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BIG ORANGE BIG IDEAS

Experimental and Computational Investigation of High-Entropy Alloys (HEAs) for Elevated-Temperature Applications

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Acknowledgements

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(6) National Energy Technology Laboratory (NETL) for sponsoring this project

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Outline of Presentation

(1) Potential Significance
(2) Background and Unique Behavior of HEAs
(3) Project Objectives
(4) Proposed Work
(5) Preliminary Results
(6) Conclusions
(7) Future Work
(8) Published Papers and Presentations

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Potential Significance

- Develop a suitable HEA for steam and gas turbine components operating at temperatures higher than 760° C.
- Increase the thermal efficiency of steam turbines and reduce the costs of fuel and emissions.
- Apply a computer-aided approach for designing new types of alloys applicable for the design and development of other high-temperature materials.

http://middleeast.geblogs.com/en/lang-en/ge-steam-turbines-to-help-saudi-arabia-boost-power-output-and-efficiency-2/

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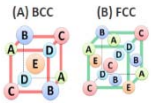
Background and Unique Behavior

- Most alloy systems are based on a single principal element to form the matrix.
- Multiple principal elements were expected to yield many intermetallic compounds creating a complex microstructure.
- However, simple face-centered cubic (FCC) and body-centered cubic (BCC) structures are possible and thermodynamically favorable.



[1] Yeh JW, Chen SK, Lin SJ, Gan JY, Chin TS, Shun TT, Tsau CH, Chang SY. *Advanced Engineering Materials*, 2004; 6: 299-303.
[2] Cantor B, Chang ITH, Knight P, Vincent AJB. *Mater Sci Eng A Struct Mater*, 2004; 375: 213-218.

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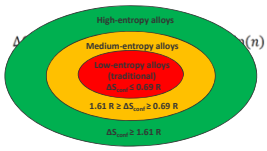
Background and Unique Behavior (Cont'd)



High entropy of mixing and sluggish diffusion yield simple face-centered cubic (FCC) and body-centered cubic (BCC) solid solutions with dispersed nano-phases.

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Background and Unique Behavior (Cont'd)

- HEAs are composed of five or more elements in equimolar ratios.
- A large number of HEAs are potentially possible for various applications. For example, 13 elements could generate 7099 HEAs.

$$C_5^{13} + C_6^{13} + C_7^{13} + C_8^{13} + C_9^{13} + C_{10}^{13} + C_{11}^{13} + C_{12}^{13} + C_{13}^{13} = 7099$$

where C_n^m is the number of combinations of m elements taken n times

- Show great high-temperature strengths, corrosion, wear, and fatigue resistance.

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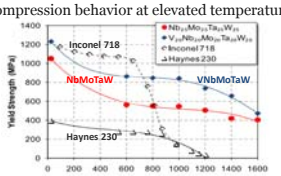
Background and Unique Behavior (Cont'd)

Compression behavior at elevated temperatures



The yield stress of both alloys dropped by 30 - 40% between room temperature and 600 °C, but was relatively insensitive to temperature above 600 °C, comparing favorably with conventional superalloys.

Senkov, O.N., G.B. Wilks, J.M. Scott, and D.B. Miracle. Intermetallics, 2011. 19(5): pp. 698-706.
Senkov, O.N., G.B. Wilks, D.B. Miracle, C.P. Chuang, P.K. Liaw, Intermetallics, 2010, 18: pp. 1758-1765.

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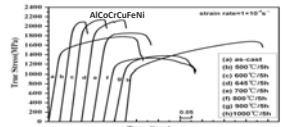
Background and Unique Behavior (Cont'd)

Compression behavior at room temperature
BCC + FCC matrix, heat-treatment effect



Compressive true stress–strain curves of AlCoCrCuFeNi after heat-treatment for 5 h at different temperatures.
The mechanical properties of this alloy have a strong correlation with the aging temperature.

Wen, L.H., H.C. Kou, J.S. Li, H. Chang, X.Y. Xue, and L. Zhou. Intermetallics, 2009. 17(4): pp. 266-269.

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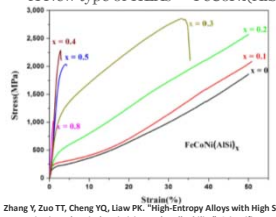
Background and Unique Behavior (Cont'd)
A New type of HEAs ---FeCoNi(AlSi)x



Zhang Y, Zuo TT, Cheng YQ, Liaw PK. "High-Entropy Alloys with High Saturation Magnetization, Electrical Resistivity, and Malleability". Scientific Reports, 2013; 3.

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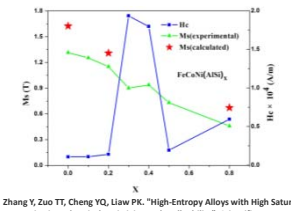
Background and Unique Behavior (Cont'd)
A New type of HEAs ---FeCoNi(AlSi)x



Zhang Y, Zuo TT, Cheng YQ, Liaw PK. "High-Entropy Alloys with High Saturation Magnetization, Electrical Resistivity, and Malleability". Scientific Reports, 2013; 3.

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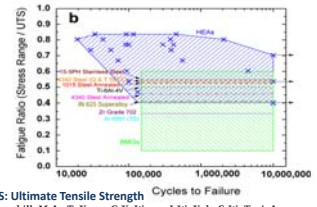
Background and Unique Behavior (Cont'd)
Good Fatigue Resistance of Al0.5CoCrCuFeNi



UTS: Ultimate Tensile Strength

Hemphill, M.A., T. Yuan, G.Y. Wang, J.W. Yeh, C.W. Tsai, A. Chuang, and P.K. Liaw. Acta Materialia, 2012, 60: pp. 5723-5734

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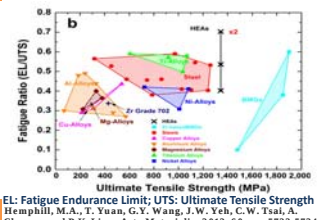
Background and Unique Behavior (Cont'd)
Good Fatigue Resistance of Al0.5CoCrCuFeNi



EL: Fatigue Endurance Limit; UTS: Ultimate Tensile Strength

Hemphill, M.A., T. Yuan, G.Y. Wang, J.W. Yeh, C.W. Tsai, A. Chuang, and P.K. Liaw. Acta Materialia, 2012, 60: pp. 5723-5734

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Background and Unique Behavior (Cont'd)
View from Thermodynamics

- $\Delta G_{mix} = \Delta H_{mix} - T\Delta S_{mix}$
  - More stable with smaller ΔG_{mix}
  - Higher ΔS_{mix} favors smaller ΔG_{mix}
  - ΔH_{mix} cannot be ignored
- It is stability competition among all the phases in the system that determines the final microstructure
  - Which phases are stable?
  - How many of each phase are there ?

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Project Objectives

- Perform fundamental studies on the $Al_xCrCuFeMnNi$ high-entropy alloy (HEA) system for use in boilers, and steam and gas turbines at temperatures above 760°C and a stress of 35 MPa.
- Develop the thermodynamic database and identify the potential HEAs within the Al-Cr-Cu-Fe-Mn-Ni system for further experimental investigation.
- Optimize microstructures to identify the best combined strength, ductility, and creep resistance.

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Proposed Work

- ❖Fabricate HEAs, for use in mechanical tests, sufficiently free of casting and aging defects suitable for future mechanical tests
- ❖Provide thermodynamic descriptions of the $Al_xCrCuFeMnNi$ HEA system of favorable compositions
- ❖Conduct ex-situ mechanical and creep experiments of selected alloy systems at elevated temperatures to characterize material properties
- ❖Perform microstructural characterization using SEM, TEM, and Local-Electrode Atom-Probe (LEAP) to analyze microstructures affecting mechanical and creep behavior
- ❖Carry out in-situ creep and mechanical-behavior characterizations using neutron/synchrotron diffraction

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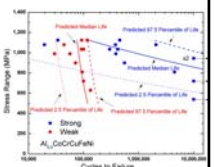
Preliminary Results - Alloy Fabrication

- Fabricate multiple alloy compositions belonging to the $Al_xCrCuFeMnNi$ system varying the Al content from 0 to 2.
- Alloys sensitive to impurities and defects introduced during casting and processing
- Reduce defects during fabrication



Hemphill, M.A., T. Yuan, G.Y. Wang, J.W. Yeh, C.W. Tsai, A. Chuang, and P.K. Liaw. Acta Materialia, 2012, 60: pp. 5723-5734

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Weibull Mixture Predictive Model

Hemphill, M.A., T. Yuan, G.Y. Wang, J.W. Yeh, C.W. Tsai, A. Chuang, and P.K. Liaw. Acta Materialia, 2012, 60: pp. 5723-5734

- Two groups appear, weak and strong groups with the weak group tending to fail more quickly than the strong group.
- A Weibull mixture model is applied to the probability density function (PDF) and cumulative distribution function (CDF), respectively

$$f(N|p,\alpha_w,\beta_w,\alpha_s,\beta_s) = p\frac{\beta_w}{\alpha_w}\left(\frac{N}{\alpha_w}\right)^{\beta_w-1}\exp\left[-\left(\frac{N}{\alpha_w}\right)^{\beta_w}\right] + (1-p)\frac{\beta_s}{\alpha_s}\left(\frac{N}{\alpha_s}\right)^{\beta_s-1}\exp\left[-\left(\frac{N}{\alpha_s}\right)^{\beta_s}\right]$$

$$F(N|p,\alpha_w,\beta_w,\alpha_s,\beta_s) = p\left\{1-\exp\left[-\left(\frac{N}{\alpha_w}\right)^{\beta_w}\right]\right\} + (1-p)\left\{1-\exp\left[-\left(\frac{N}{\alpha_s}\right)^{\beta_s}\right]\right\}$$

- Seven unknown parameters where p is the fraction belonging to the weak group and the subscripts w and s indicate the strong or weak group, respectively:

$$c_w, d_w, \beta_w, c_s, d_s, \beta_s, p$$

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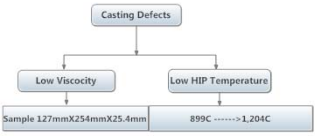
Preliminary Results - Alloy Fabrication (Cont'd)

- Al0.8CrCuFeMnNi
- Φ 25.4 mm (1 inch) × 762 mm (30 inch)
- Vacuum Cast → Hot Isostatic Pressing 899 °C, 103MPa, 2h.
- Differential Thermal Analysis (DTA) 100 °C---1,600 °C
  - No thermal transitions: 100 to around 980 °C.
  - Three transitions occurred at 980 °C, 1,173 °C, and 1,358 °C.
  - Discernible liquidus is not observed up to 1,600 °C.

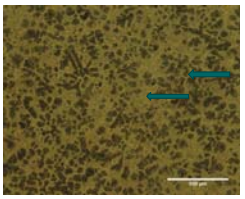
Preliminary Results - Alloy Fabrication (Cont'd)

- Cast in vacuum



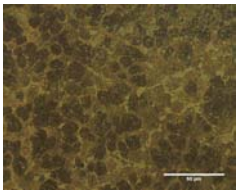
- Arc-Melting (ϕ10 mm × 50 mm)
- Vacuum Induction Melting (VIM) + Hot Working/Cold Work and Full Annealing Process

Preliminary Results – Microstructures (Cont'd)



OM images of Al0.8CrCuFeMnNi

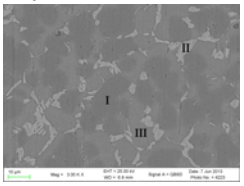
Preliminary Results – Microstructures (Cont'd)



OM images of Al0.8CrCuFeMnNi

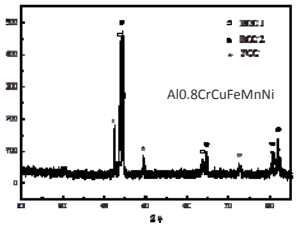
white-phase layers appear along the grain boundary

Preliminary Results - Microstructures - SEM



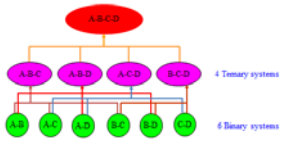
Atomic %	Al	Cr	Mn	Fe	Ni	Cu
I	8.12	31.88	15.47	29.04	9.05	6.44
II	6.97	1.65	19.12	3.11	9.9	59.25
III	20.97	4.86	19.31	6.59	28.13	20.14

Preliminary Results - XRD Patterns



Preliminary Results - Thermodynamic Modeling

Construct A Database



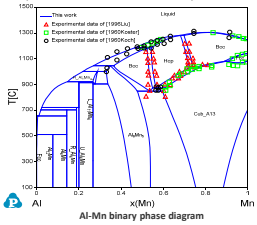
Preliminary Results - Thermodynamic Modeling

Constituent binary and ternary systems of the Al-Cr-Cu-Fe-Mn-Ni system

Binary Systems				
Al-Cr	Al-Cu	Al-Fe	Al-Mn	Al-Ni
Cr-Cu	Cr-Fe	Cr-Mn	Cr-Ni	Cu-Fe
Cu-Mn	Cu-Ni	Fe-Mn	Fe-Ni	Mn-Ni
Ternary Systems				
Al-Cr-Cu	Al-Cr-Fe	Al-Cr-Mn	Al-Cr-Ni	Al-Cu-Fe
Al-Cu-Mn	Al-Cu-Ni	Al-Fe-Mn	Al-Fe-Ni	Al-Mn-Ni
Cr-Cu-Fe	Cr-Cu-Mn	Cr-Cu-Ni	Cr-Fe-Mn	Cr-Fe-Ni
Cr-Mn-Ni	Cu-Fe-Mn	Cu-Fe-Ni	Cu-Mn-Ni	Fe-Mn-Ni

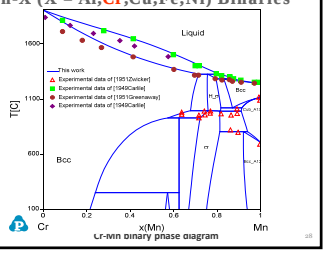
- There are **15** constituent binary systems.
- There are **20** constituent ternary systems.

Preliminary Results - Mn-X (X = Al,Cr,Cu,Fe,Ni) Binaries



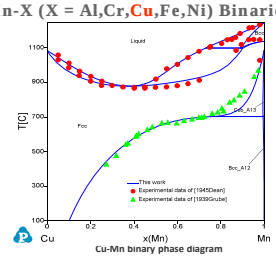
Al-Mn binary phase diagram

Preliminary Results
Mn-X (X = Al,Cr,Cu,Fe,Ni) Binaries



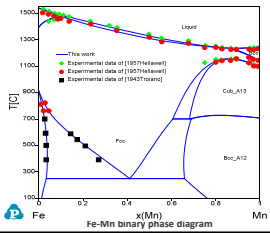
Cr-Mn binary phase diagram

Preliminary Results
Mn-X (X = Al,Cr,Cu,Fe,Ni) Binaries



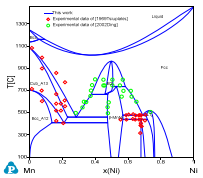
Cu-Mn binary phase diagram

Preliminary Results -
Mn-X (X = Al,Cr,Cu,Fe,Ni) Binaries



Fe-Mn binary phase diagram

Preliminary Results -
Mn-X (X = Al,Cr,Cu,Fe,Ni) Binaries

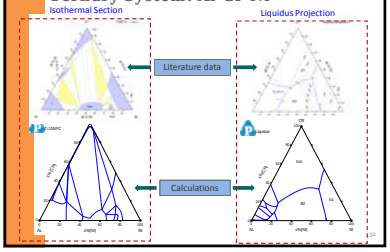


Mn-Ni binary phase diagram

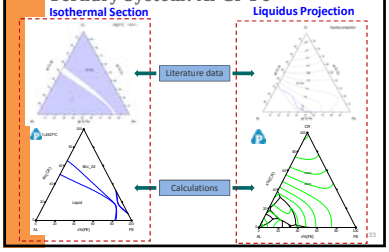
All 15 constituent binary systems have been thermodynamically modeled.

Experimental validation of these Constituent binary systems are done!

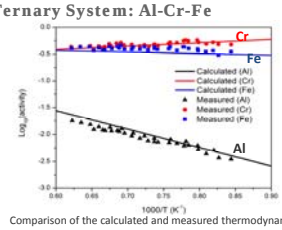
Preliminary Results
Ternary System: Al-Cr-Ni



Preliminary Results
Ternary System: Al-Cr-Fe



Preliminary Results
Ternary System: Al-Cr-Fe



Comparison of the calculated and measured thermodynamic activities of Al, Cr, and Fe in the alloy with target composition Fe-19.9Al-30.2Cr (at.%)

Preliminary Results- Summary of Thermodynamic Modeling

Binary Systems

Al-Cr	Al-Cu	Al-Fe	Al-Mn	Al-Ni
Cr-Cu	Cr-Fe	Cr-Mn	Cr-Ni	Cu-Fe
Cu-Mn	Cu-Ni	Fe-Mn	Fe-Ni	Mn-Ni

Ternary Systems

Al-Cr-Cu	Al-Cr-Fe	Al-Cr-Mn	Al-Cr-Ni	Al-Cu-Fe
Al-Cu-Mn	Al-Cu-Ni	Al-Fe-Mn	Al-Fe-Ni	Al-Mn-Ni
Cr-Cu-Fe	Cr-Cu-Mn	Cr-Cu-Ni	Cr-Fe-Mn	Cr-Fe-Ni
Cr-Mn-Ni	Cu-Fe-Mn	Cu-Fe-Ni	Cu-Mn-Ni	Fe-Mn-Ni

Thermodynamically modeled

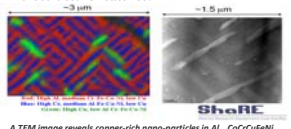
Need to do next

Future Work
Mechanical Behavior

- Fabricate alloys without casting defects
- Characterize the mechanical properties of the refined alloys
  - ❑ Conduct nanoindentation experiments
  - ❑ Perform compression tests
  - ❑ Carry out creep tests ex-situ as well as in-situ neutron diffraction
- Study the performance of HEAs after aging

Future Work (Cont'd)

- Conventional and high-resolution transmission electron microscope (TEM) to determine the microstructure features.



A TEM image reveals copper-rich nano-particles in $Al_{1.3}CoCrCuFeNi$.

- Shape and average size of the main phases and deformation mechanisms, such as the interaction between dislocations and phase boundaries during mechanical-property tests

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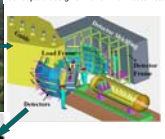
Future Work

- In-situ neutron-diffraction experiments

Spallation Neutron Source (SNS), Oak Ridge National Laboratory (ORNL)

Conceptual design of VULCAN diffractometer



The multifunctional load-frame developed for VULCAN diffractometer

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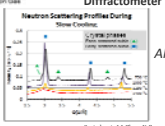
Future Work

- In-situ neutron-diffraction experiments

The Nanoscale-Ordered Materials Diffractometer (NOMAD)



$Al_{1.3}CoCrCuFeNi$

Santodonato LJ, Zhang Y, Feygenson M, NomFleed J, Tang Z, Weber R, Gao M, and Liaw PK, unpublished results, 2011

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Future Work

Thermodynamic Modeling

- Continue to work on the thermodynamic modeling of the other constituent ternary systems.
- Provide experimental validation of the developed thermodynamic database based on the experimental data from both literature and our team members.
- Provide thermodynamic predictions to identify potential HEAs within the Al-Cr-Cu-Fe-Mn-Ni system.

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Published Papers and Presentations

Papers:

[1] Zhang Y, Zuo TT, Cheng YQ, Liaw PK. "High-Entropy Alloys with High Saturation Magnetization, Electrical Resistivity, and Malleability". *Scientific Reports*, 2013; 3.

[2] Guo W, Dmowski W, Noh JY, Rack P, Liaw PK, Egami T. "Local Atomic Structure of a High-Entropy Alloy: An X-Ray and Neutron Scattering Study". *Metallurgical and Materials Transactions a-Physical Metallurgy and Materials Science*, 2013; 44A: 1994.

[3] Hemphill MA, Yuan T, Wang G, Yeh J, Tsai C, Chuang A, Liaw PK. "Fatigue Behavior of Al0.5CoCrCuFeNi High Entropy Alloys". *Acta Materialia*, 2012.

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Published papers and presentations

[4] Yang X, Zhang Y, Liaw PK. "Microstructure and Compressive Properties of NbTiVTaAlx High Entropy Alloys". *Procedia Engineering*, 2012; 36: 292.

[5] Zhang Y, Yang X, Liaw PK. "Alloy Design and Properties Optimization of High-Entropy Alloys". *JOM*, 2012; 64: 830.

[6] Zhang Y, Zuo T, Liao W, Liaw PK. "Processing and Properties of High-Entropy Alloys and Micro-and Nano-Wires". *ECS Transactions*, 2012; 41: 49.

[7] Zhang C, Zhang F, Chen SL, Cao WS. "Computational Thermodynamics Aided High-Entropy Alloy Design". JOM, 2012; 64(7): 839-845.

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Published Papers and Presentations

Presentation:

❖ 2013 TMS Meeting , San Antonio, TX, USA, March 3-9, 2013

- Automatic Fabrication of High - Entropy Alloys and Their Properties, Y. Yokoyama, X. Xie, J. Antonaglia, M. Hemphill, T. Zhi, T. Yuan, G. Wang, C. Tsai, J. Yeh, A. Chuang, K. Dahmen, P. K. Liaw (invited)
- Non-Equilibrium and Equilibrium Phases in AlCoCrFeNi High-Entropy Alloys, Z. Tang, O. Senkov, C. Parish, L. Santodonato, D. Miracle, G. Wang, C. Zhang, F. Zhang, P. K. Liaw
- Ordering Behavior in the Al(x)CoCrCuFeNi High - Entropy Alloys, L. Santodonato, Y. Zhang, M. Gao, C. Parish, M. Feygenson, Z. Tang, J. Neuefeind, R. Weber, P. K. Liaw
- Computational Modeling of High-Entropy Alloys, M. Gao, D. Tafen, J. Hawk, Y. Wang, M. Widom, L. Santodonato, P. K. Liaw(invited)
- Statistical Fatigue - Life Modeling for High - Entropy Alloys, T. Yuan, M. Hemphill, Z. Tang, G. Wang, A. Chuang, C. Tsai, J. Yeh, P. K. Liaw (invited).

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- A Combinatorial Approach to the Investigation of Metal Systems that Form Both High Entropy Alloys and Bulk Metallic Glasses, B. Welk, P. K. Liaw, M. Gibson, H. Fraser
- Minor Phase and Defect Effects on Fatigue Behavior of Wrought Al0.5CoCrCuFeNi High - Entropy Alloys, Z. Tang, M. Hemphill, T. Yuan, G. Wang, J. Yeh, C. Tsai, P. K. Liaw
- Phase Separation and Intermetallic Formation in "High - Entropy" Alloys, C. Parish, M. Miller, L. Santodonato, Z. Tang, P. K. Liaw.
- Computational Thermodynamics Aided High - Entropy Alloy Design, C. Zhang, F. Zhang, S. Chen, W. Cao, J. Zhu, Z. Tang, P. K. Liaw

❖ The 9th International Conference on Bulk Metallic Glass (BMG-IX) 2012, Xiamen, China

- Zhang C, Zhang F, Chen SL, Cao WS, Tang Z and Liaw PK. "Computational Thermodynamics Aided High-Entropy Alloy Design".

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Conclusions

- ❖ We have fabricated bulk Al0.8CrCuFeMnNi samples, even though there are some cast defects existing.
- ❖ The Al0.8CrCuFeMnNi alloy has typical dendritic grains. White-phase layers appear along the grain boundary.
- ❖ The Al0.8CrCuFeMnNi alloy has three phases, disordered BCC1, BCC2, and FCC phases.
- ❖ All the constituent binary systems have been thermodynamically modeled.
- ❖ Al-Cr-Fe and Al-Cr-Ni ternary phase diagrams are in good agreement with the literature data.
- ❖ Future work has been identified.

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BIG ORANGE BIG IDEAS

Thank You!

Background and Unique Behavior

Selection Criteria in Literature

- Higher entropy of mixing[1] ($\Delta S_{mix} > 1.61R$)
- Smaller enthalpy of mixing[1] ($-15 < H_{mix} < 5kJ/mol$)
- Less atomic size difference ($1 \leq \delta \leq 6$)[1]

$$\delta = \sqrt{\sum_{i=1}^{n} c_i\left(1 - \frac{r_i}{\bar{r}}\right)^2}, \bar{r} = \sum_{i=1}^{n} c_i r_i$$

- Valence electron concentration[2]

$(bcc)\ 6.87 \leq VEC = \sum_{i=1}^{n} c_i (VEC)_i < 8\ (fcc)$

**Semi-empirical, system dependent, limited predictability*

[1] Y. Zhang and Y.J. Zhou, Materials Science Forum 2007, 561-565: 1337
[2] S. Guo, C. Ng, J. Lu and C.T. Liu, JAP 2011, 109: 103505

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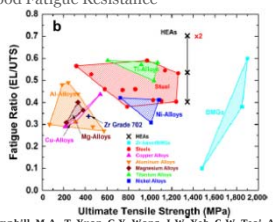
Background and Unique Behavior

Good Fatigue Resistance



Hemphill, M.A., T. Yuan, G.Y. Wang, J.W. Yeh, C.W. Tsai, A. Chuang, and P.K. Liaw. Acta Materialia, 2012, 60: pp. 5723-5734

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Background and Unique Behavior

Thermodynamic description

- When we mix n elements together to form a disordered phase, the entropy of mixing is:

$$\Delta S_{mix} = -R\sum_{i=1}^{n} x_i \ln x_i$$

where x is the mole fraction of element i.

- *When equimolar of every element is used, the entropy of mixing: ΔS_{mix} reaches a maximum*

Design of HEA: Mix equimolar multi-components to form disordered phase

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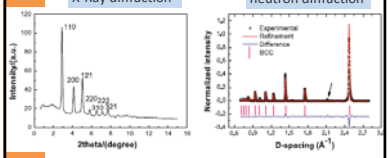
Preliminary results - Local Atomic Structure of ZrNbHf

X-Ray diffraction | neutron diffraction



Thin film | Bulk sample

Guo W, Dmowski W, Noh JY, Rack P, Liaw PK, Egami T. Metallurgical and Materials Transactions a-Physical Metallurgy and Materials Science, 2013; 44A: 1994

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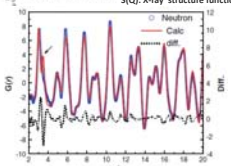
Preliminary results - Local Atomic Structure ZrNbHf

The pair distribution function (PDF)

$$G(r) = \frac{2}{\pi}\int_0^\infty Q[S(Q) - 1]\sin(Qr)dQ$$

Q: scattering vecter, r: interatomic distance
S(Q): X-ray structure function

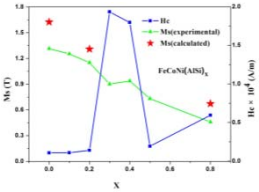


Guo W, Dmowski W, Noh JY, Rack P, Liaw PK, Egami T. Metallurgical and Materials Transactions a-Physical Metallurgy and Materials Science, 2013; 44A: 1994

51

Background and Unique Behavior

A New type of HEAs ---FeCoNi(AlSi)x

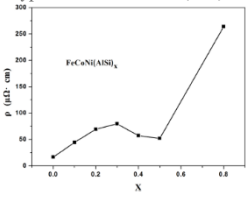


Zhang Y, Zuo TT, Cheng YQ, Liaw PK. "High-Entropy Alloys with High Saturation Magnetization, Electrical Resistivity, and Malleability". Scientific Reports, 2013; 3.

52

Background and Unique Behavior

A New type of HEAs ---FeCoNi(AlSi)x



Zhang Y, Zuo TT, Cheng YQ, Liaw PK. "High-Entropy Alloys with High Saturation Magnetization, Electrical Resistivity, and Malleability". Scientific Reports, 2013; 3.

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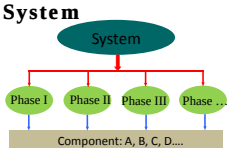
Approach

Alloy System



$$G^P = \sum X_i \cdot G_i^P + \sum w_{i-j}^P \cdot G_{i-j}^P + \sum w_{i-j-k}^P \cdot G_{i-j-k}^P$$

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Approach

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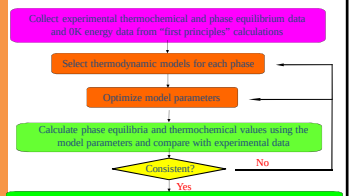
Development of Thermodynamic database



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Background and Unique Behavior (Cont'd)

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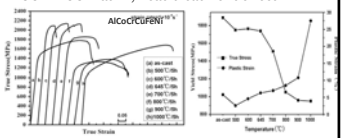
Compression behavior at room temperature
BCC + FCC matrix, heat-treatment effect



Compressive true stress–strain curves of AlCoCrCuFeNi after heat-treatment for 5 h at different temperatures.
The mechanical properties of this alloy have a strong correlation with the aging temperature.

Wen, L.H., H.C. Kou, J.S. Li, H. Chang, X.Y. Xue, and L. Zhou. Intermetallics, 2009. 17(4): pp. 266-269.