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High-throughput nanoscale mechanical property mapping under high temperature and high vacuum environment

Bartosz K. Nowakowski[1], Eric Hintsala[1], Douglas Stauffer[1], Bernard R. Becker[1]

[1]*Bruker Nano Surfaces, Eden Prairie, MN, USA*

Bartosz.Nowakowski@bruker.com

Abstract

Materials development for next-generation alloys in critical applications requires high-throughput methodologies. This is due to the number of alloy permutations, processing routes, and operando conditions to be evaluated. Nanoindentation can provide statistical datasets at length scales that allow not just macro behaviour, but behaviour at microstructural length scales to be evaluated. A new nanomechanical test system, capable of elevated temperature testing under vacuum, enables mechanical mapping of oxidation sensitive materials. As a demonstration of the system's capabilities, a dual phase $FeNiCoCrMnAl_{0.3}$ high entropy alloy is evaluated which is an alloy of interest for nuclear applications owing to its potential to absorb substantial radiation defects. We correlate the mechanical maps made via nanoindentation with crystallographic maps made via electron backscatter diffraction. We also explore the automatic assignment of indents to the two phases using machine learning clustering techniques with reasonable success. This alloy is found to have stable mechanical properties in the temperature range evaluated, which will prompt further studies at higher temperatures and under simulated radiation damage.

Nanoindentation, High Entropy Alloys, Electron Microscopy, Machine Learning

1. Introduction

For the development of advanced alloys designed to be utilized in extreme environments, the ability to test individual phases or microstructural features at the desired operating temperatures is valuable. For instance, softening by annealing or formation of unwanted new phases can degrade the performance of the materials. To do these measurements, nanoindentation is a high throughput technique with the necessary resolution that only requires a flat polished specimen, from which many thousands of measurements can be extracted. One limitation is oxidation or other contamination of the sample surface at high temperatures, which interferes especially with a surface characterization technique [1,2] like nanoindentation. For this purpose, development of a high vacuum, high temperature nanoindenter was undertaken. This system can perform an indent and reposition a sample within seconds, which has numerous benefits [3]. The most obvious is the feasibility of maps with a large number of data points, but also reduced tip-sample reactions which can produce rapid probe wear and influence of thermal drift. An ideal test case is a dual phase high entropy alloy. The high temperature performance of these alloys is also of concern since they are in a relatively high energy metastable state by virtue of their processing route [4]. The number of potential alloys in a given HEA family is massive and it is hoped that exploring these compositional spaces will yield alloys with superior combinations of properties for different applications. This means a lot of microstructural-level mechanical data is needed to help with the screening and alloy development process [5].

2. High Vacuum Nanoindentation Platform

A commercially available vacuum chamber (Kurt Lesker Co.) was used as the enclosure for the vacuum nanoindenter, which can reach the 1E-7 torr range. The internals follow the tried and true formula from the Hysitron TI-980 TriboIndenter (Bruker) platform, except redesigned for use in a high vacuum. The gantry has been changed from granite to invar to aid in temperature stability. Vacuum compatible, stepper motor driven precision positioning stages are utilized with added water cooling for the motors and appropriate vacuum compatible lubrication. The transducer is based upon a multi-range nanoprobe (Bruker), which is a piezo actuated system with a capacitive, flexure-based load cell sensor.

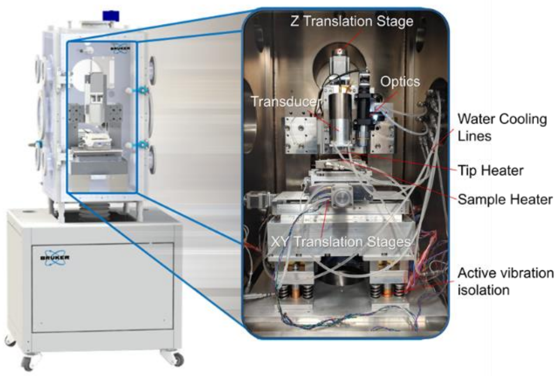

Figure 1. Layout of the Vacuum Nanoindenter system

The indenter tip and sample are heated independently using resistive heater coils under closed-loop control with feedback from thermocouples and can reach temperatures in excess of 800°C. Materials for the remaining components were carefully selected for vacuum and additional water cooling was also integrated into the metrology head to combat radiative heating from the high-temperature system. Thermal breaks and active water cooling are utilized to minimize heat flow into the rest of the system. Finally, the system has two types of vibration isolation, which are active piezo system underneath the gantry and passive vibration isolation air shock feet isolating the chamber from the floor. Indents are optically targeted using a 10x objective lens with a LED light source. When operating in mapping mode, the tip is retracted and actuated using the piezo while the stage is moved underneath to the next specified position.

3. Methods and Materials: Case study on FeCrCoMnAl0.3

The HEA samples were provided by collaborators from Los Alamos National Laboratory. This material is of interest for nuclear applications since there's potential for the absorption of many radiation defects [6]. It has a microstructure consisting of dendrites of fcc material, which is Ni-rich. The interdendritic regions were an eutectic lamellar mixture of Cr-rich BCC phase and the FCC phase. Mapping was carried out using a diamond Berkovich probe with 10um indent spacing and 1um indent depth in displacement-controlled loading. The loading time was 1s and the total time between indentation was 3s giving a complete map in just under 5 min. The indentation grids were correlated using fiducial markers with SEM based post-mortem analysis. The clustering analysis was performed using a K-means algorithm from sci-kit learn [7]. The only required input was the number of clusters to sort the data into.

4. Results: Case study on FeCrCoMnAl0.3

A 12x12 hardness map is shown for each of three temperatures as measured under vacuum: RT, 300°C and 400°C. It can be generally observed that the highest hardness regions indeed correlate to the darker, minor phase in the microstructure which is the BCC/FCC interdendritic region. For the clustering, we chose to use 3 clusters, to attempt to capture the pure response of the two distinct phases and an extra cluster for tests landing on or near the interface (since a nanoindentation test has a long-range stress field).

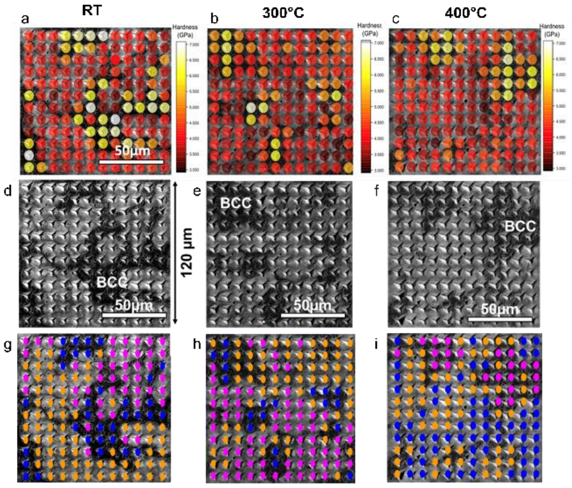

Figure 2. Comparison of Hardness (a-c), post-mortem SEM (middle-f) and clustering (bottom-i) for RT, 300°C and 400°C.

The final results of the hardness of the two distinct phases (having removed the interface points from the clustering method, resulting in 72% data usage) give Figure 3. It can be observed that the agreement between the manual method and machine-learned clustering is reasonable. The overall property change was a slight decrease in hardness for both material phases, which indicates good thermal stability of the mechanical properties in the tested temperature range [8].

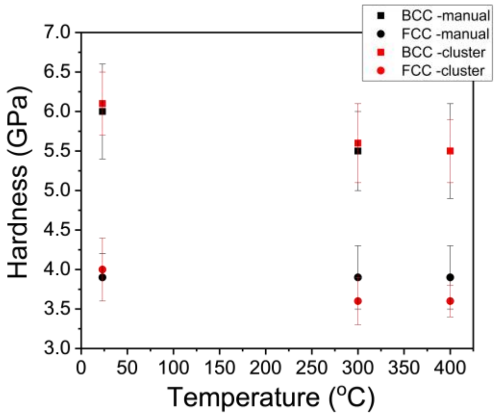

Figure 3. Comparison of Hardness as assigned to the two phases of this microstructure as a function of temperature for both the manual assignment and machine learning clustering technique.

5. Closing

In summary, high throughput evaluation of individual properties of microstructural phases is a valuable goal for alloy development in advanced applications. The use of a high vacuum environment helps to protect the sample surface at elevated temperatures and improve measurement accuracy for nanoindentation techniques. We successfully demonstrated this capability on a dual phase high entropy alloy, complete with the initial evaluation of a high-throughput analysis scheme using machine learning based on K-means clustering.

Future work would include evaluation of additional data for the clustering scheme – this would include some kind of spatial weighting or correlated structural and chemical data from other techniques. For the alloy family studied here, the introduction of simulated radiation damage will also be tested in a similar scheme to that presented here.

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