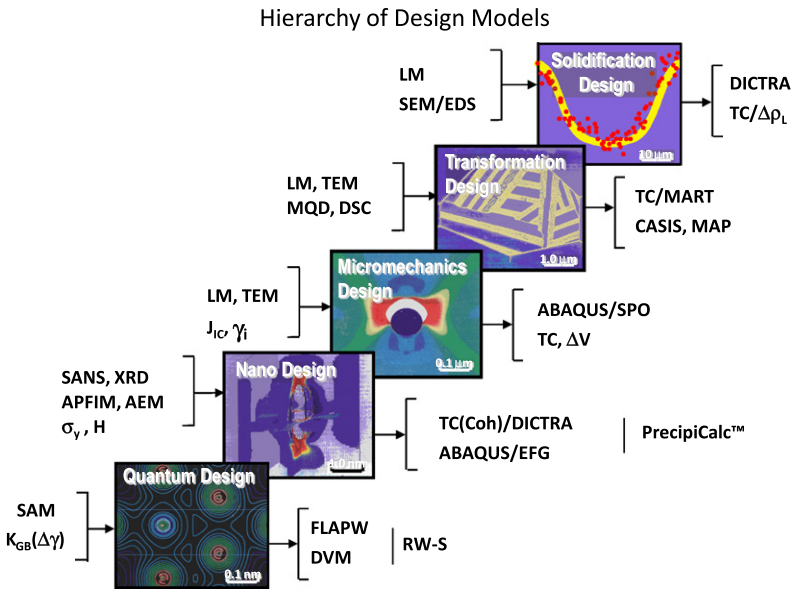


Fig. 4. Multiscale hierarchy of interdisciplinary mechanistic models supporting computational materials design. The acronyms are as described in Olson [14].

the alloy is represented by the flow-block diagram in Fig. 5. The sequential processing steps experienced by the alloy are depicted in the left-most column of the flow-block diagram and are constrained to existing processes for steels employed in current structural aircraft applications to maximize manufacturability. The subcomponents of the alloy system are connected by process–structure and structure–property relationships, as addressed by the suite of mechanistic modeling tools just described.

Application of the system flow-block diagram in conjunction with the computational models begins with the identification of key design trade-offs motivated by the full set of quantitative property objectives represented by the right column. The early conceptual design then defines the specific microstructural subsystems represented in the central column. To address the strong conflict between corrosion resistance and strengthening efficiency a thermodynamic model for the metastable spinel oxide was developed to precisely control the passive film defining corrosion resistance [17]. The multicomponent thermodynamics of the bcc alloy matrix were then exploited to achieve a Cr potential providing a passive film equivalent to a 15Cr

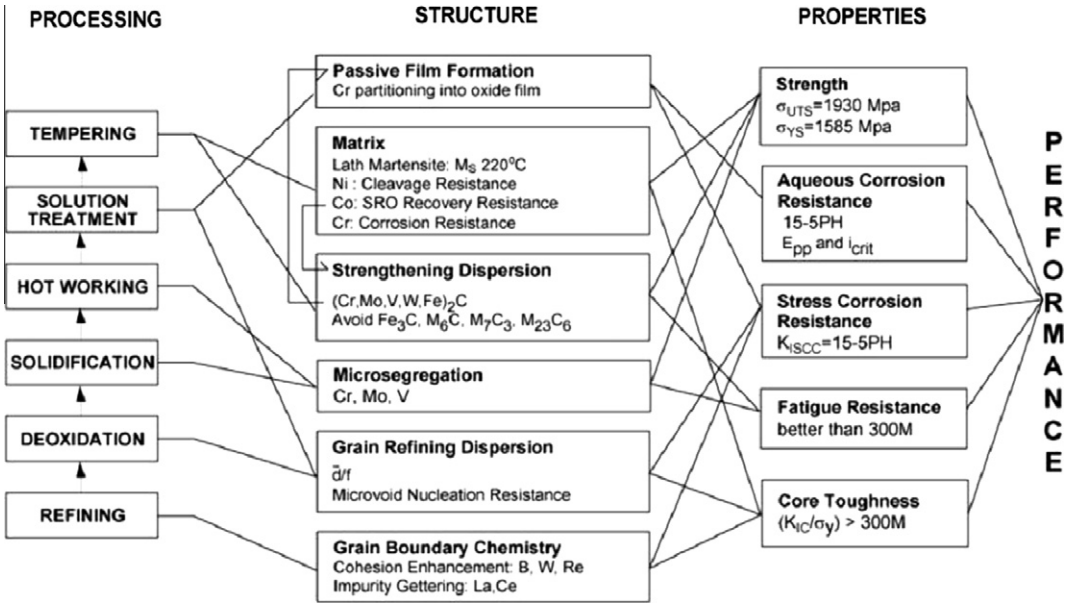


Fig. 5. Flow-block system diagram for ultrahigh strength corrosion-resistant steels, indicating the desired property objectives, the microstructural subsystems, and the required sequence of processing steps. Links between system blocks indicate quantitative models needed to effect the design via science-based computation [31].

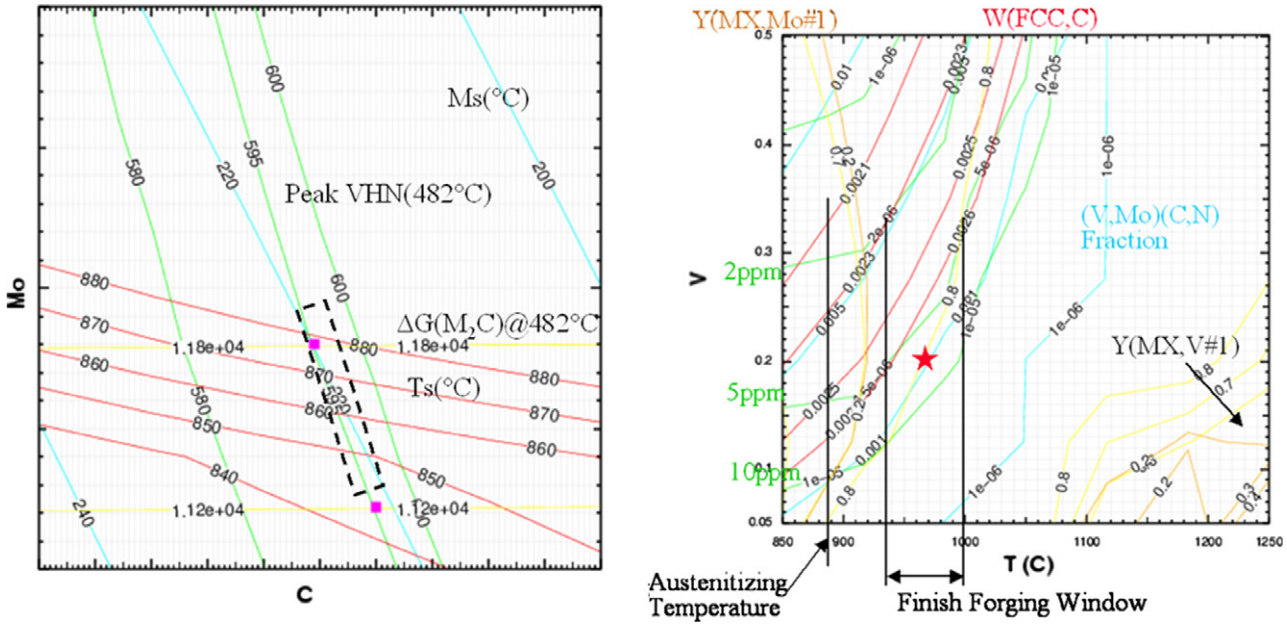
alloy using less than 12Cr in the design. In support of this a detailed reassessment of the fcc/bcc thermodynamics of the base Fe–Cr–Co–Ni system was undertaken to simultaneously maintain accurate control of the allotropic transformation behavior.

Integration of the new information with the models in Section 3 was undertaken by graphical parametric design in which mechanistic understanding motivated the definition of key scalar parameters computable from phase composition vectors and diffusion matrices via the fundamental CALPHAD databases. An example is presented in Fig. 6. With other composition variables fixed by the initial estimates, Fig. 6a depicts the dependencies on two composition variables (Mo and C) of primary importance to the nanoscale carbide strengthening dispersion. Contours of the computed solution temperature T_s and martensite start temperature M_s are superimposed as processability constraints. With a secondary hardening tempering temperature of 482 °C selected from the initial rate constant estimates the contours are shown for the precipitation driving force for coherent M2C carbides (taking into account prior precipitation of metastable Fe3C carbides under "paraequilibrium" constraints against substitutional partitioning). This driving force, together with a total phase fraction defined by the C content, predicts the contours of peak hardness (VHN) defining the outlined region meeting the property requirements within constraints. Similarly, Fig. 6b depicts attributes of the grain refining MX carbonitride dispersion vs. a composition variable (V) and the process temperature (T). Attributes of importance here are the MX phase fraction (for Zener pinning) and component site fraction (for interfacial adhesion). Process temperature windows define constraints of full solubility during homogenization, precipitation during hot work, and stability during austenitizing/solution treatment. In addition to meeting specified parameter values, the slopes defined by the contour plots support sensitivity assessment for robust design.

Using similar cross plots additional constraints on ductile fracture and grain boundary chemistry are used to complete the design optimization and uniquely identify the alloy composition and process temperatures (each with specified tolerances) that represent the best compromise of the diverse design goals and constraints. By iterative design and prototyping with full scale microstructural evaluation to test all models local correction factors are passed back to design models with increased accuracy in the new composition region of interest. In this way accurate design solutions are typically achieved within three iterations of prototyping. For the very challenging objectives of Ferrium S53 design successful achievement of all objectives required five iterations spanning a 3 year period. Experience has shown that the number of required iterations and the associated timescale will steadily decrease as the accuracy of the general CALPHAD databases continues to increase.

5. Extending computational design to qualification and implementation

The design of Ferrium S53 steel ran concurrently with the DARPA-AIM initiative on accelerated materials



(a) Strengthening Dispersion Design

(b) Grain Pinning Dispersion Design

Fig. 6. Parametric design cross plots employed to achieve the required microstructural attributes within the processing constraints for (a) strengthening dispersion and (b) grain refining dispersion [31].

technology transition. The steel was included in the AIM program to serve as a first demonstrator of the new methodology. This began by running the design models within the iSIGHT design integration software [28] to apply Monte Carlo simulation of parametric sensitivities in support of a higher level of robust design practice for reduced intrinsic variation within allowed composition and process tolerances. To circumvent the traditional problem of process scale-up in empirically developed materials Ferrium S53 was the first example of a "design for scale" approach in which process simulation was used to constrain the upfront alloy design to be compatible with full scale processing. As summarized in Fig. 7, solidification simulations employed the DICTRA multicomponent diffusion code [52] for candidate design compositions under the cooling rates of full scale production ingots. The acceptable level of microsegration represented in the first panel was defined by simulating its response to homogenization treatment, as represented in the second. As the production scale was increased sections of as-solidified ingots were obtained to measure the microsegregation for comparison with model predictions, as depicted in the middle panels. The summary of tensile properties in the last panel demonstrates no significant loss of properties (notably including the reduction in area, %RA) with increasing scale from the 300 to the 24,000 lb. level, successfully eliminating scale-up from alloy development.

The greatest challenge of the DARPA-AIM program was forecasting the manufacturing variation to arrive at the minimum design-allowable properties aided by the limited data. Using a new method developed under the AIM initiative, prediction of Ferrium S53 property design minimums, which normally require 10 production heats of alloy and 300 individual observations, were completed using only three heats and 30 individual observations. A large simulated dataset with over 300 simulations was produced using the expected process variation and mechanistic models of alloy strength. The simulated property distribution was calibrated for only 20 experimental observations from two production heats by a linear transformation method. This analysis indicated that the 1% minimum ultimate tensile strength was below the 1930 MPa design goal by at least 10 MPa. This early indication allowed additional process optimization to be completed, increasing the ultimate tensile strength by 10 MPa. The AIM analysis was repeated after a new temper was optimized, using 30 data produced from three heats of the alloy, and is shown by the solid curve in Fig. 8. The new property minimum estimate is 1930 MPa (280 ksi), meeting the property objectives. The resulting full experimental data set of 10 heats and over

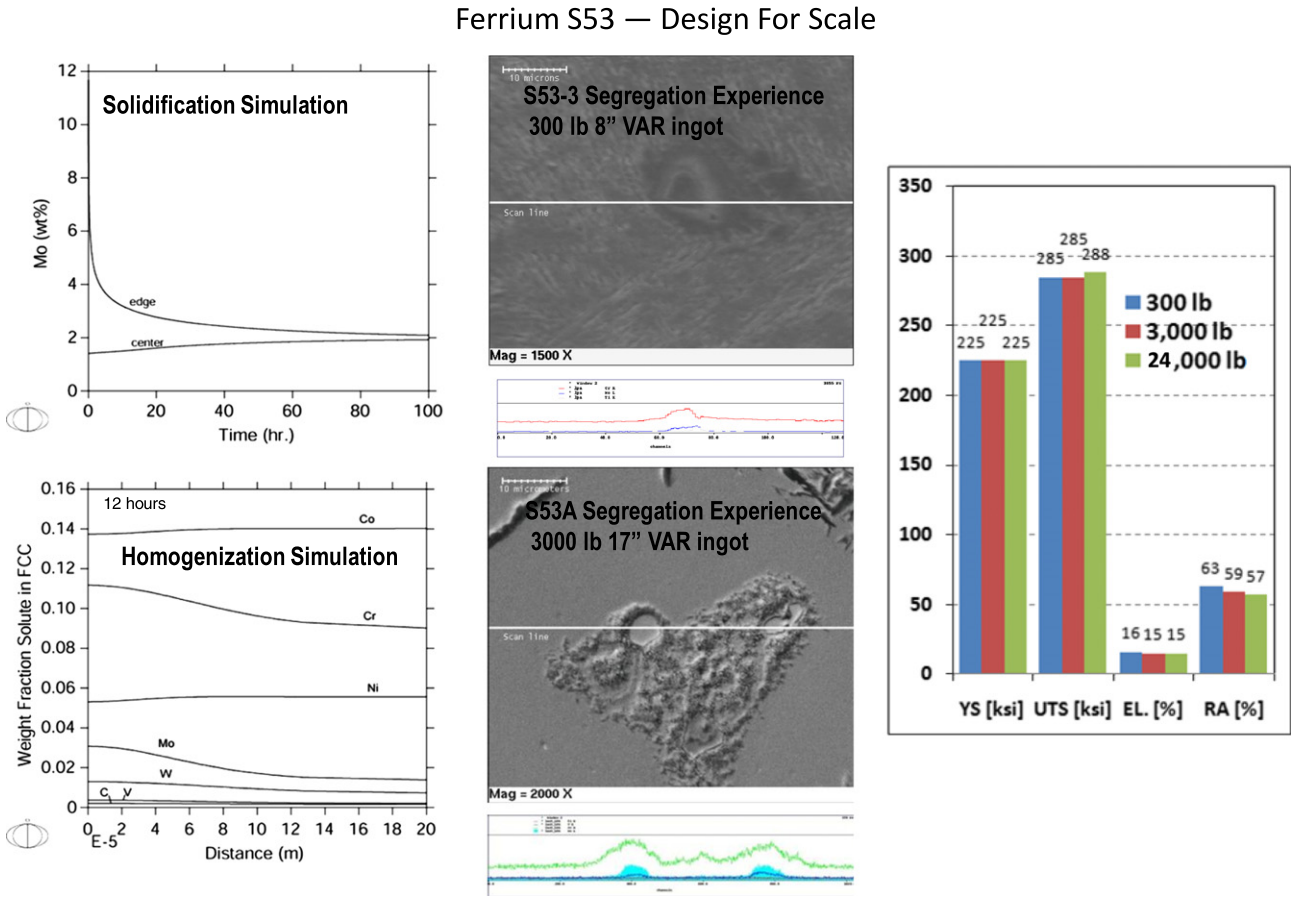


Fig. 7. Simulation of the solidification microsegregation and homogenization response in Ferrium S53, validated by ingot microanalysis, to constrain the design for full-scale production, as demonstrated by no loss of tensile properties with increasing scale [31].

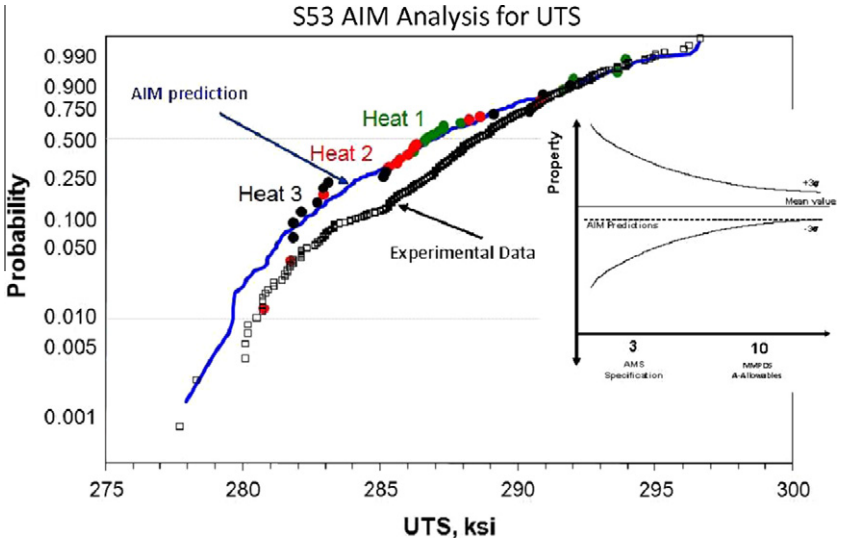


Fig. 8. AIM analysis of the ultimate tensile strength of S53 using simulation results and 30 data from three individual heats agrees well with the completed dataset from 10 heats and over 600 observations in determining the 1% minimum design allowable [31].

600 individual observations are represented by the square points in Fig. 8. The AIM prediction is within 7 MPa (1 ksi) of the 1% minimum tensile strength statistically defined by the 10 heats data. In this AIM example over a year of development and in excess of US$500,000 would have to be expended to meet the property goals if data development had proceeded and the property deficit discovered when the full dataset was complete. The first flight of a Ferrium S53 landing gear occurred on 17 December 2010.

Building on the success of Ferrium S53 a second landing gear steel design for the high toughness levels required for carrier-based aircraft has been undertaken, designated Ferrium M54. Fig. 9 summarizes the timeline of technology readiness levels for these two steels at the landing gear level, along with their corresponding materials development milestones. While the timescale to qualified design allowable for Ferrium S53 was 8.5 years, Ferrium M54 met its objectives in the first iteration of design, and is on track to reach materials qualification within 5 years.

6. Research/education integration

Recognizing that engineering disciplines are defined by what can be practiced with a Bachelors degree, it was the vision of the founders of the SRG design consortium in

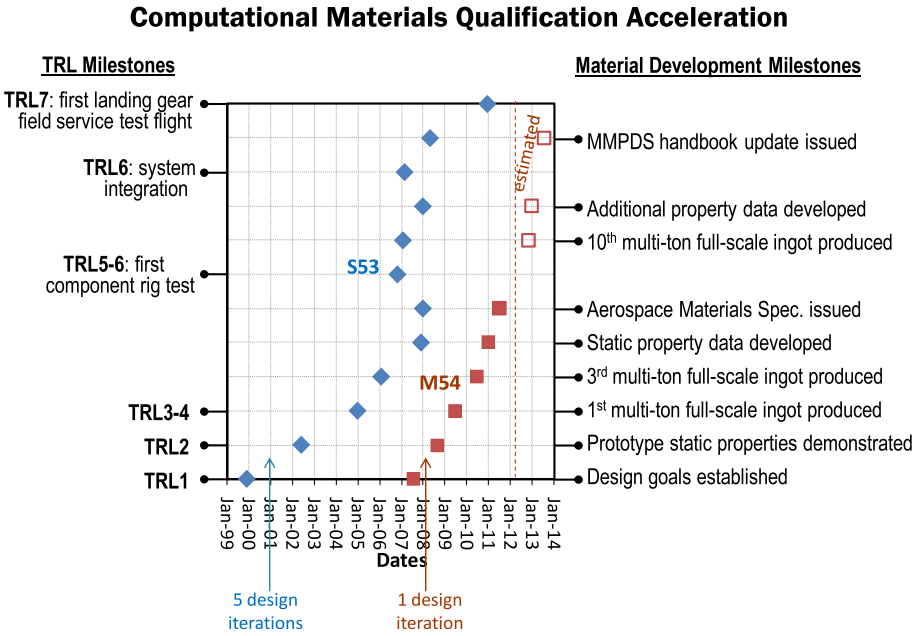


Fig. 9. Time line of landing gear level technology readiness levels (TRL) and the corresponding materials development milestones for computationally designed S53 and M54 alloys.

1985 to develop a methodology of computational design that can be taught to undergraduates. This has motivated a series of materials design education innovations at Northwestern University, beginning with the teaching of an undergraduate Materials Design course in 1989 [66]. In a unique integration of research and education annual iterations of theoretical design optimization techniques employing the newest experimental measurements and model/simulation predictions are conducted by teams of materials science undergraduates in this novel class. The course features a series of computational sessions teaching the suite of computational design tools grounded in the materials genome fundamental databases [67] and the graphical parametric design integration approach of Fig. 6. Fostering a high level of technical design, the undergraduate teams are coached by doctoral students participating in funded design projects [68,69]. These doctoral students are in turn assisted by a broader group of graduate students contributing to projects under a special interdisciplinary doctoral cluster program in Predictive Science and Engineering Design (PSED) supported by Northwestern's Graduate School. A seminar course under the PSED program explores the role of computational tools in the interdisciplinary integration of predictive science and engineering design. A central outreach activity to introduce the new technology to a broader audience is a new masters (MS) certificate program in Integrated Computational Materials Engineering, in which first year MS students also participate in the interdisciplinary PSED seminars, leading to a culminative integrative project within the Materials Design course. Undergraduates taking the Materials Design course in their third year can participate in the experimental validation of their design prototypes in their senior projects in the following year. To enhance recruitment to the materials program teams of students from a special "Murphy Scholars" section of a freshman level Engineering Design and Communication course also collaborated with the undergraduate design teams, adding exploration of device applications of the new materials [70]. As summarized in Fig. 10, the design, development, and

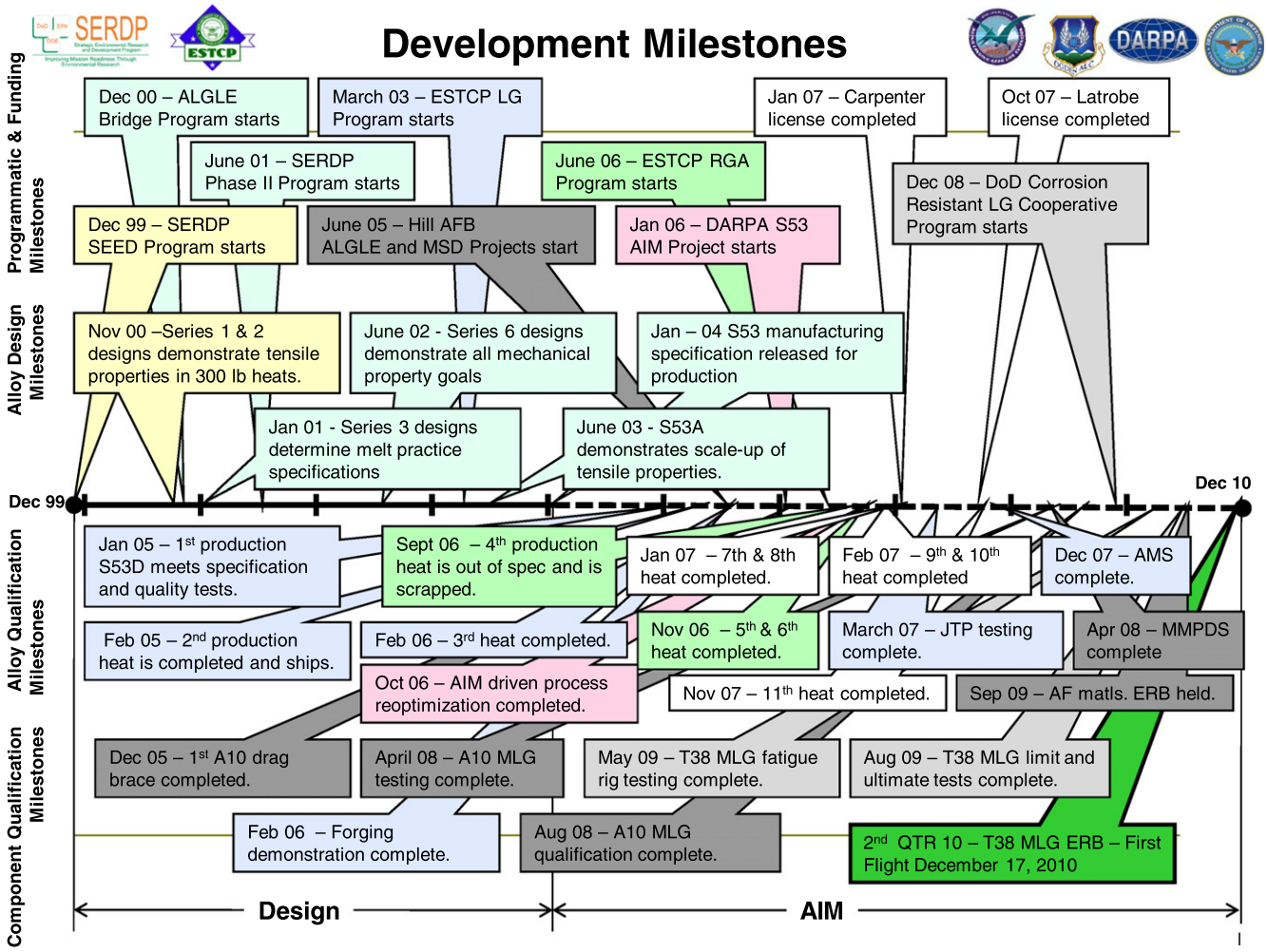


Fig. 10. Case study summary chart of the computational design and qualification of Ferrium S53 landing gear steel. The top level represents the programmatic and funding milestones of the multi-agency effort. The second level represents iterative alloy design milestones leading to the composition and processing specifications. The bottom levels represent alloy and component level AIM-based qualification, leading to the first flight in December 2010.

accelerated flight qualification of Ferrium S53 as the first fully computationally designed and qualified material now serves as the central case study for all of these education initiatives.

7. Conclusions

Moving beyond a system of "technology by accident" grounded in near random walk scientific discovery to a new system of tightly integrated science-based engineering constitutes a true revolution in materials technology, offering substantial societal benefit, as well as a new justification for scientific investment. Its emergence has re-established ferrous metallurgy as the true intellectual frontier of the materials enterprise. Our unique depth of scientific understanding in ferrous alloys will continue to serve as the central template for a general methodology of computational design for all materials, fully grounded in fundamental genomic databases.

Acknowledgements

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