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Investigation of Fundamental Mechanisms for Multi-Scale Modeling of Complex Concentrated Alloys for Aircraft Structural Applications

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14. ABSTRACT

The principal objective of the proposed program is to investigate fundamental mechanisms related to phase stability/transformations, deformation, and oxidation of complex concentrated alloys (CCAs), including the sub-class of single solid solution high entropy alloys, for high temperature aircraft structural applications. This effort will focus on the application of multi-scale characterization and modeling tools towards the understanding of these fundamental mechanisms. While these novel CCA alloys hold a lot of promise for future application, there exist a lot of gaps in the fundamental understanding and knowledge base related to these concentrated alloys involving complex chemistries and microstructures. Hence the motivation for the proposed program. While there are an exponentially expanding number of CCAs being developed and investigated by numerous research groups worldwide, with wide ranging elements, phases, microstructures and properties, the present proposal will be focused only on those CCAs that can potentially be applied for high temperature structural applications of interest to the U.S. Air Force. Therefore, the group of CCAs to be investigated will involve microstructures consisting of the body centered cubic (bcc) and ordered B2 phases, and typically involve refractory elements. Additionally, fundamental mechanisms in FCC based CCAs, containing ordered intermetallic precipitates were also investigated.

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Objectives:

The principal objective of the proposed program is to investigate fundamental mechanisms related to phase stability/transformations, deformation, and oxidation of complex concentrated alloys (CCAs), including the sub-class of single solid solution high entropy alloys, for high temperature aircraft structural applications. This effort will focus on the application of multi-scale characterization and modeling tools towards the understanding of these fundamental mechanisms. While these novel CCA alloys hold a lot of promise for future application, there exist a lot of gaps in the fundamental understanding and knowledge base related to these concentrated alloys involving complex chemistries and microstructures. Hence the motivation for the proposed program. While there are an exponentially expanding number of CCAs being developed and investigated by numerous research groups worldwide, with wide ranging elements, phases, microstructures and properties, the present proposal will be focused only on those CCAs that can potentially be applied for high temperature structural applications of interest to the U.S. Air Force. Therefore, the group of CCAs to be investigated will involve microstructures consisting of the body centered cubic (bcc) and ordered B2 phases, and typically involve refractory elements. Additionally, fundamental mechanisms in FCC based CCAs, containing ordered intermetallic precipitates were also investigated.

These objectives have been addressed via the following task. The results aid in addressing key issues and fundamental gaps in our understanding that limit the potential application of these next generation materials for aircraft structural components. The completed tasks have been briefly outlined below:

Task 1: *Fundamental investigation of phase stability and transformation mechanisms in CCAs*

The fundamental gaps with respect to phase stability and the underlying phase transformation mechanisms leading to the *complex multi-scale and multi-phase microstructures in CCAs* were investigated. These fundamental mechanisms were directly fed into multi-scale models to predict and control microstructures in refractory metal CCAs. Based on a combination of high throughput computational techniques (in collaboration with AFRL partners) and combinatorial experiments, promising alloy compositions that had been optimized for the bcc and B2 phases were identified and investigated, coupled with critical properties such as stiffness, and tensile/compressive properties. As a validation, we used the new knowledge provided by this improved modeling capability to intentionally invert current microstructures that consist of discrete bcc precipitates surrounded by a continuous B2 matrix. This phase inversion overcame the brittleness associated with the present microstructure as expected, resulting in high compressive ductility. On the other hand, phase stability in Al/Ti containing 3d transition metal (Co-Cr-Fe-Ni) based CCAs was also investigated. This study resulted in a thermodynamics-based alloy design strategy to control and tune the degree of local chemical ordering in multi-component alloys. Progressive increase in Al and Ti concentration led to alloys with a near-random solid solution, short-range or long-range ordering. Interestingly, good tensile ductility was maintained in all alloys while simultaneously increasing the yield stress.

Task 2: *Fundamental investigation of deformation mechanisms in CCAs*:

The deformation mechanisms in candidate CCAs, identified from Task 1, were investigated in order to develop strategies for optimizing these to achieve acceptable levels of tensile ductility and toughness. These studies were conducted on both single-phase bcc-based CCAs, B2 precipitation strengthened bcc-based CCAs, as well as $L1_2$ precipitation strengthened fcc-based CCAs. There were multiple challenges with respect to deformation mechanisms that were investigated. Firstly, the challenge of slip transmission from the disordered bcc phase to the ordered B2 phase. Secondly, the challenge of sufficient slip systems operative within the ordered B2 phase at different temperatures. Thirdly, the tension-compression asymmetry inherent in the deformation of bcc phases.

The post-deformation microscopy of 3d transition metal-based alloys has also revealed interesting results. Local chemical ordering in these alloys appeared to contribute to a planar slip-based strain hardening mechanism. These results establish a new design paradigm for CCAs, whereby, intentionally introducing local chemical ordering within the single phase solid solution, based on thermodynamic principles, can not only increase the yield stress, but also trigger a planar slip initiated strain hardening mechanism, leading to a better balance of strength and ductility.

<u>Task 3:</u> Additive manufacturing of CCAs: Additive manufacturing have been utilized to accelerate the exploration of compositional space in transition metal based CCAs by investigating compositionally graded CCAs. Additionally, the effect of additive manufacturing process on the microstructure and associated mechanical properties of precipitation strengthenable FCC based CCAs were investigated in detail.

Accomplishments:

Under this project, we have investigated two class of alloy systems – BCC Al-Nb-Mo-Ta-Ti-V-Zr based refractory high entropy alloys (HEAs)/ complex concentrated alloys (CCAs) and FCC Al-Ti-Co-Cr-Fe-Ni based HEAs/CCAs.

FCC-based HEAs/CCAs:

During the initial period, we focused on the phase transformation pathway in the RHEA, $Al_{0.5}NbTa_{0.8}Ti_{1.5}V_{0.2}Zr$ which showed an optimized strength ductility combination. Typically, Al-containing refractory high-entropy alloys (RHEAs), comprising a two-phase ordered B2 + BCC microstructure resembling the FCC+$L1_2$ superalloy microstructure, exhibit extraordinarily high yield strengths, but poor ductility at room temperature, limiting their engineering application. The poor ductility is attributed to the continuous matrix being the ordered B2 phase in these alloys. Our initial study presented a novel approach to microstructural engineering of RHEAs to form an “inverted” BCC + B2 microstructure with discrete B2 precipitates dispersed within a continuous BCC matrix, resulting in improved room temperature compressive ductility, while maintaining high yield strength at both room and elevated temperature. Following this study, the microstructural evolution in this RHEA during phase inversion phenomenon was also studied in detail. Quenching from a high temperature single phase field, this RHEA exhibits a co-continuous mixture of a disordered BCC and an ordered B2 phase, that upon isothermal annealing at 600 °C develops via spinodal decomposition into a continuous B2 matrix with discrete cuboidal BCC precipitates aligned along the <001> directions. Longer term annealing at 600 °C results in the development of necking constrictions along the B2 channels, eventually pinching-off these channels and making the BCC phase continuous with discrete B2 precipitates. This inversion process was found to be driven by a reduction in the total interface energy and total elastic strain energy. This phenomenon of "phase inversion", is presumably the first ever experimental evidence in metallic alloys.

In addition to the $Al_{0.5}NbTa_{0.8}Ti_{1.5}V_{0.2}Zr$ RHEA, we also studied $Al_{0.5}Mo_{0.5}NbTa_{0.5}TiZr$ RHEA which also exhibited a superalloy-like microstructure comprising a two-phase ordered B2 + BCC microstructure, exhibit extraordinarily high yield strengths, but poor ductility at room temperature. This study established that annealing of such microstructures at high temperatures for extended time periods can lead to the formation of ordered omega type phases, derived from the parent B2 phase. The solutionized condition of this alloy consists of a co-continuous mixture of BCC and B2 phases, which transforms via spinodal decomposition concomitant with ordering, into a continuous B2 matrix with discrete BCC precipitates at the early stages of annealing. With increasing annealing time at 800 ° C, isolated islands of BCC phase embedded with discrete, fine-scale pockets of the B2 and hP18 (P63/mcm) phase develop, where the hP18 phase is an ordered derivative of the omega phase, commonly found in titanium and zirconium base alloys. The transformation of the parent B2 phase to the hP18 ordered omega type phase can be rationalized via a combined displacive and diffusional transformation, including rejection/enrichment of certain alloying elements. First-principles based DFT calculations were also carried out to compare the stability of the B2 versus hP18 phases, as a function of composition. Although hP18, B2 and BCC all co-exist following long term annealing, hP18 does not directly nucleate within the BCC grains but prefers to form via the intermediate B2 phase, indicating a high nucleation barrier for homogeneous nucleation. The universal nature of this phenomenon is demonstrated using examples from other RHEAs, and the resultant impact on the high temperature stability of the BCC + B2 microstructure is discussed.

Advancing the study on RHEAs, we focused our attention on developing a new ductile RHEA based on the composition of the B2 phase in a two-phase BCC+B2 mixture. While this new alloy was expected to be a single B2 phase, on processing this alloy exhibited a nano-scale mixture of co-continuous BCC and B2 phases. Interestingly, this alloy exhibits excellent room temperature compressive yield strength (~1075MPa) and high strain to failure ~55%. Deformation studies on this new RHEA are presently underway.

FCC-based HEAs/CCAs:

On the other hand, we have studied the competing precipitation between two intermetallic phases, L12 and B2 in an FCC based HEA, Al0.3CoFeNi. We found that thermomechanical processing can be used to produce a complex four-phase mixture in this alloy that results in ~1500 MPa yield stress with ~12% tensile ductility at room temperature. In addition, we have also investigated two new aspects of these alloys in the following reporting period. In the FCC based HEAs, we have studied atomic distributions in random HEA solid solutions and their propensity for local chemical ordering. We have developed a simple solution thermodynamics approach for tailoring the local ordering tendency in HEAs from a random solid solution in a model CoFeNi alloy, to short and long-range ordering, by adding controlled amounts of Al and Ti. This change in the degree of chemical ordering has a strong influence on the tensile yield strength and ductility of the alloy.

Following this study, we focused our attention on the FCC Al-Ti-Co-Cr-Fe-Ni based HEAs. We have developed a thermodynamics-based approach for tuning the chemical ordering in HEAs. This change in the degree of chemical ordering has a strong influence on the tensile yield strength and ductility of the alloys. During the current reporting period, we have investigated the deformation mechanisms of these alloys. We found that strength-ductility paradox can be surmounted by introducing and tailoring the extent of ordering. Post-deformation microscopy revealed that chemical ordering causes localized slip along multiple {111} planes whose subsequent interactions trigger a dynamic Hall-Petch like effect by continually refining the slip length. The progressively increasing local order systematically correlates with enhancement of yield and flow stresses, strain hardenability, and ductility.

Summary of principal contributions resulting from this project:

The fundamental mechanistic understanding related to CCAs (including RCCAs) that was developed under this program can be summarized in the following points. Details regarding each of these points are available in the cited references.

1. Synergistic/concomitant spinodal decomposition (phase separation or clustering) accompanied by chemical ordering tendencies within the complex concentrated BCC solid solution of the RCCA leads to the homogeneous BCC + B2 microstructure, resembling that of superalloys [1, 8].

2. Stability of the ordered B2 phase at high temperatures, especially in multi-component systems, is not well-established. Often the B2 phase forming in RCCAs is a metastable phase which transforms to an equilibrium ordered omega-type phase ($B8_2$ or $D8_8$ type) on long-term annealing at elevated temperatures [4, 5, 7].
3. BCC + B2 forming RCCAs often exhibit a continuous ordered B2 matrix with discrete BCC precipitates, which can be described as an inverted microstructure with respect to traditional superalloys. The continuous B2 matrix presumably leads to poor ductility/plasticity in the alloy. The results from the previous program clearly demonstrated that the microstructure in RCCAs can be inverted to form a continuous BCC matrix with discrete ordered B2 precipitates [1, 2]. However, the fundamental mechanisms and phase transformation pathway underlying this phase inversion need further investigation in order to develop a predict modeling capability.
4. Thermo-mechanical processing is a powerful tool to engineer the phase transformation pathways in CCAs (including RCCAs) and can be effectively employed to accelerate the formation of equilibrium phases versus transitionary metastable phases [7, 15, 29].
5. The ordered intermetallic phases forming in CCAs, such as B2 and $L1_2$, typically have multi-component chemistries, with complex site occupancies. Such complex site occupancies could potentially influence the nature of inter-atomic bonding in these intermetallic phases, impacting their deformation mechanisms and mechanical properties [13].
6. Interestingly, alloys designed based on the composition of the B2 phase in a two-phase BCC+B2 mixture in an RCCA equilibrated at high temperatures, did not form a single B2 phase. Rather these compositions decomposed into a nanoscale interconnected mixture of BCC+B2, resembling a spinodally decomposed microstructure. These alloys can exhibit excellent compressive plasticity at room temperature unlike most other RCCAs [5, 6].
7. With regards to FCC-based CCAs, based on 3d transition elements, the B2 intermetallic phase was found to be a potent strengthening phase in the FCC matrix, in addition to the well-established $L1_2$ (gamma prime) phase. High temperature FCC + B2 phase stability can be achieved in these CCAs [12, 14, 29].

8. FCC-based CCAs containing higher Al concentrations lead to a competition between stabilities of both the FCC and BCC phases (since higher Al content promotes BCC formation in CoCrFeNi based systems). Additionally, higher Al also promotes a strong chemical ordering tendency in these CCAs. This competition has been exploited to design a novel nano-lamellar eutectoid-like alloy consisting of alternating lamellae of FCC + L12 and BCC + B2 with exceptionally high room temperature yield strength (>1500 MPa) while preserving ~15% tensile ductility [20].

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