



## Data Article

# Characterization data of an $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$ multi-principal element alloy continuous composition spread library



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## ABSTRACT

The data provided in this article is related to the research article entitled "Phase stabilization and oxidation of a continuous composition spread multi-principal element  $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$  alloy" [1]. This data article describes the high-throughput synthesis and characterization processes of an  $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$  alloy system. Continuous composition spread (CCS) thin-film libraries were synthesized by co-depositing an AlFeNiTiVZr metal alloy target and Cr target via magnetron sputtering. Post-processing was performed on the sample libraries with a vacuum anneal at 873 K and an air anneal at 873 K. Compositional data was determined via WDS in order to verify parameters provided by an in-house sputter model. Crystallographic data was captured via synchrotron diffraction and diffractograms were compared as a function of the change in Cr concentration. These measurements were taken in order to observe phase behavior after

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oxidation throughout the composition library. Furthermore, vibrational spectrographic data is provided of the oxidized library to show surface speciation along the composition gradient of the alloy system. The structural and oxidative behavior of the  $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$  alloy can be analysed using the data provided in this article. Additionally, this characterization dataset can be utilized in machine learning algorithms for determining important features and parameters for future hypothesis generation of functional multi-principal element alloys (MPEAs).

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## Specifications Table

Subject	Materials Science
Specific subject area	Multi-Principal Element Alloys and High-Throughput Experimentation
Type of data	Table, Figures
How data were acquired	WDS: Electron Probe Microanalyzer (EPMA) JXA 8900R Microprobe XRD: Stanford Synchrotron Radiation Lightsource 1–5 Beamline, MarCCD Detector Raman: Horiba XploraPLUS Raman Microscope
Data format	Raw, Analyzed
Parameters for data collection	Seven continuous composition spread libraries of $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$ were synthesized via magnetron sputtering co-deposition on a 7.62 cm diameter Si wafer. Six samples were annealed in vacuum at 873 K for 6 h. Afterward five of the sample libraries were individually annealed in air at 873 K for 1, 3, 6, 12, and 18 h. All seven libraries were characterized after post-processing.
Description of data collection	WDS measurements were taken using an acceleration voltage of 15 kV and standard references for each of the constituent elements. XRD patterns were obtained via synchrotron diffraction with an incidence angle of $6^\circ$ and an x-ray wavelength of 0.9762 Å (12.7 keV). Raman spectra were measured using a 25 mW, 473 nm solid state laser and a 2400 gr/mm grating. The laser was focused using a 20x microscope objective (NA = 0.40, WD = 12 mm). All characterization data was collected using rapid serial measurements on x-y transitional stages.
Data source location	Institution: University of South Carolina City/Town/Region: Columbia, South Carolina Country: United States of America
Data accessibility	Data are within the article
Related research article	B. Ruiz-Yi, T. Williams, J.K. Bunn, F. Ren, N. Al Hasan, I. Takeuchi, J. Hattrick-Simpers, A. Mehta, Phase stabilization and oxidation of a continuous composition spread multi-principal element $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$ alloy, <i>J. Alloys Compd. In Press.</i> [1]

## Value of the Data

- The experimental data presented here provides phase behavioural observations of the  $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$  multi-principal element alloy (MPEA) system as a function of composition using high-throughput x-ray diffraction and Raman spectral measurements, which can be used as a basis for additional high-throughput studies on other MPEA materials with engineering applications.

- This data can provide an example of the results high-throughput experimentation provides when utilized in the study of a complex composition space in a functional material.
- This dataset can be used for future applied studies on the  $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$  system after compositional regions of interest are discovered, and it can be utilized as a dataset for machine learning algorithms to determine feature importance in MPEA discovery and functionality.

## 1. Data Description

Multi-principal element alloys are a class of materials that contains multiple constituent elements and no individual element can be considered the base of the alloy system [2]. The data presented in this section summarizes the characterization of as-deposited and post-processed continuous composition spread (CCS) thin-film libraries of the  $(\text{AlFeNiTiVZr})_{1-x}\text{Cr}_x$  alloy system. This system was selected based off the desire to introduce a chromium dopant into a previously reported alloy system [3]. This data is comprised of elemental composition information, crystallographic diffractograms, and vibrational spectra. The compositional data that was determined using wavelength-dispersive spectroscopy (WDS) across a single axis of the library and can be seen in Table 1. The chromium atomic percentage in the sample libraries was fit to a quadratic model as a function of sample position, which can be seen in Fig. 1. Structural data was obtained via high-throughput synchrotron diffraction on the as-deposited library, solely vacuum annealed library, and all libraries that were annealed in air. Waterfall plots of the diffraction patterns were constructed to observe the structures as a function of composition and can be seen in Fig. 2a-2f. The raw unprocessed diffraction data has been supplied as a supplementary spreadsheet for ease of readability. Raman spectra of the air annealed CCS libraries were measured as well. Raman heatmaps and corresponding characteristic spectra of the 1 h, 6 h, and 18 h air anneal can be

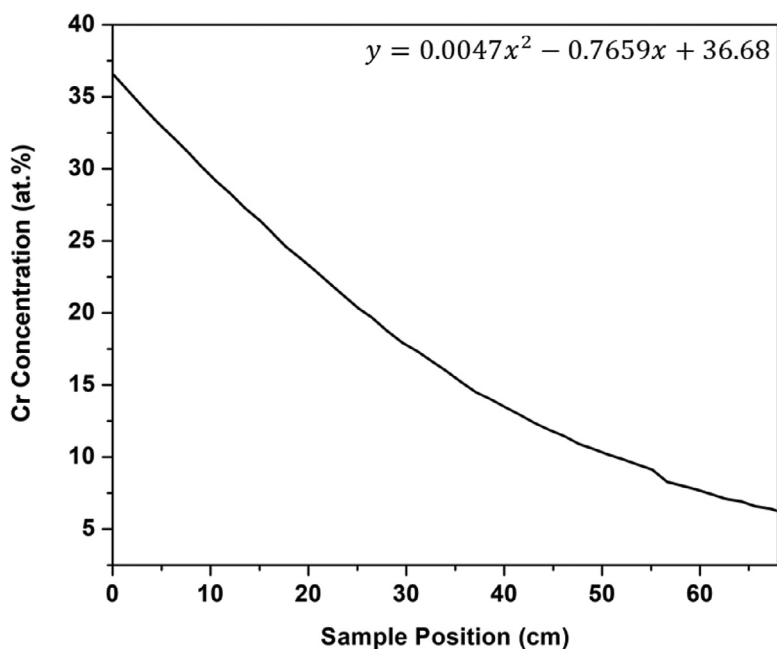


Fig. 1. Quadratic model of the Cr concentration vs sample position. The  $R^2$  of the equation is 0.998.

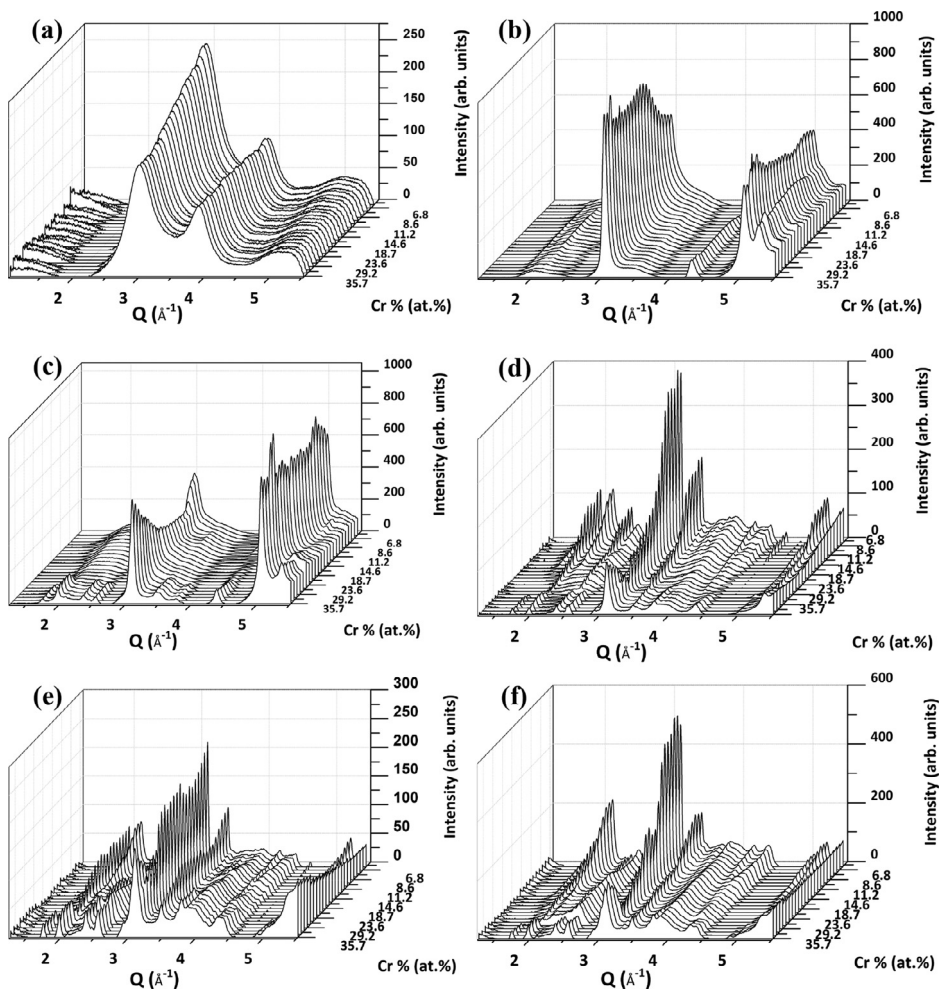
**Table 1**  
Wavelength-dispersive spectrographic measurements of the (AlFeNiTiVZr)<sub>1-x</sub>Cr<sub>x</sub> thin film library.

Average Composition							Position
Al at.%	Fe at.%	Ni at.%	Ti at.%	V at.%	Zr at.%	Cr at.%	y mm
23.6	13.3	10.5	17.1	16.2	13.2	6.1	68.7
23.4	13.5	10.7	17.0	16.1	13.0	6.4	67.2
23.2	13.4	10.8	16.9	16.0	13.1	6.6	65.7
23.5	13.5	10.8	16.6	15.9	12.7	6.9	64.2
23.1	13.4	11.0	16.5	16.0	12.9	7.1	62.7
23.0	13.6	11.1	16.5	16.0	12.5	7.4	61.2
22.9	13.4	11.1	16.4	15.9	12.6	7.7	59.6
22.8	13.4	11.1	16.2	16.0	12.6	8.0	58.2
23.0	13.3	11.1	16.2	15.7	12.4	8.3	56.6
22.3	13.5	11.2	15.9	15.6	12.3	9.1	55.1
22.2	13.5	11.3	15.8	15.3	12.3	9.5	53.6
22.3	13.7	11.4	15.6	15.2	12.1	9.8	52.2
21.6	13.7	11.6	15.5	15.2	12.2	10.2	50.5
21.4	13.8	11.7	15.5	15.0	12.1	10.5	49.1
21.4	13.9	11.8	15.3	14.9	11.9	10.9	47.6
21.1	13.9	11.9	15.2	14.6	11.8	11.4	46.1
20.9	13.9	11.9	15.1	14.6	11.8	11.9	44.6
20.9	13.9	11.9	14.9	14.4	11.6	12.3	43.1
20.4	13.8	12.0	14.9	14.2	11.7	12.9	41.6
20.2	13.8	11.9	14.8	14.1	11.7	13.5	40.1
20.0	13.7	12.0	14.6	14.1	11.6	14.0	38.6
19.8	13.6	11.9	14.6	13.9	11.7	14.5	37.1
19.4	13.6	11.9	14.4	13.8	11.7	15.2	35.6
19.4	13.6	11.9	14.2	13.6	11.3	16.0	34.0
19.2	13.5	11.8	14.1	13.4	11.3	16.6	32.6
19.2	13.4	11.8	13.9	13.3	11.1	17.3	31.1
18.7	13.2	11.7	13.8	13.2	11.4	17.9	29.6
18.4	13.1	11.7	13.7	13.1	11.3	18.8	28.0
18.3	13.0	11.6	13.4	12.8	11.1	19.7	26.5
18.2	12.9	11.6	13.4	12.8	10.8	20.3	25.1
17.6	12.7	11.5	13.1	12.4	10.9	21.9	22.5
17.3	12.5	11.3	13.0	12.2	10.8	22.9	20.6
17.2	12.4	11.3	12.8	12.0	10.5	23.8	19.1
16.5	12.3	11.4	12.8	11.8	10.6	24.6	17.6
16.0	12.1	11.1	12.6	11.3	10.5	26.3	15.2
15.9	12.1	11.0	12.3	11.1	10.5	27.2	13.6
15.7	11.9	10.9	12.2	10.8	10.2	28.2	12.2
15.6	11.8	10.9	12.0	10.6	10.0	29.1	10.7
15.3	11.6	10.6	11.8	10.5	10.0	30.1	9.1
15.1	11.5	10.6	11.6	10.3	9.8	31.2	7.6
14.7	11.3	10.5	11.3	10.2	9.7	32.3	6.1
14.6	11.2	10.4	11.2	9.9	9.6	33.2	4.6
14.2	11.1	10.2	11.2	9.7	9.2	34.3	3.1
13.9	10.8	10.0	10.9	9.6	9.3	35.4	1.6
13.7	10.6	10.0	10.6	9.4	9.2	36.6	0.0

seen in Figs. 3-5. Similarly to the diffraction patterns, raw Raman spectrographic data has also been provided in a supplementary spreadsheet.

**2. Experimental Design, Materials and Methods**

The (AlFeNiTiVZr)<sub>1-x</sub>Cr<sub>x</sub> MPEAs thin films were synthesized using physical vapor deposition (PVD) via a 5-gun AJA Orion class magnetron sputtering chamber. Seven samples were synthesized on a 7.62 cm Si wafer using conditions predicted by an in-house sputter model to estimate



**Fig. 2.** Waterfall plots of the synchrotron diffraction data of the CCS samples. Spectra are shown with: (a) as-deposited CCS, (b) vacuum annealed for 6 h, (c) vacuum annealed for 6 h and then annealed in air for 1 h, (d) vacuum annealed for 6 h and then annealed in air for 3 h, (e) vacuum annealed for 6 h and then annealed in air for 6 h, and (f) vacuum annealed for 6 h and then annealed in air for 18 h. Both vacuum and air anneals took place at 873 K.

Cr atomic percentage between 0 and 30 at.% [4]. The sample libraries were deposited at room temperature with an AlFeNiTiVZr metal alloy target sputtered with a radio frequency (RF) sputter source with 150 W of power supplied at a 14 mm tilt and a Cr metal target supplied with 15 W direct current (DC) power at a 7 mm tilt. The materials were angled 144° apart from one another, and at 0.667 Pa Ar sputter pressure. This resulted in 100 nm thin films. After deposition, a single sample was left as deposited. All subsequent samples were annealed in vacuum at 873 K for six hours. Five of the vacuum annealed samples were then annealed in air at 873 K, each at different anneal times. The annealing times were 1, 3, 6, 12, and 18 h.

WDS measurements were taken in order to validate the in-house sputter model. Measurements were obtained using an Electron Probe Microanalyzer (EPMA) JXA 8900R Microprobe with an acceleration voltage of 15 kV. A standard was used to compare against each element with the (AlFeNiTiVZr)<sub>1-x</sub>Cr<sub>x</sub> system. Additional measurements were taken in order to maintain minimal

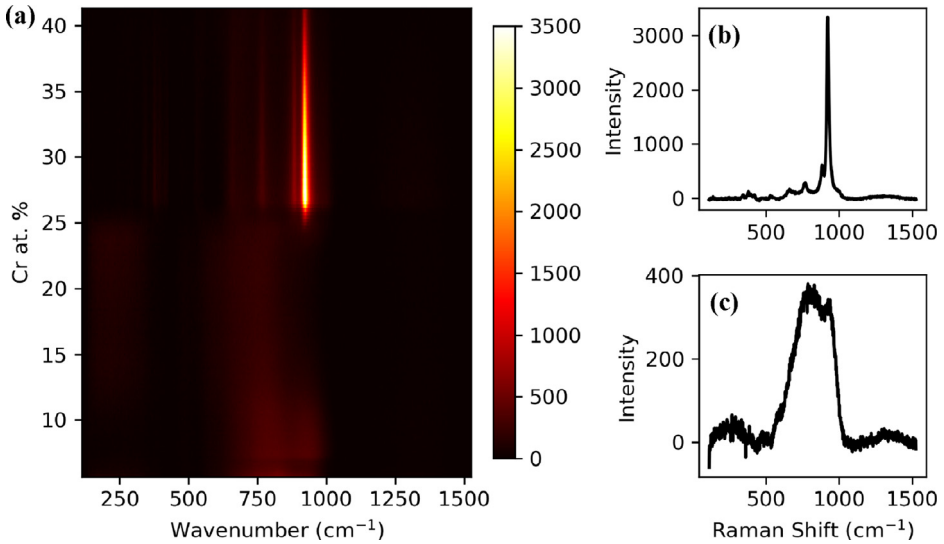


Fig. 3. Raman spectra for 1-h anneal in air. (a) Heatmap (b) 30 at.% Cr (c) 7 at.% Cr.

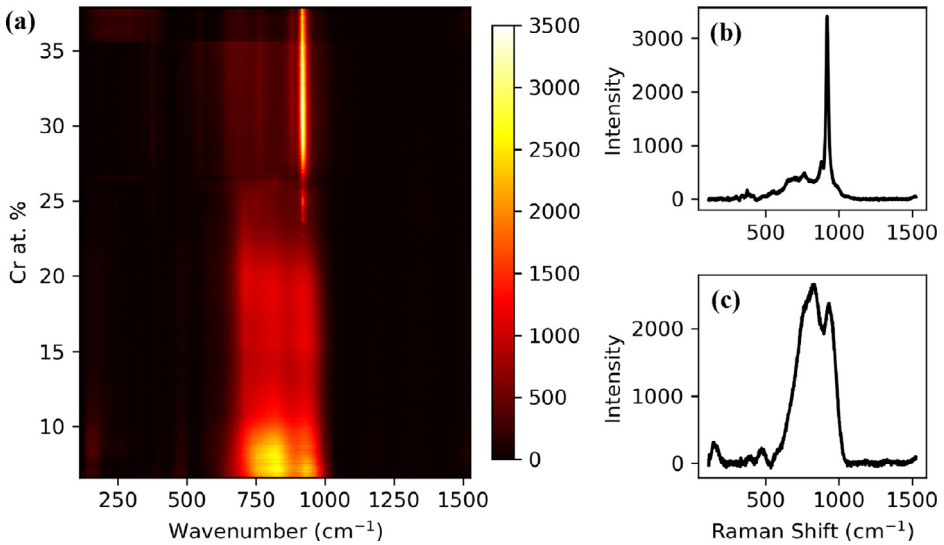
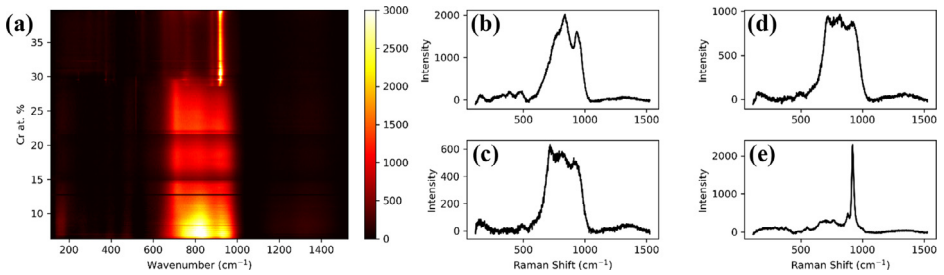


Fig. 4. Raman spectra for 6-h anneal in air. (a) Heatmap (b) 30 at.% Cr (c) 7 at.% Cr.

fluctuations between validation measurements. The largest average variation between measurements was 0.1 at.%.

X-ray diffraction patterns were measured using the 1–5 beamline at the Stanford Synchrotron Radiation Lightsource (SSRL) at SLAC. Samples were mounted on an x-y translational stage and were aligned with an incidence angle of 6° with the x-ray source. The wavelength of the x-ray source was 0.9762 Å (12.7 keV). Measurements were taken at room temperature across a grid of 441 points on a MarCCD. The sample to detector distance and incidence angles were calibrated using a LaB<sub>6</sub> standard. The 2-D area diffractograms were converted to 1-D patterns via an azimuthal integration from an “on-the-fly data assessment” Python script [5].



**Fig. 5.** Raman spectra for 18-h anneal in air. (a) Heatmap (b) 5 at.% Cr (c) 15 at.% Cr (d) 20 at.% Cr (e) 35 at.% Cr.

Raman spectroscopy was performed using a Horiba XploraPLUS Raman microscope. Samples were placed on an x-y translational stage and focused with a 20x microscope objective with a numerical aperture (NA) of 0.40, working distance (WD) of 12 mm, and spot size of approximately 1.44  $\mu\text{m}$ . The excitation light source was a 25 mW, 473 nm blue solid-state laser. A 2400 lines/mm holographic grating was used to separate the Raman scattered light for the thermoelectrically air cooled Sincerity CCD detector. The Raman spectra were captured over a 5 second acquisition time and averaged over 3 spectral accumulations. The distance between the sample measurements was 0.4 mm and samples were moved on a single axis to measure a total of 191 points.

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships which have or could be perceived to have influenced the work reported in this article.

### Acknowledgments

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### Supplementary Materials

Supplementary material associated with this article can be found in the online version at doi: [10.1016/j.dib.2021.106758](https://doi.org/10.1016/j.dib.2021.106758).

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