Sputtering Yield As Structural and Compositional Signature in Refractory High Entropy Alloys

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Goal

Establish sputtering yield as a compositional, structural, mechanical property and processing signature for refractory high entropy alloys.

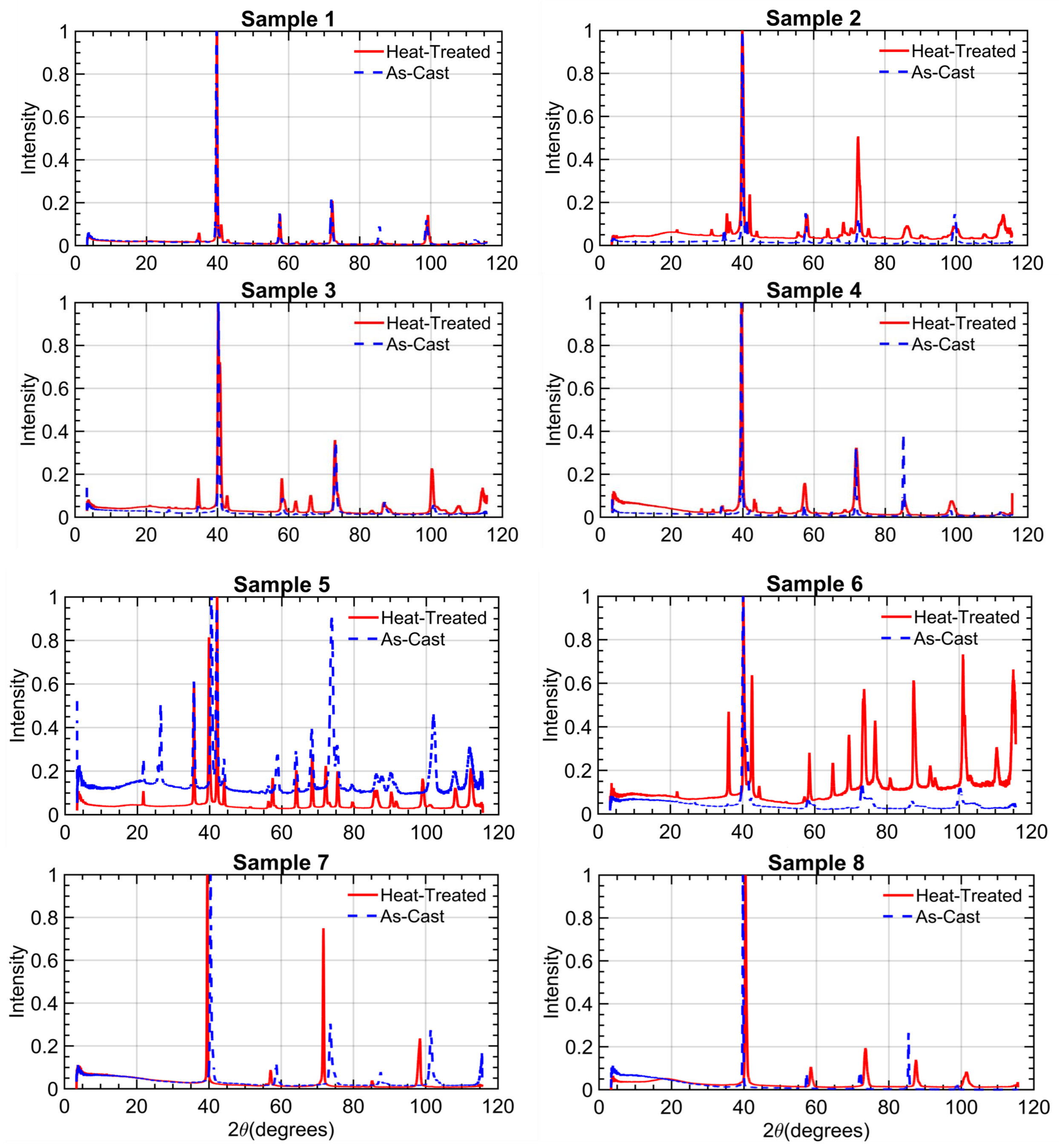
Introduction

Plasma-facing components (PFCs) in fusion reactors will become activated within their service lifetime. This is a non-proliferation risk, and proper steps must be taken to ensure these materials are properly identified and handled. Refractory high entropy alloys (RHEAs) have been proposed as PFC materials, but can contain upwards of five different elements with widely varied compositions, making characterizing their activation risk within an operating fusion reactor a challenge. This work proposes sputtering yield as a compositional, structural and processing signature, and describes the fabrication, heat-treatment, and materials characterization of alloy systems of interest. Select heat-treated samples were sputtered using a focused-ion beam, and measured materials properties of interest were compared to find relations to sputtering rate.

Methods

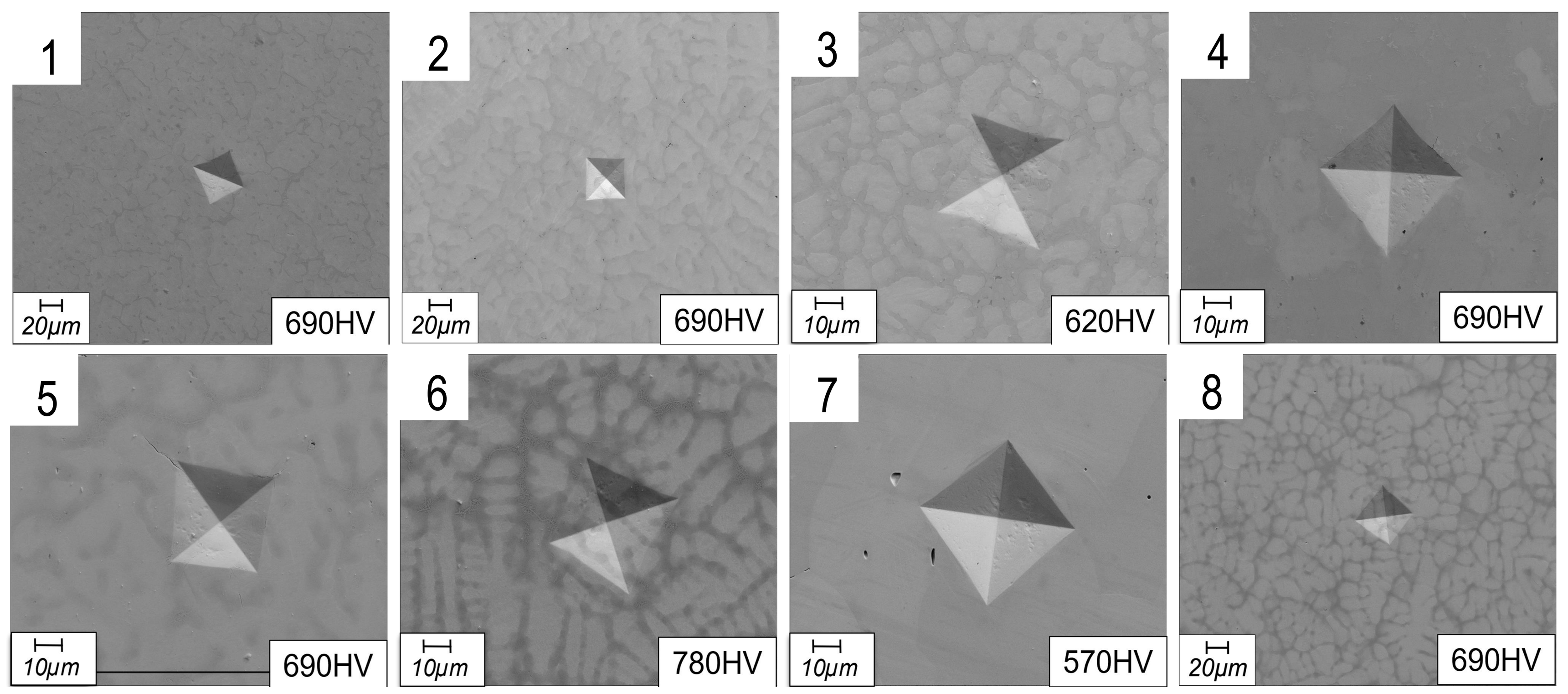
1. Samples were arc-melted into ~1" diameter coupons and cut in half by diameter.
2. One half of each sample were left as-cast, the other half were heat-treated at 1400 ^{O}C for 24 hr.
3. Each half-sample was measured for Vicker's Hardness, phase structure, and compositional homogeneity.
4. Identical volumes of select heat-treated samples were sputtered using a Ga^{+} 30 kV focused-ion beam. The measured time for each sample to sputter gave the sputtering rate.

X-ray diffraction

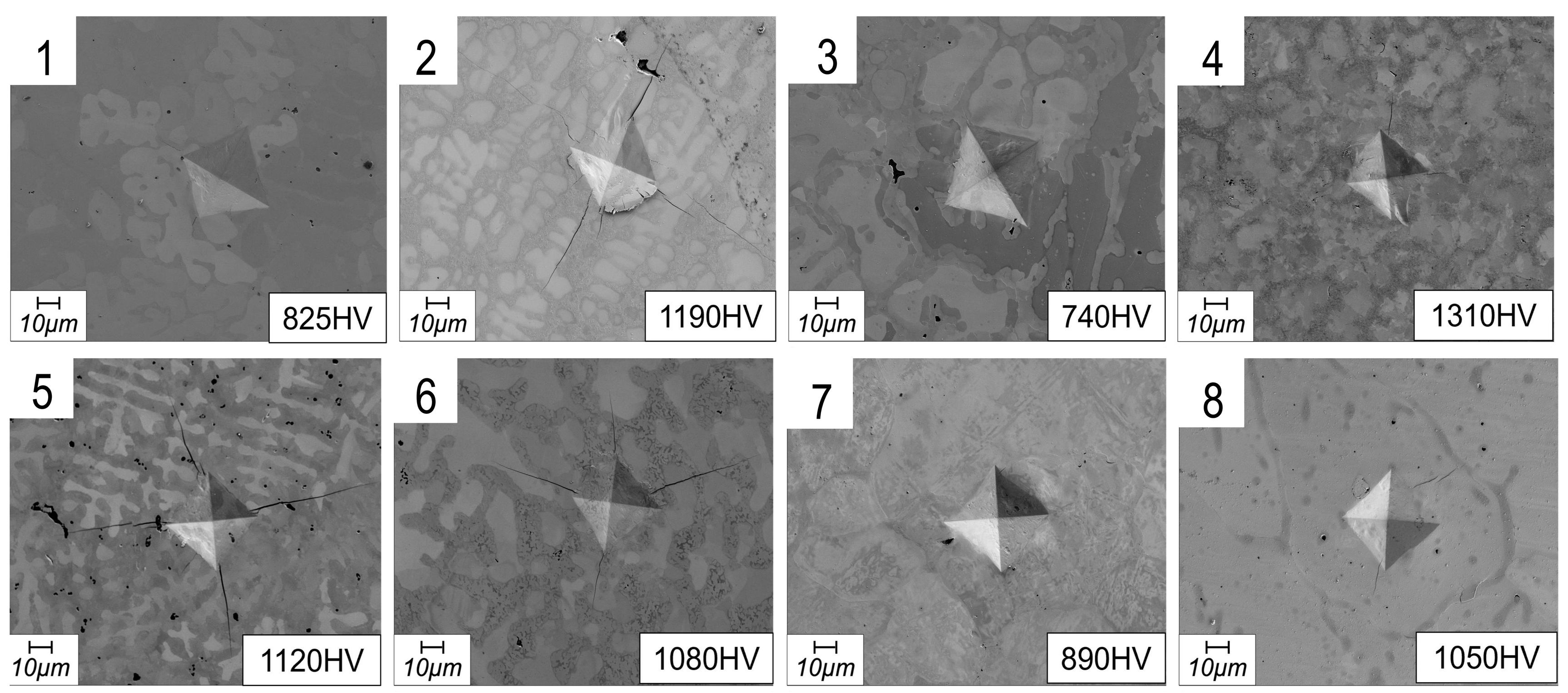


All samples showed characteristic BCC peaks at ~39^{O}, with some showing a characteristic Laves peak at ~35^{O}.

Arc-cast

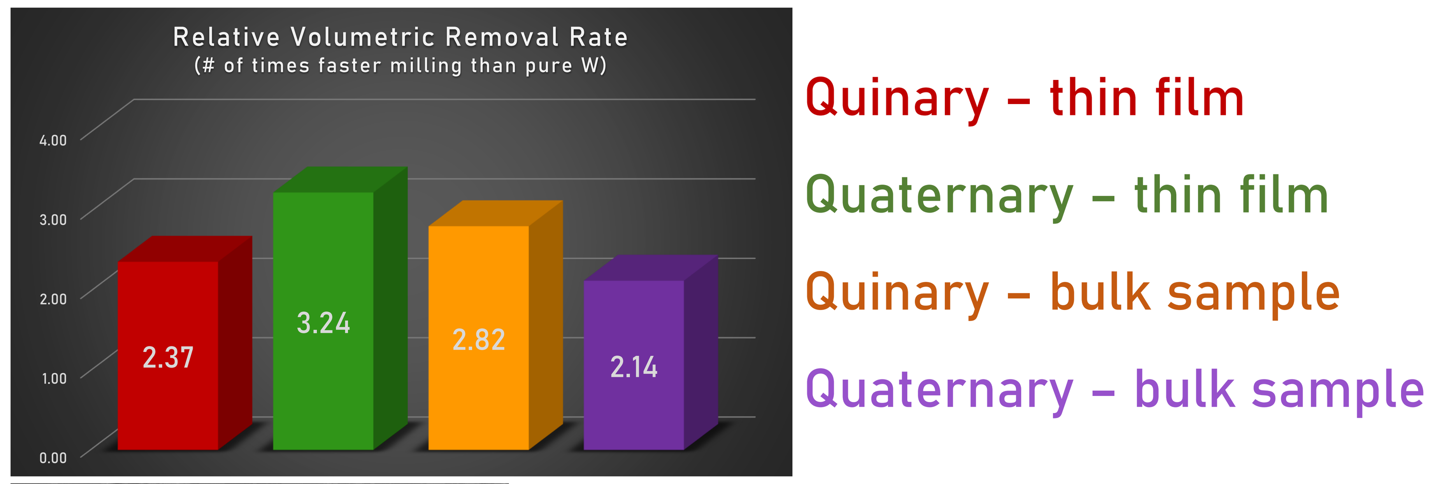


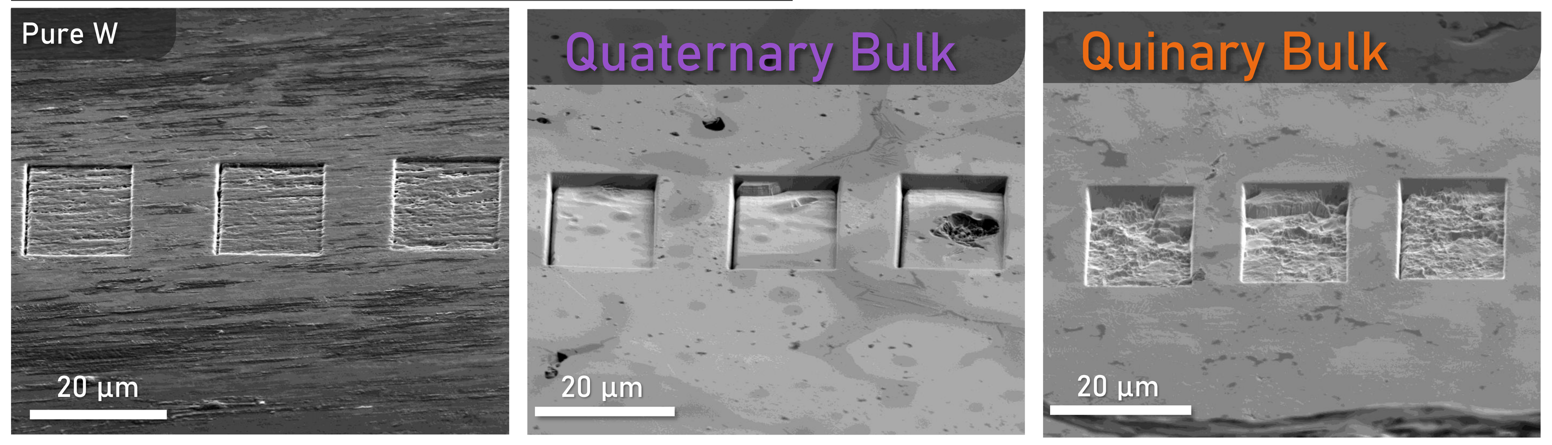
Heat-treated (1400 ^{O}C/24 hr)



Microindentation on WTaVCr and WTaCrVHf alloys showed increased hardness in heat-treated samples with minimal cracking.

Sputtering Data





Both RHEAs (center, right) showed preferential sputtering compared to W due to the introduction of sputtering-susceptible elements.

Results/Major Findings

1. All samples showed at least one BCC phase, as expected for RHEAs.
2. Multiple samples showed evidence of formation of Laves phases in XRD, validated with EDS line scans suggesting AB_2 compound formation.
3. All heat-treated samples showed higher hardness than their as-cast counterparts.
4. Heat-treated **sample 4** showed the highest hardness at 1310 HV.
5. Heat-treated **sample 8** showed the lowest sputtering rate, suggesting sputtering resistance is not directly related to increased hardness.

Discussion

Results show sputtering rate is a viable signature for identifying difference in composition compared to pure W. While the RHEA samples had a higher sputtering rate compared to pure W, further investigation is necessary to measure preferential sputtering rate of each constituent element within the alloys. Of interest is whether sputtering is a property dictated strictly by the atomic ratio of the constituent elements within a RHEAs, known as a linear rule of mixtures. Sputtering rate is known to relate to composition, but whether removal rate is affected by the fundamental properties of an HEA is currently unknown. While heat-treatment was successful in precipitating out a hard intermetallic Laves phase in multiple samples, the limited number of samples measured using sputtering prevented prediction of whether presence of a Laves phase affected removal rate. Future plans to increase duration of heat-treatment will increase the number of available samples, providing more robust comparison. Because hardness and sputtering do not appear to share a relationship, sputtering may not be a viable processing signature to identify whether a sample was heat-treated. Further work comparing arc-cast to heat-treated samples would be necessary in order to verify.

Conclusions/Relevance to Program Objectives

1. Sputtering yield has been shown to be a feasible signature for composition within RHEAs.
2. No relation was found between hardness and sputtering in RHEA samples, suggesting sputtering as a processing signature is currently non-viable.
3. Data normalization is recommended to minimize variances in sputtering data based on day-to-day focused-ion beam performance.
4. Future work may include replicating sputtering studies with samples under higher lengths of heat-treatment durations, and/or after experiencing fusion-relevant damage, i.e. irradiation, oxidation, high-flux plasma fluxes and fluences.
5. Continuing to develop sputtering as a signature will establish a new method to evaluate activation risk of potential high entropy materials used as PFCs.

ADDLab Alloy Design and Development

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NNSA National Nuclear Security Administration