



Stress evolution in sputtered vanadium-tungsten alloys

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ABSTRACT

While metal alloy films are used in many applications, there remains a rather limited series of studies of their stress evolution during growth as compared to elemental films. Here, we provide measurements of the sputter deposition stress evolution of V_xW_{1-x} alloys, which is a system that forms a body centered cubic solid solution across all its compositions. By increasing either the tungsten content or the growth rate, we note an increasing trend towards a compressive stress state. Since the crystalline size is approximately the same for all alloys and growth rates, we conclude that the increasing tungsten content enhances the effect of energetic particle deposition (atomic peening) on the stress facilitating this change. The results suggest that stress can be understood in terms of the same physical processes that have been proposed previously to explain stress in elemental films. These mechanisms, described in the text include effects of non-energetic growth kinetics, microstructural evolution and energetic particle bombardment.

1. Introduction

Alloy films have strong technological relevance with a multitude of applications in magnetic storage [1,2], catalysis [3], and hard coatings [4]. Understanding the development of stress is important because it can impact the film's performance and reliability. However, compared with elemental films, there are far fewer measurements of stress evolution in sputtered alloy films. Those that have been performed generally have not examined systematic changes in the processing parameters, which is useful for elucidating the underlying mechanisms controlling the stress evolution.

In this paper, we have selected to study the W-V system, which forms a complete body centered cubic (BCC) solid solution over all compositions. The measurements were performed at different growth rates for different compositions to obtain a comprehensive set of residual stress data that could be compared with prior stress studies of elemental films grown under similar deposition conditions. By doing so, the growth rate dependence of the stress provides a useful means for determining the mechanisms that control stress in a solid solution alloy film.

2. Background

Previous stress evolution measurements in Ni-Mn [5] and Cu-Al [6] have shown that stress changes may occur due to the difference in the concentrations of the two species. Specific work by the authors of this paper have shown concentration behavior in a number of binary alloys that include Fe-Pt [1], W-Ti, Cu-Ag, Cu-Ni, Cu-V [7–9], and the W-Fe, W-

Cr, Fe-Cr [10,11] series. In some of these systems (e.g., Cu-V, W-Fe and W-Cr), the stress varied continuously from the behavior of one element to the other as the composition changed. Often, the stress is found to become more compressive for higher content of the material with larger atomic number and/or higher melting point. But this behavior is not universal. For the Fe-Pt and Cu-Ni alloys, the measured stress was below the single element metal values. This type of stress response has been reported in other alloys as well, e.g., Angay et al. [12] noted this behavior in the binary Mo-Al alloy and the ternary Mo-Al-N alloy and Cu-Gc alloy by Furgeaud et al [13]. Hence, the stress thickness behavior in alloys cannot be assumed to vary in 'Vegard's law rule-of-mixture' manner according to their composition.

One of the challenging issues in alloy stress evolution is to understand how the alloy's thermodynamic properties such as the enthalpies of mixing and segregation energies contribute to stress evolution. Such properties may affect the mechanisms of adatom mobility, microstructure evolution and other processes that contribute to the stress evolution. Recognizing the complexity of the processing space (pressure and rate) and material design opportunities (thermodynamic energies in alloys) with respect to residual stress evolution, we have specifically sought to identify an alloy for study where these characteristics are well understood. Specifically, by using a solid solution, the confounding influence of chemical partitioning is mitigated and the effect of alloy mixing on the growth stresses can more easily be evaluated as a function of the overall growth rate.

Tungsten is of significant technical interest because of its refractory characteristics and is a well characterized in its elemental deposited film

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state [14–18]. However, in the bulk, tungsten is well known to have limited ductility at ambient temperatures [19]. In contrast, vanadium has significantly more ductility than tungsten [20] and the alloying of tungsten with such solutes is a well-established method to change its mechanical ductility [21]. While this work will assess the residual stress evolution under deposition, the bulk mechanical properties highlight how solid solution mixing can induce mechanical property changes.

3. Experimental and analytical procedure

Single element (vanadium and tungsten) and binary alloy (V_xW_{1-x} , where x is the atomic composition, denoted as V - (at.% of W) W) thin films were deposited at room temperature onto Si 100 wafers in an AJA ATC-1500 sputtering chamber. The base pressure in the chamber was 1.33×10^{-5} Pa and the substrates were rotated at 30 rpm during the deposition. The deposition pressure for all films was 0.27 Pa, which was obtained by the flow of ultra-high purity Ar at 15 standard cubic centimeter per minute with a throttled gate valve to the turbomolecular pump. The thin films were deposited by DC magnetron sputtering from a 2-inch diameter tungsten target with a purity of 99.95 % and up to three 2-inch diameter vanadium targets with a purity of 99.9 %. The target-substrate distance was approximately 18 cm and the deposition rate for each target was adjusted to obtain the total deposition rate and composition desired.

The pure vanadium samples were deposited at rates from 0.02 to 0.36 nm/s. For the alloys, the deposited compositions were V-25 W (at. %), V-33 W (at.%) and V-50 W (at.%) in the range of 0.08 nm/s to 0.36 nm/s. The film thicknesses ranged from 200 to 400 nm with the stress-thickness responses recorded over that range. The deposition rate for each source was measured by a quartz crystal microbalance in the chamber, and the total deposition rate was confirmed by cross-section measurements in the scanning electron microscopy (SEM).

The real-time stress evolutions were captured by a k-Space Associates® multibeam optical sensor system (MOS). The laser dot array was generated by two sets of etalons that reflected the spits into a charge coupled device camera. The obtained stress evolution change, created by the curvature bend of the substrate during growth, is expressed by the product of the stress and film thickness using the Stoney equation [22,23]:

$$\frac{\sigma}{h_f} = \frac{M_s}{6h_s^2} \kappa \quad (1)$$

where κ is the measured curvature, M_s is the biaxial modulus of the substrate and h_s is the substrate thickness. The product of the average stress, σ , over the thickness of film and the film thickness, h_f generated the stress-thickness (σh_f) responses during growth and can be derived by integrating the stress along the film thickness, i.e.,

$$\sigma h_f = \int_0^{h_f} \sigma dz \quad (2)$$

where z is the direction normal to the substrate-film interface and σ is the stress on the parallel plane that is z above the interface. If the stress in the previously deposited layers does not change, the slope of the stress-thickness vs. thickness ($d(\sigma h_f)/dh_f$) is equal to the stress in the newly deposited layers at the surface (termed the incremental stress).

The composition of the deposited alloy thin films was measured by X-ray Energy Dispersive Spectroscopy (EDS) on SEM in a Thermo Scientific Quattro S microscope. The voltage used in the SEM varied from 5 kV to 10 kV for imaging, but only 10 kV was used for performing EDS measurements. The film's texture and lattice parameter were characterized by X-ray Diffraction (XRD) on a Bruker D8 Discovery 2D X-ray Diffractometer with a Cu K α source. Grain size measurements were estimated from the reflected intensities using the Scherrer equation, i.e.,

$$D = \frac{K\lambda}{\Delta 2\theta \cos \theta} \quad (3)$$

where D is the measured crystalline size, which may be smaller or equal to the grain size. K is the dimensionless shape factor with a value of 0.89 used based on the cubic symmetry of the structure [24], λ is the line broadening at half the maximum intensity (FWHM), and θ is the Bragg angle for the reflection. As some of the line broadening is from the optics in the diffractometer XRD system, this contribution was removed from our analysis by experimentally measuring instrumental broadening, $\Delta 2\theta_{inst}$, by collected a peak breadth from a large grain size sample (grain size $> 1 \mu m$) and subtracting this width from the width of the much more finer grain sizes in our films, $\Delta 2\theta_{film}$, using

$$\Delta 2\theta_{film} = \Delta 2\theta_{measured} - \Delta 2\theta_{inst} \quad (4)$$

Finally, the large penetration distance of x-rays enabled the measured crystallite size to be effectively an average over the thickness of the film based on our film thickness being at or > 400 nm. This was confirmed by the capturing of the Si substrate XRD intensity from its single crystal reflections.

4. Results

4.1. Stress thickness evolution in V_xW_{1-x} films

Stress-thickness measurements were made for films with different compositions at different growth rates in the range of 0.08–0.36 nm/s with these results for elemental vanadium, tungsten and alloy compositions of V-25 W, V-33 W and V-50 W plotted in Fig. 1. These films were grown at a constant growth rate up to the maximum thickness as shown in this figure with the growth rate corresponding to each curve indicated in the legend. For each measurement, the slope of the stress-thickness reaches a constant value as the thickness increases. This indicates that the stress in the already-deposited layer is not evolving so that the slope can be attributed to the incremental stress as described in the experimental section. The dependence of incremental stress on the growth rate and composition will be discussed more fully in Section 5 of this paper. However, some general features can be observed in the stress-thickness curves and are shared below.

For vanadium, the stress exhibited a tensile response for all the growth rates, i.e., a positive slope). The dependence on growth rate was not monotonic, showing that the stress becomes more tensile with increasing growth rate at the lowest deposition rates (< 0.16 nm/s), but then becomes less tensile at higher growth rates (> 0.16 nm/s). For tungsten, the stress was compressive and kept similar magnitude for all the growth rates. For V-25 W film, the stress was tensile at the lowest growth rate and as the growth rate increased, the slope decreased and the stress became less tensile and more compressive. For V-33 W and V-50 W films, the stress was compressive at the lowest growth rate and became more compressive at higher growth rates. For elemental tungsten, the stress was compressive and did not change significantly with the growth rate.

4.2. Incremental stress

In addition to the full stress-thickness data, the incremental stress is useful for comparing the stress at different compositions and the respective growth rates. Recall that the incremental stress is the stress in new layers being deposited on the film after it has become continuous and uniform. It is obtained by calculating the slope of the stress-thickness curve at large thickness, where it becomes invariant with respect to the thickness. The incremental stress depends on the processing conditions and the film's grain size at the final measured film thickness, but it does not depend on the history of the film's stress evolution at lower thicknesses. Therefore, it is a convenient means for

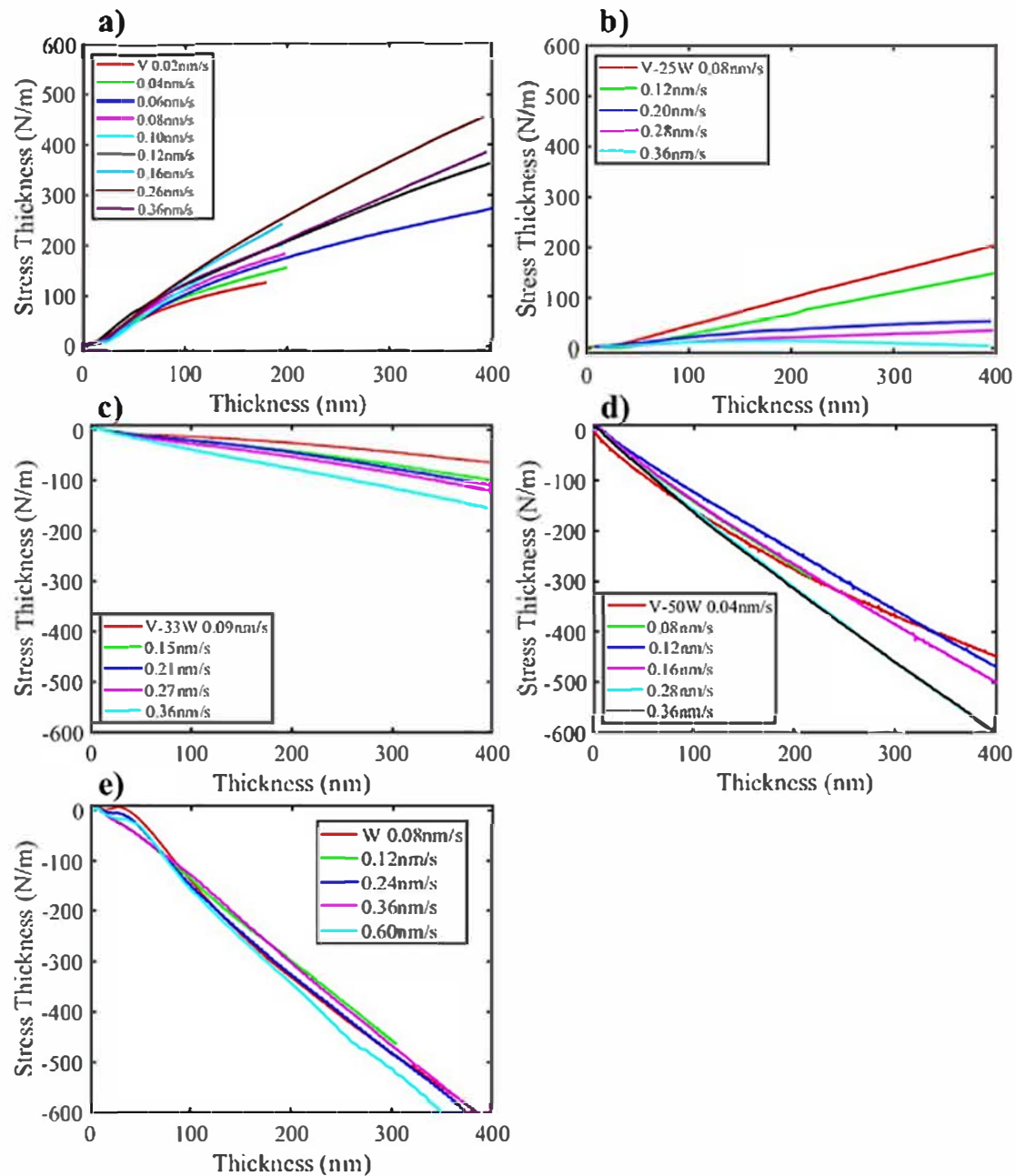


Fig. 1. Stress thickness data for film compositions of a) V, b) V-25 W, c) V-33 W, d) V-50 W and e) W. The growth rates are indicated in the legend of each figure.

comparing growth of different compositions and growth rates when the grain sizes are similar and not changing, as in this study.

Fig. 2 is a plot of the incremental stress as a function of growth rate for the different compositions. Several important trends can be noted. For each growth rate, the incremental stress tends to become less tensile, or equivalently, more compressive when the fraction of tungsten increases in the alloy. For the alloys containing tungsten, the stress tends to become less tensile or, equivalently, more compressive for higher growth rates. The major deviation from this behavior is for elemental vanadium which shows different dependence on the growth rate than the tungsten-containing alloys. Here, the incremental stress becomes less tensile at lower growth rates, similar to what is observed for materials with higher adatom mobility under non-energetic growth conditions [25].

4.3. XRD measurements

All XRD scans confirmed a BCC solid solution, as is thermodynamically predicted, a preferred [110] growth texture, Fig. 3(a), and a shift in lattice parameter with composition, Fig. 3(b), following a rule-of-mixture trend. Measurements of the crystallite size for different compositions and growth rates determined from the XRD measurements are shown in Fig. 3(c). For consistency, all crystallite size measurements discussed here were made at a film thickness of 400 nm; however, as a point of reference, measurements of 200 nm films with the same processing conditions had essentially the same crystallite sizes as the 400 nm thick films which indicate that the crystallite size was not strongly dependent on film thickness and is consistent with the invariant slope of the stress-thickness plots in Fig. 1. The crystallite or grain size results for the different compositions have very similar sizes for all compositions with elemental vanadium being slightly smaller in size than the other

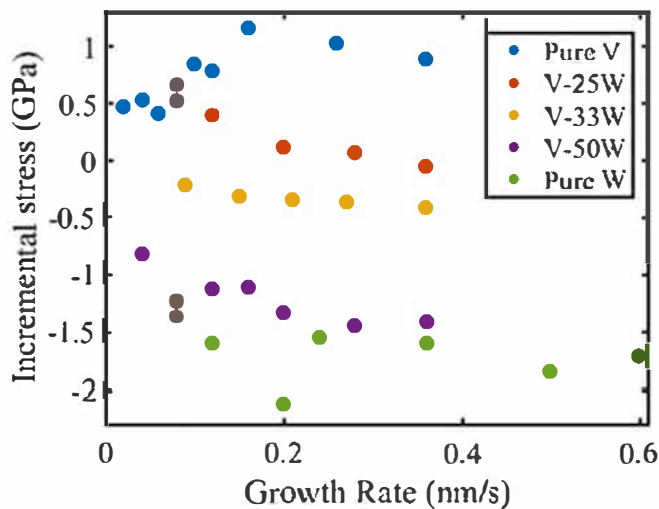


Fig. 2. Incremental stress vs. growth rate for films deposited with the composition indicated in the legend.

compositions. In general, there appears to be weak dependence of crystallite size with respect to growth rate.

5. Discussion

To understand these features of stress, one can consider mechanisms that have been used previously to model stress evolution in sputter-deposited, elemental metal films. These mechanisms, described in [26] include the effects of non-energetic growth kinetics, microstructural evolution and energetic particle bombardment. Performed on systems with a single atomic species, this previous work provides insight into how growth kinetics, microstructure evolution, and energetic particle interactions may contribute to stress. Since the alloy in this work forms a solid solution, one can invoke similar concepts in the single-phase films deposited. These mechanisms include the effects of 1) deformation and defect production by energetic particles, 2) processes of non-energetic film growth, and 3) grain size evolution, with detailed descriptions of these found in references [23,27,28] for the interested reader.

An important feature of sputter deposition is that the arriving particles have higher kinetic energy than those deposited by other means such as evaporation. It has been well-documented that the transfer of momentum from the incoming particles can modify the film's growth behavior and properties [29]. The energetic species include both the deposited species (V and W) and the background gas (Ar in this case); their energy can be modelled by Monte Carlo methods [30,31] and is typically <400 eV.

Such energetic particle bombardment has been proposed to contribute to compressive stress in the film by either collision-induced densification (referred to as atomic peening) [32,33] or by the trapping of ion-induced defects in the bulk of the film [27,34]. These mechanisms (atomic peening and defect trapping) were incorporated into an analytical model that has been used to model the stress in a number of elemental sputtered metal films for a range of processing condition (growth rate, temperature, pressure) [26]. The ability of the model to obtain good quantitative agreement with the measurements suggests that the mechanisms included in it are a useful way to consider the results in the alloy films.

The analysis of various elemental sputter-deposited films shows that the two energetic mechanisms in the model have a larger impact on compressive stress generation for materials with higher melting points (or equivalently, lower adatom mobility for the same temperature) and for higher atomic mass [26]. This observation can be invoked to explain the dependence of the stress on composition observed for the alloys. For V-W alloys with higher tungsten content, the melting point increases, which suggests that the corresponding energetic terms controlling stress would also increase with tungsten content. This is consistent with the incremental stress changes seen in Fig. 2. For the same growth rate, the incremental stress became more compressive when the tungsten content is increased.

For each measured composition (except elemental vanadium), the stress tends to be more compressive when the growth rate is increased. This dependence is also consistent with what has been seen in single element sputtered films that have relatively low atomic mobility. Such behavior is attributed to an enhancement in the trapping of particle-induced defects when the sputter deposition rate is increased. For very low mobility materials, e.g., elemental tungsten, the defect trapping may saturate so that the dependence on the growth rate (i.e., slope of the stress vs. growth rate data in Fig. 2) may become weaker than it is for compositions with lower tungsten content.

The effects of energetic particles described above do not explain why the stress is observed to become less tensile at lower growth rates for elemental vanadium. To explain this behavior, we consider the effect of other stress generating mechanisms that are included in the model. These are due to non-energetic growth kinetics, but also occur during sputter deposition [26]. These mechanisms were initially proposed to explain the stress evolution in evaporated and electrodeposited films [25]. The stress in these cases was attributed to competing tensile and compressive stress generating mechanisms that occur at the triple junction between adjacent grains as new segments of grain boundaries were formed. The tensile stress is proposed to be generated due to attraction between adjacent grains that lowers the energy by forming new segments of grain boundary. The compressive stress is proposed to be due to the insertion of adatoms into the grain boundary, driven by deposition-induced supersaturation on the surface. Modeling of these materials suggests that the resulting stress is growth rate and material

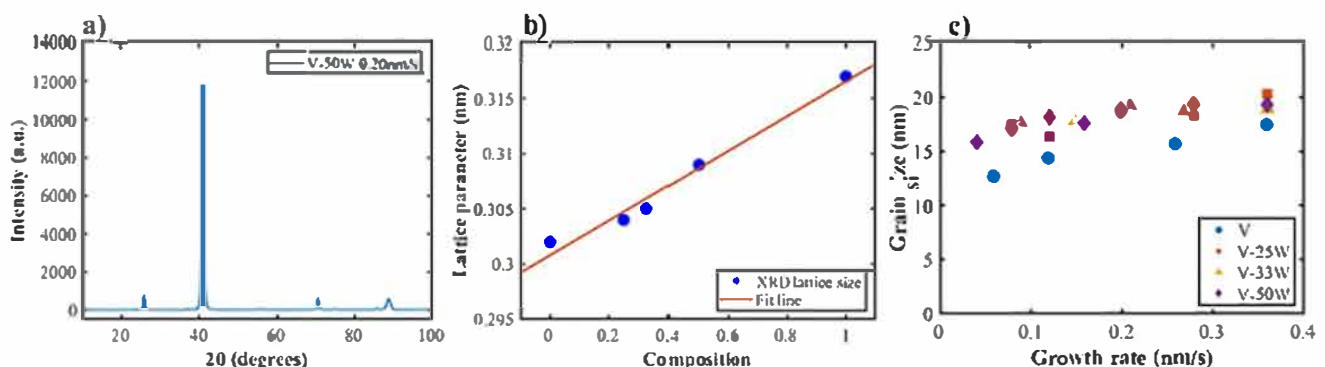


Fig. 3. a) XRD patterns for V-50 W films deposited. b) Lattice parameter vs atomic fraction of W. c) Grain size vs. growth rate for 400 nm films deposited with the composition indicated in the legend.

dependent. For materials with low adatom mobility or high growth rates, the tensile stress mechanism is dominant; for high adatom mobility or at low growth rates, the compressive stress mechanism is dominant. For materials with sufficient atomic mobility, the stress will transition from compressive to increasingly tensile as the growth rate is increased.

For the tungsten-containing alloys, the atomic mobility is expected to be low so that the stress due to non-energetic growth kinetics is tensile and weakly dependent on the growth rate. The observed incremental stress in Fig. 2 is the sum of this tensile stress from non-energetic growth with compressive stress from the energetic particle bombardment. The observed growth rate dependence is attributed primarily to the energetic component.

However, for elemental vanadium, the atomic mobility appears to be sufficient to allow the non-energetic compressive stress generating process to be active. The tendency for the stress to be less tensile at lower growth rates is consistent with what is seen in non-energetic growth if the atomic mobility is sufficiently high. Note that the mechanism for less tensile stress at low growth rates in elemental vanadium is fundamentally different from the trend towards more compressive stress with higher tungsten content. It is believed to be a result of the insertion of adatoms into the grain boundaries, whereas higher tungsten content enhances the energetic effects of atomic peening.

Finally, the role of the grain size and grain growth in stress evolution must also be considered. Most importantly, the slope of the measured stress-thickness was observed to change very slowly with thickness when the incremental stress was determined (i.e., at the largest measured thickness values). This suggests that the grain size, either at the surface or in the bulk of the film, is not changing significantly with the thickness at this point. The lack of grain growth is further supported by the fact that the XRD measurements of the crystallite size was the same (within error) for measurements made at 200 and 400 nm film thickness under the same conditions. Furthermore, since the measured size is similar for all the compositions and growth rates, differences in the magnitude of the grain size cannot be used to explain the observed difference in the incremental stress. Therefore, microstructural evolution is not expected to contribute significantly to the stress.

The above analysis suggests that energetic particle effects, enhanced by the higher content of tungsten, appears to be the most significant influence on the observed growth stress changes. This explains both the tendency to become more compressive with more tungsten for the same growth rate and to become more compressive with higher growth rate for most of the compositions. The difference in the behavior of elemental vanadium is attributed to stress generation by non-energetic growth processes.

6. Conclusion

Using a model solid solution vanadium-tungsten alloy, the changes in growth stress and grain size were characterized as a function of growth rate and composition. The crystallite size was relatively invariant for each alloy across the range of growth rates studied. As tungsten was added to the vanadium solution, the incremental growth stress trended towards a more compressive stress state, which can be explained by enhancement of the atomic peening effect by the addition of tungsten. The effect of the growth rate on stress in the alloys is consistent with mechanisms proposed for single element thin films deposited by sputtering [26], e.g., Co, Cr, Mo and W. Future work is directed towards quantitative modeling to relate the kinetic parameters that control stress in binary alloys to the parameters that control stress in the individual component systems.

CRediT authorship contribution statement

Tong Su: Experiments, Characterization, Software, Validation, Formal analysis, Investigation, Data curation, Writing original draft,

Writing review & editing, Visualization.

Jarod Robinson: Experiments.

Gregory B. Thompson: Writing review & editing, Methodology.

Eric Chason: Conceptualization, Methodology, Resources, Writing original draft, Writing review & editing, Visualization, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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