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**CHARACTERIZING HIGH ENTROPY ALLOYS
FOR HYPERSONIC APPLICATIONS**

April 23, 2020

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ABSTRACT

In this paper, the properties of a new and broad class of materials, high entropy alloys (HEAs), were investigated and evaluated for hypersonic applications. The plan was to identify candidate hypersonic HEAs and model the high-temperature strength using new advanced material models that account for asymmetry and anisotropy characterized with available test data. After accessing a local database of HEAs and their material properties in collaboration with Dr. Gorsse et al., it was realized the knowledge of HEAs is currently very broad but lacks depth. While hundreds of HEAs have been created and tested, none so far have both sufficient data and the desired properties to more accurately model high-temperature strength. Further research is required to begin bringing HEAs into the world of constitutive modeling and simulation, which is the next step to real world applications. Several HEAs have been identified as having high potential for further research.

INTRODUCTION

High temperature material performance is currently one of the top priorities of Air Force research, as the extreme conditions of hypersonic flight continue to be explored. High entropy alloys (or HEAs) are alloys composed of 5 or more metallic elements of 5 to 35 atomic percent each and are named for the high atomic entropy that results from having multiple principle elements. This relatively new type of alloy was introduced on the principle that the increased entropy would result in a much more stable solid solution. The atoms of various sizes and various bonding energies also cause a more disordered lattice structure. This helps slow displacements, due to the large fluctuations in lattice potential energy between lattice sites, which increases the strength of the material [1,26]. The increased entropy of mixing makes intermetallic phases less likely to occur, preventing regions of brittle material within the microstructure. When these secondary phases do occur, they are usually small and well dispersed, and thus can facilitate dispersion strengthening, especially with proper heat treatment [2]. Dispersed intermetallic phases especially are ideal for high temperature materials [1]. If intermetallic phases are considered undesirable for a specific application, HEAs can be designed to prevent them as well, by choosing combinations of similar group number metals. These elements have smaller differences in

electronegativity and are more likely to form a solid solution. This is consistent with the Hume-Rothery rules, although they are only desired for binary alloys [29].

Since HEAs can be composed of 5 or more elements, there are numerous potential combinations. After eliminating impossible elements, such as noble gases, radioactive elements, and metals that are liquid at room temperature, 45 elements can be combined into HEAs. Even when only looking at equimolar combinations, there are more than one million potential 5-element combinations, and one hundred billion potential 13-element combinations [1]. For a sense of scale, the difference between those two numbers is the same as the difference in size between Mt. Fuji and the sun [3]. Most samples of HEAs are created by arc-melting the elemental components, which may be in granules or some other form, under a vacuum or argon atmosphere. It is standard for the samples to be flipped and remelted four or more times to improve chemical homogeneity [20-24].

Motivation and Objectives

For this project, high entropy alloys were investigated to see if they are suitable for hypersonic applications. Hypersonic flight is defined as flight reaching speeds above Mach 5 [27]. At these speeds, “the temperature of the flow around the aircraft is so great that the chemistry of the gas must be considered [28]”, and in the molecular bonds can vibrate or even break, which would create plasma. Shockwaves and expansions cause massive variations in air density and pressure that need to be considered as well [28]. Due to these extreme conditions of high speed, temperature, and pressure, finding alloys that are sufficiently light, strong, heat resistant, and suitable for manufacturing is not a simple process. High entropy alloys add an entirely new world of alloys to explore. One of the most intriguing aspects of this combination is the potential to create customized HEAs. Creating and using composition models such as the rule-of-mixtures means that the properties of individual elements can be used to estimate the properties of the resulting alloy [1]. Rather than adjusting a design to known alloys, eventually it could become possible to enter one’s material parameters into a computer and be given an output of potential HEAs and their costs. This would reduce the need for redesign based on material limitations. It would especially help with highly specialized designs used in extreme environments, such as in space or on the ocean floor.

There are some drawbacks to HEAs, but most of these can be viewed as initial investment costs, which will be lessened by research and testing. One of the most obvious is that because there are so many potential HEAs, it will take a great deal of time and money to investigate these alloys. That is why it is so important for accurate, predictive composition models to be developed in order to narrow down which HEAs should be investigated, as the rule-of-mixtures is only accurate for predicting density and modulus of elasticity [1]. There also needs to be research into potential negative attributes of HEAs, such as unwanted oxidation or brittleness, which may render certain alloys inviable. Additionally, there is some cost to creating a unique alloy and introducing it to manufacturing. This will probably limit the use of tailored HEAs to projects with large budgets at first. Hopefully, these options will grow more widely available as HEAs become more commonly used and manufacturing becomes cheaper and more efficient.

Modeling the mechanical behavior of known alloys (known as constitutive modeling) accurately is a key aspect of this project, as accurate constitutive modeling can reduce waste and cost. The more accurate the models and simulations, the smaller the factors of safety that need to be used. This is vital when looking at manufacturing processes that can drastically change the properties of a material.

Although hypersonic application has frequently been mentioned when discussing high entropy alloys, it is usually a brief suggestion of potential application for the alloy in question. Some have even begun working on composition models which predict the properties of previously unexplored HEAs, but it seems that few if any have begun to move from gathering data on HEAs to attempting to use constitutive models for individual alloys, which is not surprising, given how new they are. This project explored relatively untouched territory.

EXPERIMENT

The first step was to identify a source of information that organized the hundreds of HEAs that have been researched to date. A database was found that had been created by Gorsse et al., one of the

only public databases on HEAs that currently exists [4][5]. Dr. Gorsse was contacted and generously sent a copy of the database in Excel.

The database currently has entries on almost 400 alloys and is still growing. There are some duplicates from different experiments, but even accounting for those, the number of unique alloys is quite large. These alloys were then compared to other conventional alloys in Ashby diagrams. It should be kept in mind that most HEAs being tested at this time are known to be likely to have good properties, so the current figures are likely not representative of all HEAs. In Figure 1, alloy groups of interest and the range of the current HEA database are labeled, in blue and orange. Red and green regions are standard ferrous and non-ferrous alloys, respectively.

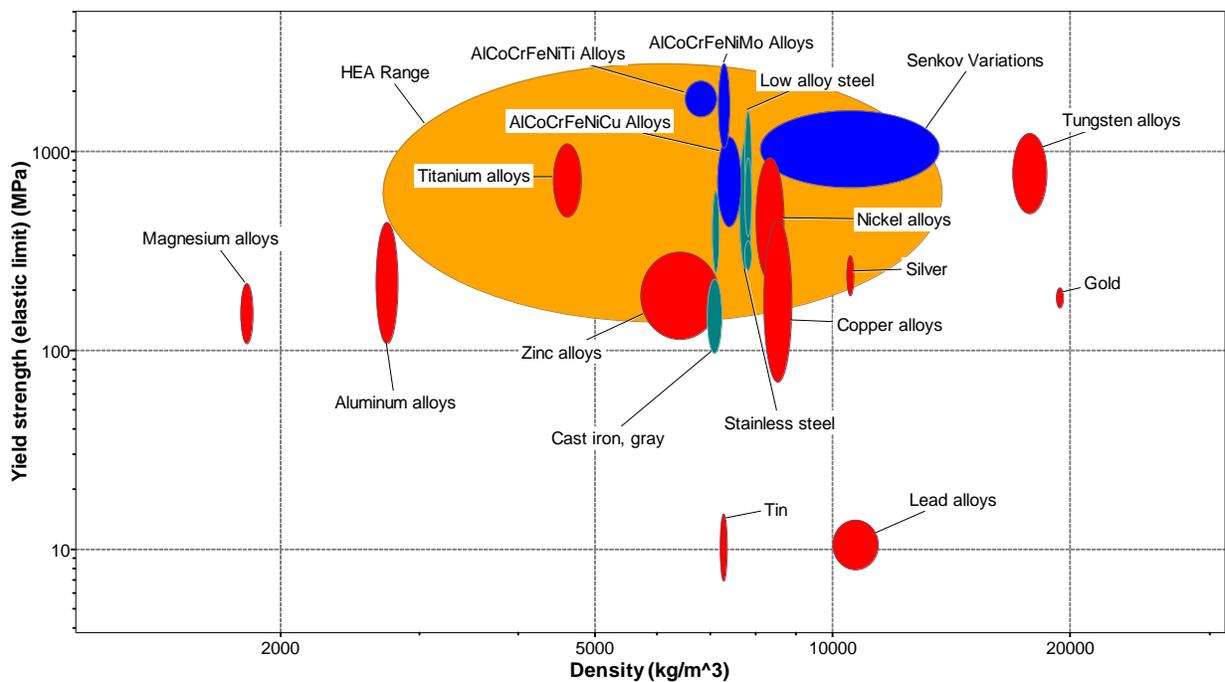


Figure 3 – Ashby Diagram of Metal Alloys, with alloys of interest labeled, created on GRANTA EduPack 2020. [4][5]

There is not as much data on HEAs over a range of temperatures. As can be seen in Figure 2, the HEAs that were tested also tend towards a higher strength at high temperature. Since there are no official “maximum service temperatures” at this time, the ranges shown for the HEAs are the highest temperatures at which a material was tested and the yield strengths at those temperatures. The yield temperatures for these alloys at room temperature are much higher. The metals are color coded as

mentioned above, while some other materials of interest include technical and non-technical ceramics, which are yellow and brownish green respectively.

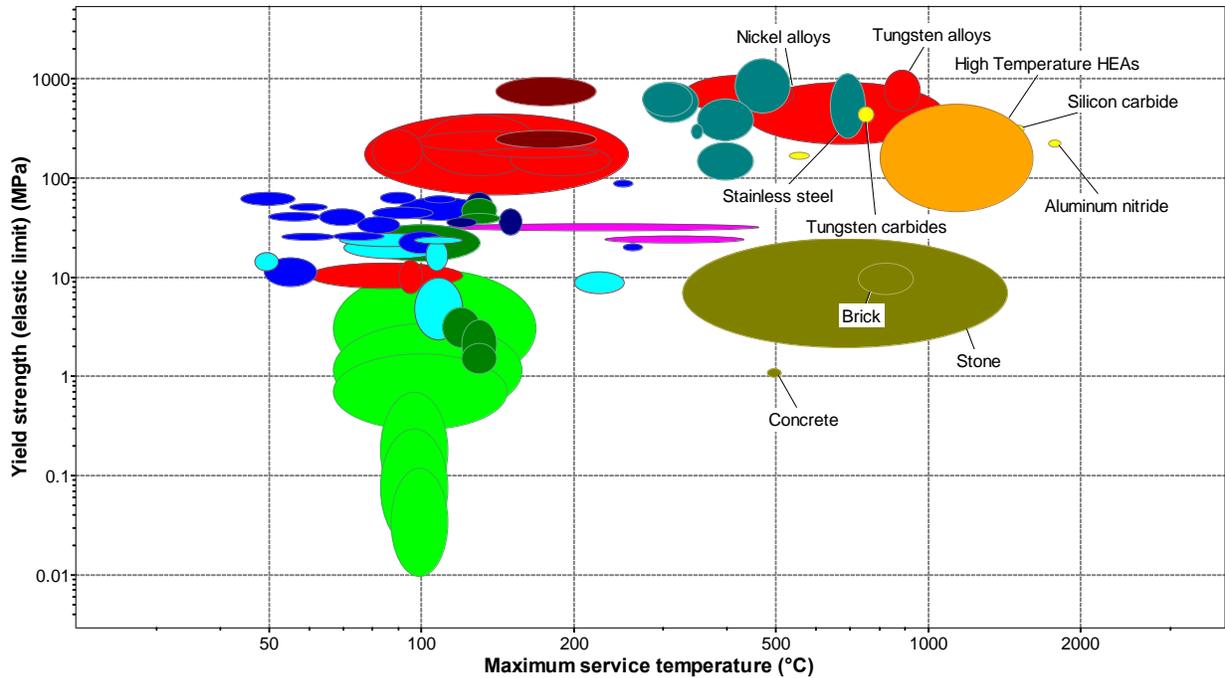


Figure 4 – Ashby Diagram Comparing Maximum Service Temperature with Yield Strength, overlaid by the corresponding scatterplot of High Entropy Alloys, created on GRANATA EduPack 2020 [4][5].

In this project, HEAs were selected that were thought to have good properties, mostly high specific strength, or high specific strength at high temperature. After looking at the options, it was decided to focus on one promising alloy as a starting point, and then, after modeling one alloy, it would become easier to model other alloys. The Senkov alloy was explored first, and along with its elemental and medium entropy alloy (MEA) components. Unfortunately, there was not enough information available to properly model the alloy. Since it is one of the most researched HEAs, aside from CrMnFeCoNi [6], it seems unlikely that there is currently sufficient information to model any other HEAs. More research and testing is required to determine if this HEA is the most appropriate for hypersonic applications. As an extension of the research done with AFRL over the summer, this semester a few more alloys of interest were identified. These also lack sufficient data for modeling at this time, but seem to merit further exploration as well.

SENKOV ALLOY: AN IN-DEPTH LOOK

The high entropy alloy that became the primary focus of this survey was the refractory HEA HfNbTaTiZr. It can be found under the name TiZrHfNbTa as well, since there a standardized naming system for HEAs doesn't exist at this time. It is also known as the Senkov alloy, named after Dr. Oleg Senkov, who has done extensive research on this HEA. It was determined that this RHEA (refractory HEA) is a material that would be very useful for hypersonic applications and requires more research. It has many characteristics that make it ideal for hypersonics. The first is its refractory nature, which means it is resistant to decomposing at high temperatures. It has a melting temperature of 2514K [7], which is 572.8K hotter than the melting temperature of titanium [17].

Additionally, it has a good combination of strength, ductility, and moderate density. The density is 9.9 g/cm³, which is about average for an HEA [7]. This could be reduced by adding aluminum or some other light element, which has been studied, but was not investigated for this paper [14]. It also is one of the few known body-centered cubic HEA that is ductile at room temperature, which is ideal for manufacturing and cold-working [18]. HfNbTaTiZr has a tensile strength of 1155 MPa [7] and a compressive strength of about 1015 MPa [8], without any special processing. Grain refining, annealing, or other treatments can increase or decrease the material's strength. Grain refinement can increase the yield strength to up to 1600 MPa with very little loss in ductility [9]. Annealing without additional treatment can result in a decrease of about 100 MPa [10]. The modulus, ductility, and hardness presented in the table below can also be changed depending on the treatment of the material.

This RHEA also experiences strain hardening. It weakens somewhat as temperature increases, but this is partially negated if the strain rate is increased [11]. This is ideal for hypersonic flight, since both temperature and strain will increase, and the loss of strength would be reduced.

Metal	Ta	Nb	Hf	Zr	Ti	Senkov
a (pm)	330.3	330.1	355.9	358.2	327.6	340.4
ρ (g/cm ³)	16.65	8.57	13.31	6.51	4.51	9.94
σ_c (MPa)	170	240	240	280	195	929
T_m (°C) [12][7]	3017	2477	2233	1855	1668	2241
Fracture Strain [8][13]						5 to >50%
Modulus (GPa) [14][8]						55 to 85
Vickers Hardness (HV) [8][9]						335 to 510

Table 1 – Table comparing composing element properties with Senkov alloy properties [19]

REFRACTORY HEAS (RHEAS)

Amongst the other alloys that were explored, there were two primary groups: RHEAs and the AlCoCrFeNi family of alloys. The RHEAs include some variations on the Senkov alloy, and add a few new elements to the mix: molybdenum, vanadium, and tungsten. Unfortunately, there is not as much information on these as there is on the Senkov alloy itself, given the number of potential combinations. A few alloys of particular interest will be discussed here, but other combinations hold potential as well.

One group of alloys not only includes the Senkov alloy, but adds a new element, creating HfMoNbTaTiZr. The variations are kept rather simple by observing dropping one of the 6 elements and observing the effects. This group has particularly good compressive strength as the environmental temperature increases, as can be seen in Figure 3. The best performing alloy, HfMoNbTaZr, keeps a compressive yield strength of 694 MPa at 1200°C. Other alloys with similar properties are HfMoNbTaTiZr and HfMoTaTiZr. They all have very high initial yield strengths and retain a large portion of their strength, even as the temperature increases. This is attributed to the disordered BCC solid solution by Tseng et al. Through a series of calculations, they were able to show that the high yield strength of these alloys is due to solid solution strengthening [20].

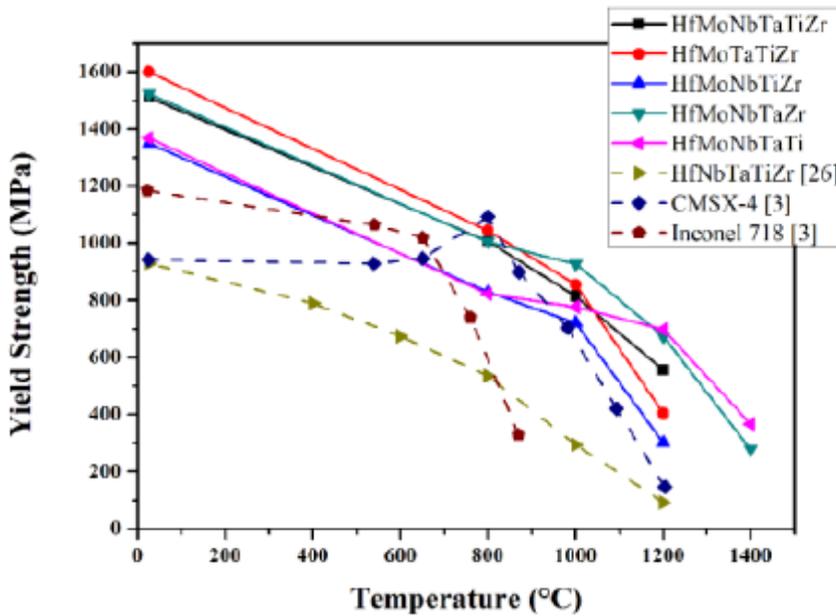


Figure 5 – Graph of the strength retention of HfMoNbTaTiZr alloys as temperature increases [20].

The other refractory HEAs of interest, NbMoTaW and VNbMoTaW, also have good strength retention as temperature increases, although they have a lower room temperature yield strength. Instead of steadily dropping in strength, there is an initial drop, followed by a plateau over a range of about 600 degrees before the material strength begins to significantly drop again. This strength retention is highly desirable for hypersonic application. These alloys also have a single-phase BCC structure, which remains the same even after 19 hours of annealing at 1400 °C. Solid solution strengthening and the boundaries between dendritic and inter-dendritic regions are both major factors in creating the high yield strength [21].

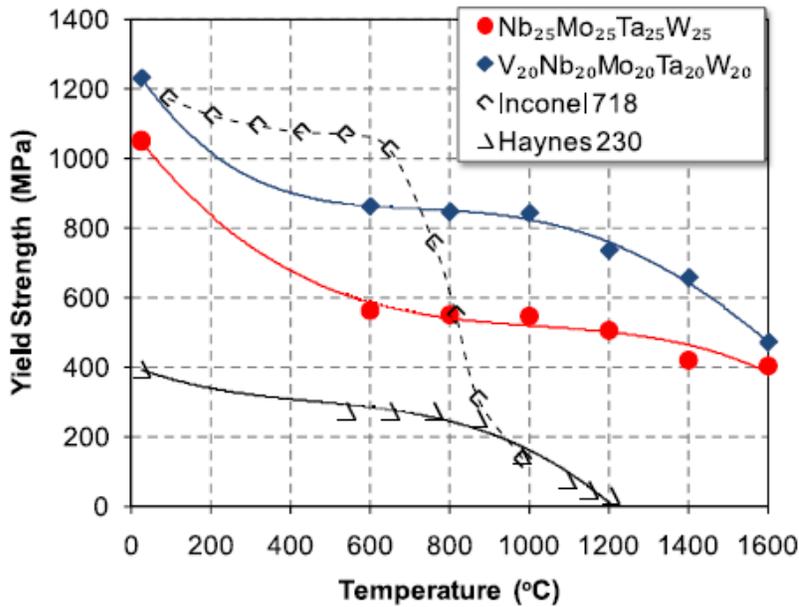


Figure 6 - Graph of strength retention of NbMoTaW and VNbMoTaW as temperature increases [21].

Since the VNbMoTaW group does not share many elements with the HfMoNbTaTiZr group, it isn't surprising that some of the material properties are dissimilar, as can be seen in Table 2. However, both utilize solid solution strengthening to keep a high yield strength, even as temperature increases. This is one of the major benefits first discussed when Yeh et al. proposed the idea of a high entropy alloy. The increased entropy results in a much more disordered, and thus, strengthened lattice structure.

One major downside of the refractory HEAs is the cost. Refractory alloys are useful because they don't break down easily at high temperatures, but that feature also raises the cost that goes into obtaining the refractory elements. Based on current market prices for pure elements, the cost by weight of each alloy has been approximated for the table below [25]. Additionally, the rule-of-mixtures has been used to approximate the density and melting temperature of these alloys (see appendix for sample calculations).

Metal	HfMoNbTaTiZr	HfMoTaTiZr	HfMoNbTaZr	NbMoTaW	VNbMoTaW
Approx. ρ (g/cm ³) [12]	9.97	10.25	11.06	13.69	12.17
σ_c at Room Temp. (MPa)	1512	1600	1524	1058	1249.976
σ_c at 1200°C (MPa)	556	404	694	506	735
Approx. Price (\$/kg) [25]	224.48	257.10	267.04	108.70	158.36
Approx. T_{melt} (°C) [12]	2312	2279	2441	2885	2690

Table 2 – Table comparing various attributes of Refractory HEAs.

AlCoCrFeNi FAMILY

While the Senkov alloy is composed mostly of more expensive refractory elements, this family of HEAs is formed primarily of more common elements, with an occasional sprinkling of a more expensive element. For this paper, three additional elements of interest will be explored: copper, titanium, and molybdenum. All can produce excellent strength properties with the right ratios. The titanium alloy has already been noted in other papers as having potential for aerospace applications.

Copper was tested in a composition of AlCoCrFeNiCu_x, with values ranging from $x = 0$ to $x = 2.5$. The alloy reached its highest strength at $x = 0.5$, with a maximum yield stress of 1,187 MPa [22]. AlCoCrFeNiTi_x was tested for $x = 0$ through $x = 1.5$. The same ratio ($x = 0.5$) worked best for this alloy as well, with a yield strength of 2,260 MPa [23]. AlCoCrFeNiMo_x only tested up to $x = 0.5$, likely due to the limitations of cost, with another maximum yield at $x = 0.5$, this time of 2,757 MPa, although due to a different kind of strengthening mechanism [24].

The causes of the increased yield strength for these alloys vary. In the copper alloy, a second FCC phase, rich in Cu, forms when $x = 0.5$, which is considered a major strengthening mechanism [22]. In the titanium alloy, dendrites form, resulting in a dendritic and inter-dendritic phases which strengthen the material [23]. Solid solution strengthening also attributed as a source of strengthening for both of

these alloys. The strengthening of molybdenum is attributed largely to the formation of a laminar structure as the amount of molybdenum increases [24].

The molybdenum alloy is of particular interest. It could potentially add some of desirable refractory properties, such as increased melting temperature, without increasing the cost by much. Unfortunately only one paper was found written about on this particular HEA, which was published in 2010 [24].

One problem with this family of elements is the lack of information on performance in higher than room temperatures. This suggests that the HEAs may melt at relatively low temperatures. The papers found so far have not supplied any melting temperature data. In the table below, the rule-of-mixture is used to approximate the density, cost, and melting temperature of the HEAs (see appendix). The approximated melting temperature is quite low, unfortunately. However, even if these HEAs are not quite as heat resistant as the Senkov HEAs, perhaps there is potential for using both alloys in hypersonics. The refractory HEAs could act as a protective outer coating while the AlCoCrFeNi alloys could act as internal structural support.

Metal	AlCoCrFeNiCu_{0.5}	AlCoCrFeNiTi_{0.5}	AlCoCrFeNiMo_{0.5}
Approx. ρ (g/cm ³) [12]	7.27	7.10	7.39
σ_c (MPa)	1,187	2,260	2,757
Plastic Strain	16.01%	23.3%	2.5%
Approx. Price (\$/kg) [25]	11.15	11.67	14.25
Approx. T_{melt} (°C)	1381	1434	1521

Table 3 – Table comparing various attributes of AlCoCrFeNi Alloys.

RESULTS AND DISCUSSION

HEAs are a relatively new group of alloys. While for many other alloys there are reams of data from repeated testing and analysis of their properties, so far there are only a handful of data points on most individual HEAs. The Senkov alloy is one of the most researched HEAs and while data has been collected on some of its attributes, there are still large gaps. Key properties are missing, including fracture toughness, creep properties, and shear strength. There is also a need for a significant amount of data on

the states of the alloy after it has gone through manufacturing processes such as cold rolling, especially in the through thickness and normal directions: such as strength (tensile, compressive, and shear) and stiffness.

Understanding the way texture, asymmetry, and anisotropy affect the properties of materials like the Senkov alloy is key, as people seek to model HEAs and other alloys. How these materials deform gives insight into what changes manufacturing processes introduce. For body centered cubic (BCC) lattice structure, which many known HEAs - including the Senkov Alloy - possess, the primary form of deformation is slip of dislocations (defects in the lattice). This occurs on the $\{101\}$ plane in the $\langle 111 \rangle$ direction, as seen on Figure 5 [17]. The alignment of the loading and slip direction along which a crystal deforms affects at least the rate at which they deform.

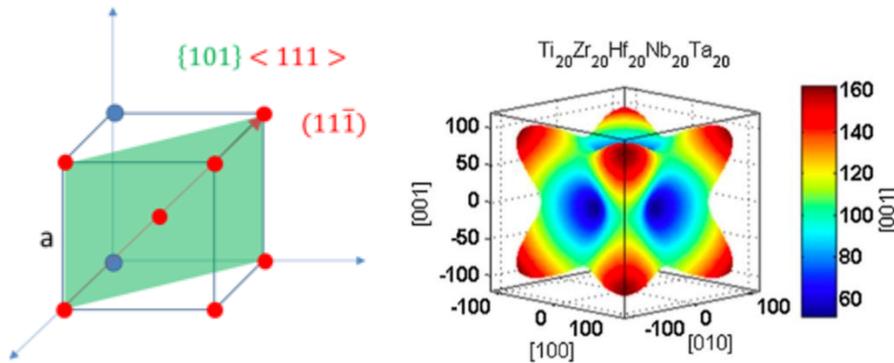


Figure 5 and 6 – A diagram showing the slip system of the Senkov alloy and an indicatrix of the Young's Modulus of the Senkov alloy depending on direction [15]

Furthermore, the orientation of the cubic crystalline structure in the grains rotate as the material deforms. This change from a random group of grain orientations to a more aligned one varies depending on the method of deformation. Compression results in a cross-section that has a concentration of corners and faces of the cube, while tension results in a concentration of long edges. As the orientations of the grains change, the strength and ductility of the material change as well, as is explained by Schmid's law. In fact, all tensor material properties change with the alignment of grains within a polycrystal. As an example, Figure 6 presents the indicatrix of the Young's modulus in a cubic unit cell. Note, the high stiffness at the corners and the low stiffness in the center of the faces.

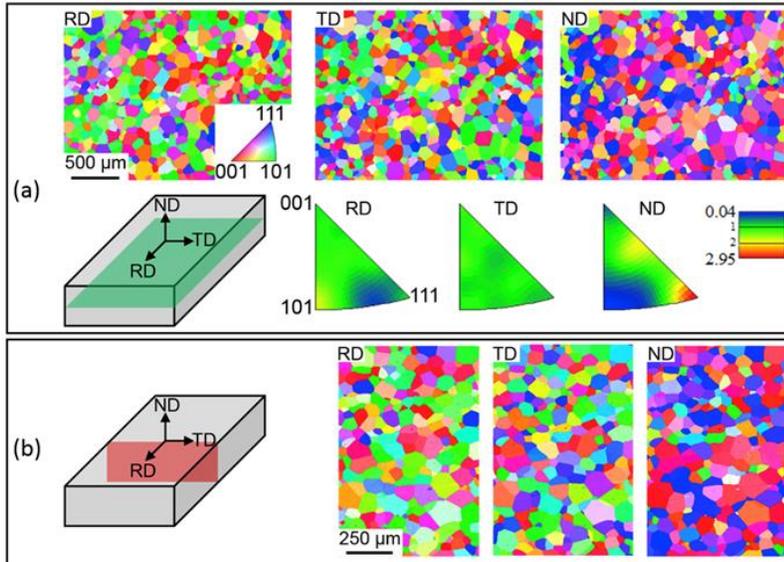


Figure 7 – Cross-sections of a cold rolled sample of the Senkov alloy, colored by orientation, and inverse pole figures to show orientation concentration [16]

Like with any other kind of deformation, the grains of the Senkov align during cold-rolling, which simultaneously places the material under compression in one direction and tension in another. This can be seen by looking at the cross-sections in the rolling direction and normal direction in Figure 7, which exhibit signs of being under tension and compression, respectively. This results in a sample that is strongly anisotropic; unfortunately, data has only been collected for the change of strength in the rolling direction, as can be seen in Table 4.

	Tensile Strength (MPa)	Compressive Strength (MPa)	Shear Strength (MPa)	Stiffness (GPa)	Creep Rate (s⁻¹)	Fracture Toughness (MPa√m)
Rolling Direction	1155	1351	--	--	--	--
Transverse Direction	--	--	--	--	--	--
Normal Direction	--	--	--	--	--	--
Completely Reversed	--	--	--	N/A	N/A	N/A

Table 4 – Table showing the gaps in data for cold rolled Senkov alloy.

Why is understanding anisotropy important? It helps inform manufacturing processes, ensures adequate factors of safety, and increases constitutive model accuracy. Understanding anisotropy improves our models which reduces costs all around.

Additionally, the costs of these new potential alloys must be considered as research continues. Regardless of the advantageous properties a material has, the cost can be a major limiting factor. AlCoCrFeNiMo_{0.5}, for example, has great properties at a lower cost and may have a great deal of potential with a little bit of research and adjustment. The following prices were calculated using a weighted average of current market prices of the pure elements [25].

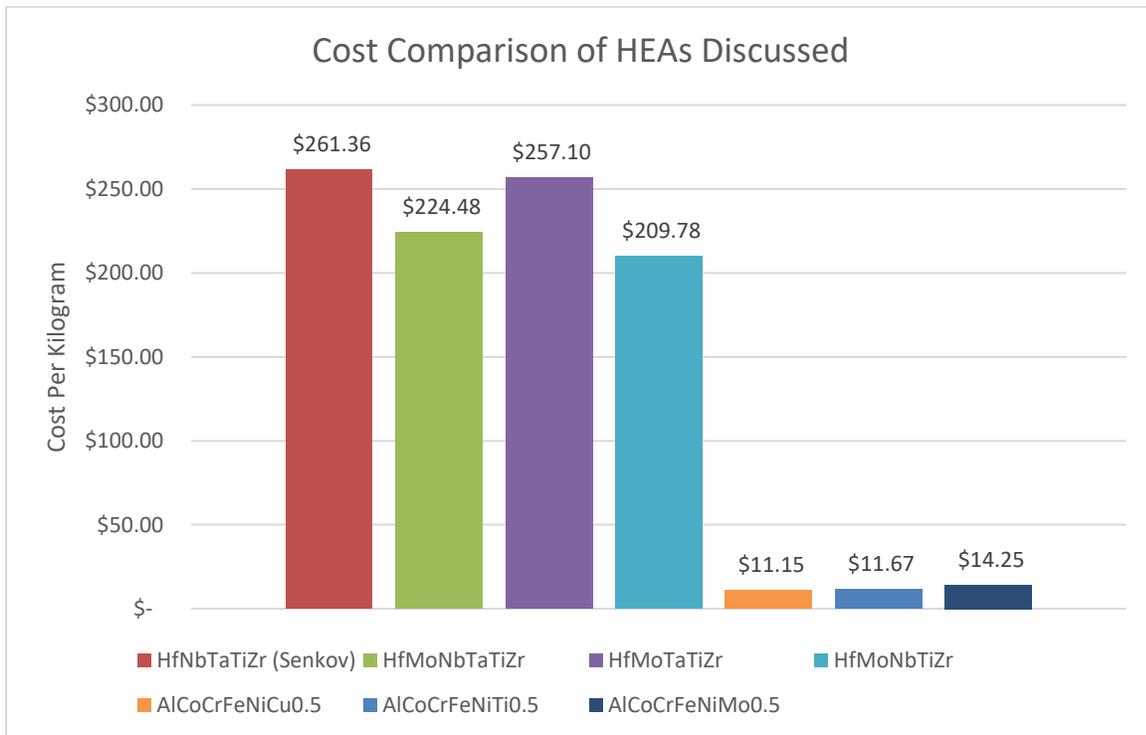


Figure 8 – Graph showing the prices of the HEAs discussed in this paper [25].

The goal of this project was to select HEAs that would be suitable for hypersonics and model them, not only in their as-cast state, but after the manufacturing process. The data on the Senkov alloy is not sufficient at this time to model its behavior using advanced material models for anisotropy under development within the Air Force Research Lab (AFRL). Additional testing to include characterization of the shear strength is required to apply the latest AFRL constitutive models.

CONCLUSION

High entropy alloys are an area of great potential that requires further exploration. By researching these HEAs further, not only will understanding of HEAs expand, but so will understanding of asymmetric alloys, and the effects of manufacturing processes on the properties of a material. This literature review has shown there are large gaps of information that need to be filled and the Senkov alloy is a great place to start, as it has both research applications and real-world applications as well. Many alloys explored in this paper could be very useful in hypersonic designs if explored properly.

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APPENDIX

Sample Calculations:

Rule-of-Mixtures: The rule-of-mixtures is essentially a weighted average.

$$\rho_{AlCoCrFeNiCu_{0.5}} = \frac{\rho_{Al} + \rho_{Co} + \rho_{Cr} + \rho_{Fe} + \rho_{Ni} + 0.5\rho_{Cu}}{5.5} = \frac{2.7 + 8.9 + 7.14 + 7.87 + 8.91 + (0.5 * 8.96)}{5.5}$$
$$= 7.27$$