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The effect of kinetics on intrinsic stress generation and evolution in sputter-deposited films at conditions of high atomic mobility

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ABSTRACT

Vapor-based metal film growth at conditions that promote high atomic mobility is typically accompanied by compressive stress formation after completion of island coalescence, while an apparent stress relaxation is observed upon deposition interruption. Despite numerous experimental studies confirming these trends, the way by which growth kinetics affect postcoalescence stress magnitude and evolution is not well understood, in particular, for sputter-deposited films. In this work, we study in situ and in real-time stress evolution during sputter-deposition of Ag and Cu films on amorphous carbon. In order to probe different conditions with respect to growth kinetics, we vary the deposition rate $F$ from 0.015 to 1.27 nm/s, and the substrate temperature $T_S$ from 298 to 413 K. We find a general trend toward smaller compressive stress magnitudes with increasing $T_S$ for both film/substrate systems. The stress-dependence on $F$ is more complex: (i) for Ag, smaller compressive stress is observed when increasing $F$; (ii) while for Cu, a nonmonotonic evolution with $F$ is seen, with a compressive stress maximum for $F = 0.102$ nm/s. Studies of postdeposition stress evolution show the occurrence of a tensile rise that becomes less pronounced with increasing $T_S$ and decreasing $F$, whereas a faster tensile rise is seen by increasing $F$ and $T_S$. We critically discuss these results in view of ex situ obtained film morphology which show that deposition-parameter-induced changes in film grain size and surface roughness are intimately linked with the stress evolution.

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I. INTRODUCTION

The evolution of stress—with respect to its type and magnitude—in vapor-deposited films is closely linked with the various film growth stages.1–4 Isolated islands that form initially on the substrate surface exhibit a smaller-than-equilibrium lattice parameter due to the Laplace pressure they are subjected to.5 An increase of the island size with continued deposition causes a reduction of the Laplace pressure, while the islands become less mobile and less prone to reshape. As a result, their lattice cannot expand following the decrease of the Laplace pressure, and compressive stress emerges. Further vapor deposition causes islands to impinge on each other, which initiates the process of coalescence.1,5,6 The energy of the coalescing island cluster is minimized by creating a grain boundary (GB), leading to island reshaping and tensile straining of the lattice.6

As the deposited layer approaches the point at which it becomes continuous, the stress evolution depends strongly on atomic mobility. For conditions that yield low mobility, new segments of GBs that form at triple junctions (i.e., the intersection of surface and GB) induce tensile stress.10,11 Moreover, energetic particle bombardment of the growing layer (as, e.g., during sputter-deposition) may lead to point-defect generation, hydrostatic lattice expansion, and compressive stress.12,13 The net effect of these two independently operating processes is a steady-state stress ($\sigma_{SS}$), the sign of which (i.e., compressive or tensile) is determined by the dominant mechanism.13,14
At high-mobility conditions, compressive stress is commonly observed in the continuous film formation regime, with its origin still being a matter of debate. In the most widely accepted explanation, Chason et al.\(^ {15,16}\) have attributed the compressive stress to adatom insertion into GBs, which are areas with a lower concentration of mobile atoms compared to the surface of the growing film. The effectiveness of this mechanism, and thereby the magnitude of the generated compressive stress, depends on film topography (i.e., terrace length) and adatom diffusivity.\(^ {16,17}\) The model proposed by Chason et al. also includes the effect of grain growth,\(^ {18}\) which causes removal of GBs and tensile stress generation.\(^ {19}\) In the case of negligible grain growth and effectively unimpeded insertion of adatoms into the GBs, a compressive $\sigma_{TS}$ is reached.

Since GBs seemingly play a key role for stress generation,\(^ {18,20,21}\) researchers have been striving to establish and understand stress-kinetics relations—kinetics is primarily governed by the substrate temperature $T_S$ and the film growth rate $F$—using films with well-controlled grain sizes (i.e., GB length) that are independent of film thickness and deposition conditions. It has been observed that stress becomes less compressive (more tensile) with increasing $F$ in the case of deposition conditions in which energetic bombardment has no appreciable effect on film growth.\(^ {22,23}\) However, this trend has been shown to reverse for sputter-deposited Cu films grown at conditions for which interactions between energetic particles and films have to be considered as well.\(^ {24}\) Studies on the effects of deposition temperature have mostly focused on the change from low- to high-mobility growth regimes showing a transition from compressive-tensile-compressive (CTC) stress vs film thickness evolution with increasing $T_S$.\(^ {25,26}\)

The studies highlighted above have provided data for benchmarking the predictions of theoretical models which describe stress evolution in films grown using thermalized vapor fluxes.\(^ {27,28}\) However, in realistic sputter-deposition scenarios grain sizes change as a function of growth conditions, while the interaction of hyperthermal species with the film surface and near-surface layers may modify stress evolution.\(^ {29,30}\) Hence, systematic investigations on the effect of kinetics on the stress generation and evolution at application-relevant sputter-deposition conditions are required. Such investigations should also address postdeposition stress evolution, as a tensile stress rise is observed at conditions of high atomic mobility, but the way by which the magnitude and time scale of this rise are affected by kinetics is not well understood. In one study, Flötotto et al.\(^ {31}\) found little influence of $F$ on the postdeposition tensile rise magnitude (for fixed grain size) and a weaker but faster tensile rise with increasing grain size. Moreover, Leib and Thompson\(^ {32}\) reported a linear dependence of the tensile stress rise on the compressive stress accumulated in the film before interruption but no $T_S$-dependence of the initial tensile rise.

Here, we study—using in situ wafer curvature measurements and ex situ morphology characterization tools—stress evolution during and after magnetron-sputter-deposition of Ag and Cu films on amorphous carbon (a-C) substrates. To systematically probe the effect of kinetics at conditions of high atomic mobility, we vary $T_S$ in the range 298–413 K, while $F$ is altered from 0.015 to 1.27 nm/s. In all cases, we observe CTC stress evolution as a function of film thickness and tensile stress rise after deposition interruption. We find that for both film/substrate systems the increase of $T_S$ leads to smaller compressive stress after continuous-layer formation and larger grain sizes (i.e., the GB length decreases). For Ag deposition over the entire probed $F$ range and for Cu deposition with $F > 0.102$ nm/s, smaller compressive stress is observed when increasing $F$. These trends are consistent with models that explain compressive stress generation as a result of adatom incorporation into GBs. However, smaller compressive stress is formed in Cu films when decreasing $F$ below 0.102 nm/s. We argue, based on chemical analysis, that adsorption of gas molecules from the background pressure (e.g., H₂O), impeding atomic surface-diffusion and incorporation into GBs, is the reason for this behavior. The postdeposition tensile stress rise becomes faster with increasing $T_S$ and $F$ and smaller in magnitude with increasing $T_S$ and decreasing $F$. We attribute the change in magnitude of the tensile rise to the decreasing grain size.

II. FILM GROWTH AND CHARACTERIZATION

Films were sputter-deposited using Ar (purity 99.999%) plasma discharges at a working pressure of $p = 0.25$ Pa in a multishot high-vacuum chamber (base pressure $\sim 8 \times 10^{-6}$ Pa). Si wafers, covered with native oxide, were used as substrates, with substrate thickness $h_i = 100 \pm 2$ nm for in situ stress measurements and $h_i = 675 \pm 20$ nm for ex situ characterization. The magnetron sources were equipped with Ag (purity 99.99%), Cu (purity 99.99%), and graphite (purity 99.99%) targets (diameter 7.62 cm), installed in a conical configuration, with a target-to-substrate-normal angle of 25° and a target-to-substrate distance of 180 mm. A 6.5 nm thick a-C diffusion barrier layer was grown in situ, prior to Ag and Cu film deposition, by sputtering the graphite target at a power of 150 W (i.e., growth rate $F = 0.01$ nm/s). For Ag and Cu films, growth rates $F$ in the range 0.025–1.27 nm/s (Ag) and 0.015–0.535 nm/s (Cu) were employed, by changing the power applied to the respective magnetrons from 5 to 300 W. Moreover, the substrate temperatures $T_S$ for Ag and Cu deposition were varied in the respective ranges 298–378 K and 298–413 K. $T_S$ was set by a resistive heater 1 h before deposition start and held constant during and after deposition. Vacuum-compatible temperature indicators (NiCrNi Corp.) that change their color irreversibly upon reaching specific $T_S$ (accuracy $\pm 2$ K for $T_S < 410$ K and $\pm 4$ K for $T_S \geq 410$ K) were used to confirm the temperature settings. Ex situ x-ray reflectivity measurements [XRD 3000 Seifert diffractometer, line focus Cu source, Ge (220) monochromator selecting K$_{Cu}$, Cu radiation] were performed to determine the film thickness and $F$, as a function of applied power and $T_S$; varying $T_S$ had only minor effects on $F$ (variation $\leq 4\%$ in the used $T_S$ range). Immediately after metal film growth, a 6.5 nm thick a-C capping layer was deposited on samples that were used for ex situ characterization, in order to minimize the interaction between film and atmospheric ambient and suppress postdeposition changes of the film morphology. More details on the ex situ film analysis are provided later in the present section.

The evolution of the substrate curvature change $\Delta \kappa$ during deposition on Si(100) substrates was monitored in situ and in real-time with a multibeam optical stress sensor (MOSs, k-Space Associates).\(^ {32,33}\) The stress-film thickness product $\sigma \times h_f$ was then calculated from $\Delta(\sigma \times h_f) = \frac{1}{2} Y_s h_f^2 \Delta \kappa$, where $Y_s = 180.5$ GPa is the Si(100)
substrate biaxial modulus. Being subject to the flux of arriving vapor atoms, the film/substrate system heats up during deposition, leading to expansion of the crystal lattices according to their thermal expansion coefficients $\alpha_i$. Once the deposition is completed, the sample temperature returns to the set $T_S$ value and thermal stress $\sigma_{th}$ builds up due to the mismatch of film and substrate $\Delta \sigma_{th}$ according to $\sigma_{th} = Y_{Ag,Cu} \Delta \sigma_{th} \Delta T_S$, where $Y_{Ag,Cu}$ are the biaxial moduli of Ag (130 GPa) and Cu (200 GPa), respectively, and $\Delta T_S$ is the deposition-induced temperature increase. The heating of the substrate due to the deposition flux and its cooling after deposition stop were measured with thermocouples placed close to the substrate on the substrate holder for depositions at $T_S = 298$ and 413 K; we find that $\Delta T_S$ is independent of $T_S$. The evolution of temperature vs time $T_S(t)$ after deposition can be described with an exponential decay function $T_S(t) = T_S + \Delta T_S \times \exp(-t/\tau)$, whereby all sets are fitted using the decay time constant $t = 2470 \pm 3$ s. The stress evolution after growth interrupt was corrected by subtracting the time-dependent thermal stress $\sigma_{th}(t) = \sigma_{th} \times (1 - \exp(-t/\tau))$; the values of $\Delta T_S$, $\sigma_{th}$, and $\sigma_{th} \times h_f$ for various values of $F$ are listed in Table S1 in the supplementary material.

All results presented in Secs. III A–III D are obtained by monitoring $\Delta x$ in a four-stage process, see Fig. 1 that shows data recorded during the growth of a Cu film at $T_S = 298$ K and $F = 0.276$ nm/s: (a) Deposition of a-C layer ($h_{f,a-C} = 7$ nm); (b) presputtering of metal target; (c) deposition of metal film; and (d) postdeposition stress monitoring. $\Delta x$—and by extension $\sigma \times h_f$—shows a first maximum at $h_{f,a-C} = 0.5$ nm [marked by an arrow in Fig. 1(a)], indicating that a continuous a-C film is formed early during deposition. During stage (b), $\Delta x$ does not change, i.e., relaxation in the a-C film does not have an appreciable effect in the metal-layer stress evolution. Moreover, we found that stress-evolution during the deposition of a-C has not been influenced by $T_S$ (data not shown here). For comparison purposes, all results corresponding to stages (c) and (d) in Fig. 1 have been shifted in Figs. 2, 3, 7, and 9 to start at the same $\sigma \times h_f = 0$ N/m value.

In situ characterization was complemented by ex situ imaging of the surface morphology of films grown at various $T_S$ and $F$ with a Nanoscope III Multimode Digital Instruments atomic force microscope (AFM) operating in tapping mode. Observations were made at film thicknesses of 65 nm and 105 nm for Cu/a-C and Ag/a-C, respectively. The acquired images can be found in the supplementary material (Fig. S1) and were analyzed with the Gwyddion software package. Statistical quantities that are used to describe thin film morphology include the root-mean square surface roughness $w = \langle (h(x) - \bar{h})^2 \rangle$ and height–height correlation function $g(r) = \langle (h(x) - \bar{h}) \cdot (h(x + r) - \bar{h}) \rangle$, where $h(x)$ is the surface height at a position $x = (x, y)$ on the surface, $\bar{h}$ is the average surface height, $\langle \ldots \rangle$ denotes the statistical average over the entire surface, and $r$ is a displacement vector.

Grain size and orientation were analyzed via electron backscatter diffraction (EBSD) with a FEI-Helios Nanolab G3 Dual Beam microscope operated at 15 kV with 11 nA and a step size of 25 nm. The acquired maps were treated with the OIM Analysis™ software, using Neighbor Confidence Index Correlation (minimum Confidence Index 0.05) and Grain Dilatation cleanup (tolerance angle 2°, minimum grain size 3 points).

X-ray photoelectron spectroscopy (XPS) was used to determine the chemical composition of Ag and Cu films. The measurements were carried out in a Kratos AXIS Ultra DLD UHV system (base pressure $5 \times 10^{-8}$ Pa), equipped with a monochromatic Al-Kα x-ray beam, a hemispherical sector analyzer, and a multichannel detector. The pass energy was 20 eV. Surface-cleaning and depth-etching were performed using a 4 keV Ar+ ion-beam, and the shift of the Ar-2p peak was used for correcting charge-induced shifts of the binding energies owing to Ar+ ions. Elemental analysis was performed with the Kratos Vision software and its sensitivity factor database.

### III. RESULTS AND DISCUSSION

We start the presentation and discussion of the results with regard to in situ stress evolution before formation of a continuous layer (Sec. III A). Data for in situ stress after continuous film formation are presented in Sec. III B and discussed in light of the ex situ film morphological characterization in Sec. III C. The final part, Sec. III D, is devoted to description and discussion of in situ data concerning postdeposition stress evolution. In order to establish a unified and material-independent picture of the effect of process parameters on the stress generation and evolution during growth of Ag and Cu films, we rescale all deposition temperatures using the homologous temperature $T_h = T_S/T_m$, where $T_m$ is the melting point of Ag or Cu given in $K$.

#### A. Stress evolution before continuous film formation

**Figure 2** presents the evolution of $\sigma \times h_f$ vs film thickness $h_f$ during growth of Ag films on a-C, for $T_h$ between 0.24 and 0.31...
(i.e., $T_h = 298 - 378 \, \text{K}$) and for three different deposition rates: (a) $F = 0.033 \, \text{nm/s}$, (b) $F = 0.149 \, \text{nm/s}$, and (c) $F = 1.27 \, \text{nm/s}$. For all films, a pronounced CTC stress evolution is observed. With increasing $T_h$, the magnitude of the initially formed (i.e., for $h_f = 0 - 10 \, \text{nm}$) incremental compressive stress ($\sigma_{\text{init}}$) increases, and the magnitude of the incremental tensile stress ($\sigma_{\text{tensile}}$) that appears thereafter (i.e., for $h_f = 10 - 65 \, \text{nm}$) decreases. For instance, $\sigma_{\text{init}}$ evolves from $\sim -21$ to $\sim -74 \, \text{MPa}$, and $\sigma_{\text{tensile}}$ varies from $\sim 150$ to $\sim 19 \, \text{MPa}$ when increasing $T_h$ from 0.25 to 0.31 for $F = 0.033 \, \text{nm/s}$ [see Fig. 2(a)]. Additionally, the onset of the tensile rise and the subsequent tensile-to-compressive stress peak are shifted to higher $h_f$ values, e.g., the thickness of the tensile-to-compressive stress peak in Fig. 2(a) changes from 12 to 65 nm at $F = 0.033 \, \text{nm/s}$, when increasing $T_h$ from 0.24 to 0.31. Conversely, increasing $F$ results in a smaller $\sigma_{\text{init}}$ [see, e.g., change from $\sim -74$ to $\sim -44 \, \text{MPa}$ when increasing $F$ from 0.033 to 1.27 nm/s for $T_h = 0.31$ in Figs. 2(a) and 2(c)], and in a smaller $h_f$ value at which the tensile-to-compressive stress transition occurs [see, e.g., the tensile-to-compressive peak position shift from 65 to 30 nm, for $T_h = 0.31$ when increasing $F$ from 0.033 to 1.27 nm/s in Figs. 2(a) and 2(c)]. $\sigma_{\text{init}}$ and $\sigma_{\text{tensile}}$ Values for representative samples are listed in Table S2 in the supporting material.

The CTC evolution of $\sigma \times h_f$ vs film thickness $h_f$ curves during growth of Cu thin films on a-C is presented in Fig. 3 for $T_h = 0.22 - 0.30$ (i.e., $T_h = 298 - 413 \, \text{K}$) and (a) $F = 0.015 \, \text{nm/s}$, (b) $F = 0.102 \, \text{nm/s}$, and (c) $F = 0.535 \, \text{nm/s}$. In qualitative agreement with the results for Ag in Fig. 2, increasing $T_h$ (decreasing $F$) leads to larger $\sigma_{\text{init}}$ Values [see also Table S2 in the supplementary material], while the tensile rise onset is shifted to higher $h_f$ values, accompanied by an increase of the tensile-to-compressive stress transition thickness. For instance, for $F = 0.015 \, \text{nm/s}$, the tensile-to-compressive transition thickness increases from 9 to 33 nm when increasing $T_h$ from 0.22 to 0.30 [Fig. 3(a)], and decreases from 33 to 17 nm when increasing $F$ from 0.015 to 0.535 nm/s for $T_h = 0.30$ [compare Figs. 3(a) and 3(c)].

The magnitude of $\sigma_{\text{init}}$ depends on the island size at which the Laplace-pressure-reduced lattice constant is locked into the island, which may increase with increasing $T_h$, yielding a smaller compressive stress. In addition, $\sigma_{\text{init}}$ scales with the size of islands formed during the early film growth stages. The latter is determined by the dynamic competition between island nucleation and growth, whereby an increase of $T_h$ and/or a decrease of $F$ enhances the effective adatom diffusivity on the substrate surface and favors formation of fewer and larger islands. Our measurements show larger values of $\sigma_{\text{init}}$ for both Ag (Fig. 2) and Cu (Fig. 3) with increasing (decreasing) $T_h(F)$, which suggests that in our experiments the mechanical load is transferred to the substrate by larger islands and that the change of the island size at which the lattice parameter becomes locked into the island is not very pronounced.

The subsequent tensile stress is caused by island coalescence and, as such, the stress magnitude is proportional to the number of island-impingement occurrences. The latter increases with increasing island number density, which can explain the larger values of $\sigma_{\text{init}}$ with decreasing $T_h$. This $\sigma_{\text{init}}$ vs $T_h$ trend has also been found by Seel et al.,8 who showed, using a model based on finite element methods, that the increasing atomic mobility at the island-substrate interface with increasing $T_h$ leads to island-sliding and reduced tensile stress formation. Figures 2 and 3 also show that the tensile stress regime extends over a larger $h_f$ range with increasing $T_h$. This is because higher temperatures promote coalescence of islands with increasingly larger sizes, so that cluster reshaping and coarsening is facilitated over longer periods of time during deposition.

The interplay among tensile stress from island-impingement and compressive stress from insertion of atoms into GBs leads to a tensile-to-compressive peak, which has been shown to coincide with formation of a continuous film. We find a trend toward a larger continuous film formation thickness with increasing $T_h$ and decreasing $F$ for both Ag and Cu films, which is a direct consequence of the larger island size and promoted coalescence, i.e., the films exhibit a more pronounced three-dimensional growth morphology, as discussed in detail in our previous work.

**B. Stress evolution after continuous film formation**

All curves presented in Fig. 2 exhibit a constant $\sigma \times h_f$ vs $h_f$ slope for $h_f \approx 70 \, \text{nm}$, indicating that a compressive steady-state...
stress ($\sigma_{SS}$) regime is reached for Ag films. Figure 4(a) plots $\sigma_{SS}$ vs $F$ for $T_h = 0.24 - 0.31$ (i.e., $T_s = 298 - 378$ K). At $T_h = 0.24$, the largest compressive $\sigma_{SS} \approx 44$ MPa is obtained at $F = 0.025$ nm/s, while increasing $F$ causes $\sigma_{SS}$ to decrease and reach a plateau value ($\approx 25$ MPa) for $F > 0.05$ nm/s. For $T_h = 0.27$, the compressive $\sigma_{SS}$ values are smaller ($\approx 20$ MPa) than those for $T_h = 0.24$ with less pronounced dependence on $F$; further increase of $T_h$ to 0.31 does not decrease $\sigma_{SS}$ further, and no $F$-dependence of $\sigma_{SS}$ is observed.

In contrast to Ag, no compressive steady-state stress regime is seen during Cu film growth in Fig. 3, which is consistent with previous reports. Hence, in Fig. 4(b), we present the compressive incremental stress $\sigma_{inc}$—calculated from the slope of the $\sigma \times h_f$ vs $h_f$ curves in the film thickness range $h_f = 50 - 65$ nm—as a function of the deposition conditions. The $\sigma_{inc}$ vs $F$ evolution at $T_h = 0.22$ (i.e., $T_s = 298$ K) is nonmonotonic; the compressive stress initially increases from $\approx 85$ MPa, reaching its maximum value ($\approx 320$ MPa) at $F = 0.102$ nm/s, above which it decreases to $\approx 220$ MPa for $F = 0.535$ nm/s. With increasing $T_h$, the dependency of $\sigma_{inc}$ on $F$ becomes less pronounced, and at the highest $T_h = 0.30$ (i.e., $T_s = 413$ K), $\sigma_{inc}$ is almost independent of $F$.

A closer analysis of $\sigma_{inc}$ vs $F$ for Ag and Cu films deposited at various $T_h$ and $F$ values (images are provided in the supplementary material), and the respective height–height correlation functions $g(r)$ were calculated, as explained in Sec. II. Due to the nonequilibrium character of vapor-based growth, deposited films have self-affine surfaces, for which $g(r)$ converges to steady-state value $g_{SS}(r)$ that is related to the root-mean square surface roughness $w$ via the expression $g_{SS}(r) = \sqrt{\frac{\pi}{2}} w$. Figure 5 plots $g^{1/2}(r)$ vs $r$ for Cu [Fig. 5(a)] and Ag [Fig. 5(b)] films grown at various $F$ and $T_h$ values. We see that $g_{SS}(r)$ (indicated by horizontal arrows) and thereby $w$ increases with increasing $T_h$ [e.g., for Ag deposited with $F = 0.033$ nm/s, $w_{Ag}(T_h = 0.24) = 3.2$ nm, and $w_{Ag}(T_h = 0.31) = 14.5$ nm] and decreasing $F$ for both film/substrate systems [e.g., for $F = 1.27$ nm/s, $w_{Ag}(T_h = 0.24) = 1.8$ nm]. We also notice that for comparable values of $T_h$ and $F$, $w_{Cu} < w_{Ag}$, e.g., for $T_h = 0.30$ and $F = 0.015$ nm/s, $w_{Cu} = 7.6$ nm, compared to $w_{Ag} = 14.5$ nm for $T_h = 0.31$ and $F = 0.033$ nm/s.

A closer analysis of $g^{1/2}(r)$ vs $r$ curves reveals local minima in the $30 - 350$ nm $r$-range. The position of the first local minimum (indicated by vertical arrows in Fig. 5) corresponds to the average mound separation distance $d$ on the film surface, which provides information on the lateral surface corrugation that is not included in the root-mean square roughness $w$. For Ag films [see Fig. 5(b)] and Cu films deposited at $T_h = 0.30$ [see Fig. 5(a)], $d$ follows the trend of $w$ (i.e., increases with increasing $T_h$ and decreases with increasing $F$).
decreasing $F$), which means that the surface is characterized by laterally larger mounds separated by deeper trenches. This trend does not hold for Cu deposited at $T_h = 0.22$, where the highest value of $d = 53$ nm is found for $F = 0.015$ nm/s, but no substantial change is seen (28 and 32 nm) when increasing $F$ from 0.102 to 0.535 nm/s.

EBSD maps indicating the grain size and orientation (color code presented by inverse pole figures) of 105 nm thick Ag films are presented in Fig. 6. For deposition at $T_h = 0.24$ (i.e., $T_S = 298$ K), the average grain size decreases slightly from $\sim 130$ to $\sim 120$ nm when increasing $F$ from 0.033 to 1.27 nm/s, which stems from a smaller population of grains with sizes larger than 200 nm for high $F$. In addition, a strong preferred (111) orientation of the grains can be found for deposition for $F = 1.27$ nm/s. For higher $T_h = 0.31$ (i.e., $T_S = 378$ K), the grain size of Ag films deposited at 0.033 nm/s increases to $\sim 200$ nm, while the grain size remains at $\sim 120$ nm for $F = 1.27$ nm/s. Independent of $F$, no preferred crystal orientation can be detected for $T_h = 0.31$. EBSD measurements for Cu films were not successful, as the lateral grain size approaches the resolution of the instrument (25 nm). This is consistent with the low value of lateral mound separation $d$ ($d \leq 53$ nm) extracted from AFM for Cu films deposited at $T_h = 0.22$ [i.e., $T_S = 298$ K, see Fig. 5(a)].

In the literature, the following mechanisms have been suggested to explain stress evolution of continuous films: (i) Formation of new GB segments at triple junctions that causes tensile stress,\textsuperscript{29,31} (ii) grain growth that may occur at high-mobility conditions and leads to tensile stress,\textsuperscript{18,27} (iii) adatom-insertion into GBs at high-mobility conditions driven by chemical potential gradient between surface and GB resulting in compressive stress;\textsuperscript{15,16} and (iv) incorporation of point defects in the grains and/or in GBs, due to energetic bombardment (i.e., “atomic peening”), yielding compressive stress.\textsuperscript{12} The final magnitude and type of film stress is the result of the superposition of these stress contributions and their respective dependence on the film morphology. With the microstructural information provided earlier in the present section, we can now discuss our experimental results in view of the interplay and interdependence of these stress-contributing processes.

The results for Ag films presented in Fig. 2 show that compressive stress develops after continuous-layer formation, which reaches a compressive steady-state. For depositions at $T_h = 0.24$, grain size does not change significantly as a function of $F$, while $\sigma_{SS}$ becomes less compressive with increasing $F$ before reaching a plateau. This trend is consistent with the model proposed by Chason et al.\textsuperscript{9} The model explains the compressive $\sigma_{SS}$ decrease in light of a reduced number of adatoms that can reach the GBs, owing to the higher density of adatoms and shorter adatom diffusion length with increasing $F$. A similar trend is also observed for Cu films at $T_h = 0.22$ for $F$ values larger than 0.102 nm/s [see Fig. 4(b)]. This is opposite to data by Kaub et al.,\textsuperscript{24} who reported an increase in compressive $\sigma_{SS}$ values for Cu grown at $T_h = 0.22$ for $F$ increasing in the range 1.2 – 2.4 nm/s. This behavior has been attributed to bombardment-induced point-defect formation and trapping due to the faster progressing growth front at larger $F$ values. Our largest $F = 0.535$ nm/s lies well outside the range in the work of Kaub et al.,\textsuperscript{24} which indicates that defect-induced stress formation is not a dominant process at our growth conditions. In contrast to low-mobility metals (as, e.g., Mo deposited at $T_h \sim 0.1$),\textsuperscript{24} the effect of energetic particle bombardment is less significant for high-mobility systems, whereby high values of $T_h$ promote defect annihilation.

Increasing $T_h$ from 0.24 to 0.31 causes the magnitude of compressive stress in Ag to decrease, most notably for $F \leq 0.033$ nm/s [see Fig. 4(a)], while the grain size increases from $\sim 130$ to $\sim 200$ nm (see Fig. 6). In a film with larger grains, fewer new GB segments form on the surface, which results in smaller tensile stress. Concurrently, fewer GBs exist into which atoms can be inserted yielding a smaller compressive stress. Figure 5(b) shows that $w$ increases with $T_h$, which has been suggested to inhibit the formation of new GB segments on the surface.\textsuperscript{25} Hence, we attribute the decrease of the magnitude of $\sigma_{SS}$ as a function of $T_h$ to reduction of the compressive stress component caused by elimination of GBs.

At $T_h = 0.31$, $\sigma_{SS}$ reaches its smallest value of $\sim 20$ MPa and it becomes practically independent of $F$ for Ag films, despite changes in the film microstructure (the grain size decreases from $\sim 200$ nm for $F = 0.033$ nm/s to $\sim 120$ nm for $F = 1.27$ nm/s, and the roughness decreases from 14.9 to 4.6 nm in the same $F$ range). This indicates that adatom surface diffusion and incorporation into GBs is not the limiting factor that determines the magnitude of $\sigma_{SS}$ at these high temperature conditions. A similar behavior is also observed for Cu films at $T_h = 0.30$ [see Fig. 4(b)].

Comparison of Figs. 4(a) and 4(b) reveals a noticeable difference between the stress evolutions of Ag and Cu films at low deposition rates: contrary to Ag film growth, a smaller compressive stress is formed when decreasing $F$ from 0.102 to 0.015 nm/s for $T_h = 0.22$. This is also reflected in the $\sigma \times h_f$ vs $h_f$ curves for $F = 0.015$ nm/s, which show that the incremental stress tends to
turn tensile for $h_I = 65$ nm (see Fig. 3). This is a known behavior for films that exhibit grain growth (e.g., Ni in Ref. 53) or under-dense morphology (e.g., Cu during deposition at high Ar pressure > 0.5 Pa). XRR measurements show that Cu films deposited at $F = 0.015$ nm/s have a mass density equal to the bulk value (8.92 g/cm$^3$), i.e., the emergence of tensile stress cannot be ascribed to the formation of under-dense films. Concurrently, the average mound separation distance increases from 28 to 53 nm for $T_h = 0.22$ when changing $F$ from 0.102 to 0.015 nm/s [compare data in Fig. 5(a)], suggesting that the grain size increases with decreasing $F$. The thickness of the tensile-to-compressive transition in Fig. 3 can be used as an approximation for the grain size upon continuous film formation. The transition thickness marginally increases from 8 to 9 nm, when decreasing $F$ from 0.102 to 0.015 nm/s, indicating similar grain sizes at continuity. Hence, the larger grain size indicated for low $F$ from AFM measurements for 65 nm thick films may be the result of grain growth at larger thicknesses.

Yu and Thompson reported that incorporation of impurities, including O$_2$, in Ni films is another factor that affects the steady-state stress magnitude.$^{54}$ They performed experiments at various oxygen partial pressures $p_O$ in the deposition chamber and showed that an increase of $p_O$ leads to more tensile stress formation, due to reduced adatom-GB interaction and thus less pronounced adatom insertion into GBs. In our experiments, we do not deliberately introduce impurity species during film growth. However, depositions are performed in a high-vacuum chamber, which has a residual atmosphere at a base pressure of $\sim 8 \times 10^{-6}$ Pa consisting mainly of H$_2$O, toward which Cu has a high affinity.$^{55}$ In order to explore the relevance of impurities for the stress formation in our films, XPS measurements and depth-profile analysis were performed on capped Ag and Cu films; an overview of the results is presented in Table I, while x-ray photoelectron spectra are presented in the supplementary material. For the chemical analysis, O-1s and Cu-2p/Ag-3d binding energies were used, other elements were not detected. After surface cleaning (i.e., $\sim 1$ nm ion-beam etching), the O/Cu ratio in Cu films deposited at $F = 0.015$ nm/s is 0.246; this value decreases with increasing deposition rate and O/Cu = 0.091 for $F = 0.535$ nm/s. The relative oxygen concentration decreases with increasing etching depth but remains nonzero for all deposition rates (e.g., O/Cu = 0.014 for $F = 0.015$ nm/s after $\sim 7$ nm etching) and decreases with increasing values of $F$ (e.g., O/Cu = 0.006 for $F = 0.535$ nm/s) for constant etching depth. For comparable values of $F$, Ag films contain no oxygen after $\sim 5$ nm ion-beam etching, which is consistent with considerably weaker affinity Ag to background gaseous impurities.$^{56}$ Hence, the nonmonotonic behavior of $\tau_{\text{incr}}$ vs $F$ for Cu may also be explained by gaseous impurities incorporation in the Cu films from the background atmosphere in the deposition chamber. The increased propensity for impurity incorporation with decreasing deposition rate can be understood in light of the interplay between the monolayer (ML) formation times of impurities $\tau_{\text{ML}}^{\text{impurities}}$ and film $\tau_{\text{ML}}^{\text{film}}$, which is a measure for the impurity-incorporation in the film, decreases with increasing deposition rate. An increase of $T_h$ leads to smaller sticking coefficient of impurity species (i.e., $\tau_{\text{ML}}^{\text{impurities}}$ increases)$^{56}$ while adatoms have higher diffusivities.$^{44}$ This can explain the decrease of $\tau_{\text{incr}}$ with decreasing $F$ is less pronounced at $T_h = 0.25$ and that almost no variation is observed at $T_h = 0.30$ [see Fig. 4(b)].

Finally, we return to a comparison among the stress evolution during Ag and Cu film growth. With the information on microstructure, microchemistry, and surface morphology presented above, we can relate the larger stress values measured in Cu films compared to Ag films to smaller grain size (i.e., higher GB number density), smaller surface roughness, and higher affinity to gas molecules from the growth atmosphere. Moreover, the biaxial modulus of the bulk Cu $Y_{\text{Cu}} = 200$ GPa is larger than bulk silver $Y_{\text{Ag}} = 130$ GPa; consequently, a comparable strain level would result in $\sim 50\%$ larger stress in Cu compared to Ag films.

### D. Postdeposition stress evolution

Postdeposition stress measurements (see Fig. 7) show that all process parameters used in this work lead to a tensile stress rise when deposition is stopped. This is a typical behavior for films grown at conditions of high atomic mobility$^{49,50}$ and it is commonly referred to as “stress relaxation,” relative to the compressive stress observed after formation of a continuous film. Experiments on stress evolution before continuous-layer formation$^{51,52}$ have shown that additional processes, including adatom–surface interactions and defect annihilation, are operative after deposition flux is turned off. Therefore, in the following, we will not use the term relaxation when presenting and discussing the evolution of $\sigma \times h_I$ as a function of time $t$ after deposition stop. We remind the reader that $T_h$ was kept constant during postdeposition stress monitoring and note that thermal stress from heating of the substrate during deposition has been corrected, as explained in Sec. II.

Figures 7(a)–(c) present the postdeposition stress evolution of Ag films for deposition rates (a) 0.033 nm/s, (b) 0.149 nm/s, and (c) 1.27 nm/s and $T_h = 0.24$, 0.25, 0.27, and 0.31 (i.e., 298, 313, 338, 378 K), after correction for $\tau_{\text{incr}}$, as a function of time $t$. For all deposition rates, we find decreasing tensile stress rise with increasing values of $T_h$, e.g., for $F = 0.033$ nm/s, $\sigma \times h_I$ decreases from 3.0 to 1.0 N/m when increasing $T_h$ from 0.24 to 0.31 [see Fig. 7(a)]. Conversely, the postdeposition tensile rise increases with increasing deposition rate, e.g., $\sigma \times h_I = 6.2$ N/m for $F = 1.27$ nm/s and $T_h = 0.24$ [compare Figs. 7(a) and 7(c)].

### Table I. Ratio of oxygen-to-copper (O/Cu) and oxygen-to-silver (O/Ag) in sputter-deposited Cu and Ag films, as determined from x-ray photoemission spectroscopy. Spectra were acquired after etching with Ar$^+$ ions, which removes surface contamination and the amorphous carbon capping layer. For Cu films, data for various deposition rates $F$ are presented.

| $F$                  | O/Cu  | O/Ag  |
|---------------------|-------|-------|
| 0.015 nm/s          | 0.246 | 0.102 |
| 0.535 nm/s          | 0.091 | 0.014 |
| 0.109 nm/s          | 0.111 | 0.012 |

For Ag films, data for various deposition rates $F$ are presented.
FIG. 7. Evolution of stress-thickness $\sigma \times h_t$ with time $t$ after deposition completion of (a)–(c) Ag and (d)–(f) Cu thin films on amorphous carbon (a-C). For Ag, deposition rates (a) $0.033\text{ nm/s}$, (b) $0.149\text{ nm/s}$, and (c) $1.27\text{ nm/s}$ and homologous temperatures $T_h = 0.24$, $0.25$, $0.27$, and $0.31$ (i.e., $298, 313, 338, 378\text{ K}$) were used, for Cu, the deposition rates were (d) $0.015\text{ nm/s}$, (e) $0.102\text{ nm/s}$, and (f) $0.535\text{ nm/s}$ and $T_h = 0.22$, $0.25$, $0.27$, and $0.30$ (i.e., $298, 338, 378, 413\text{ K}$).

The same trend can be seen for the postdeposition stress evolution of Cu films for deposition rates (d) $0.015\text{ nm/s}$, (e) $0.102\text{ nm/s}$, and (f) $0.535\text{ nm/s}$ and a comparable $T_h$-range. The magnitude of the tensile stress rise decreases with increasing $T_h$, e.g., for $F = 0.015\text{ nm/s}$, $\sigma \times h_t$ decreases from 6.1 to 2.1 N/m when increasing $T_h$ from 0.25 to 0.30 [see Fig. 7(d)]. The magnitude of the tensile rise at $T_h = 0.22$ is smaller than at 0.25 for $F \leq 0.102\text{ nm/s}$ [see Figs. 7(d) and 7(e)]. With increasing deposition rate, the tensile rise becomes more pronounced for all values of $T_h$, e.g., $\sigma \times h_t$ increases from 6 to 11 N/m when increasing $F$ from 0.015 to 0.535 nm/s at $T_h = 0.25$ [compare Figs. 7(d) and 7(f)].

Figures 8(a) and 8(b) give an overview of the postdeposition tensile stress rise $\sigma_t$ for Ag and Cu, respectively, as extracted from Fig. 7 and highlights the larger values of $\sigma_t$ in Cu films compared to Ag films. For a more complete picture of the relation between $\sigma_t$ and $F$, we calculate the relative stress $\sigma_{rel} = \sigma_t/|\sigma_c|$, where $|\sigma_c|$ denotes the absolute value of $\sigma_c$. Figures 8(c) and 8(d) present $\sigma_{rel}$ for Ag and Cu, respectively, as a function of $T_h$ for different deposition rates $F$. For Ag, two regimes can be identified: (i) for $T_h < 0.27$, variations of $T_h$ and $F$ have little influence on $\sigma_{rel}$, with a tendency of higher $\sigma_{rel}$ values at higher values of $F$; (ii) for $T_h > 0.27$, $\sigma_{rel}$ increases with $T_h$ and decreases with increasing $F$, for given $T_h$. It is also in this region where $\sigma_{rel}$ exhibits values larger than 100% for the lowest deposition rate $F = 0.033\text{ nm/s}$, which shows that the measured tensile rise cannot purely be a relaxation process. A very similar trend can be found for the postdeposition stress evolution of Cu on a-C [Fig. 8(d)]. $\sigma_{rel}$ increases with increasing $T_h$, and for $T_h > 0.27$, $\sigma_{rel}$ exhibits values larger than 100% for $F = 0.015\text{ nm/s}$. The increase in $\sigma_{rel}$ with increasing $T_h$ becomes less pronounced at high $F$, while no $\sigma_{rel} > 100\%$ were measured for $F \geq 0.102\text{ nm/s}$ in the range $0.27 < T_h < 0.31$.

In the literature, the following mechanisms have been associated with the postdeposition stress evolution: (i) thermal stress $\sigma_{th}$; (ii) grain growth; (iii) out-diffusion of atoms from GBs, as adatom supersaturation on the film surface decreases after deposition stop;\(^{17,25}\) and (iv) reshaping of GB grooves.\(^{20,21,30,63}\) All these mechanisms lead to tensile stress and are active for conditions of high mobility. Our postdeposition stress data have been corrected for the contribution due to $\sigma_{th}$, and hence thermal stress does not account for the trends observed in Fig. 8. Table S2 in the supplementary material gives an overview of $\Delta T_h$, and $\sigma_{th}$ for Ag and Cu thin films, for selected $F$ values. We note that the knowledge of $\sigma_{th}$ is especially important for Ag films deposited at high values of $F$, where $\sigma_{th}$ is responsible for 50% of the originally observed tensile rise.

Flötotto et al.\(^{32}\) have shown in experiments for which the grain size is fixed using template layers that $F$ has little influence on the magnitude of the postdeposition tensile rise $\sigma_t$, while increasing grain size—i.e., decreasing GB number density—leads to a smaller tensile rise. This is in agreement with the decrease in $\sigma_t$ [Fig. 8(a)] we find with decreasing $F$ and increasing $T_h$ at $F = 0.033\text{ nm/s}$ for Ag, for which EBSD results (Fig. 6) show that the grain size increases. For $F = 1.27\text{ nm/s}$, the grain size is nearly constant with $T_h$, while we still see a decreasing $\sigma_t$ with increasing $T_h$; which may be related to the larger compressive stress formed during growth [e.g., compare $\sigma \times h_t = -2.9\text{ N/m}$ for $T_h = 0.24$ at deposition-interrupt vs $-1.0\text{ N/m}$ for $T_h = 0.31$ in Fig. 2(c)].
While the general trend toward larger tensile rise with increasing $F$ also holds for the postdeposition stress evolution of Cu depicted in Fig. 8(b), this can be solely explained by the associated change of grain size. In particular, for depositions at $T_h = 0.22$, for which the grain size does not vary between 0.102 and 0.535 nm/s, an $\sigma_{rel}$ increase from 150 to 250 MPa is observed. Possible incorporation of residual gas species into the GB, as suggested in Sec. III C, may effectively hinder the out-diffusion of adatoms from the GBs, once the deposition is stopped, and thus decrease the tensile stress developing postdeposition.

The stress evolution during and postdeposition of Ag and Cu films is dissimilar in magnitude and is affected by changes of $F_{th}$ and $F$ to a substantially different degree, which is related to morphological and microchemical changes in the films. Nevertheless, the comparison of $\sigma_{rel} = \sigma_t/\sigma_i$ for Ag and Cu presented in Figs. 7(c) and 7(d), respectively, shows a coherent picture, indicating that the key processes governing the stress evolution are the same.

Figure 9 presents the postdeposition tensile rise $\sigma_t \times h$ normalized to $\sigma_i \times h_i$, with the final value $\sigma_t$ presented in Fig. 8(b) for Cu films, showing an overall trend toward faster tensile rise with increasing $F$ and $T_h$. While a constant $(\sigma_t \times h_t)/(\sigma_i \times h_i)$ value is reached within 200 s for the highest $T_h = 0.30$ (i.e., $T_h = 413$ K) and $F = 0.535$ nm/s, only $\approx$85% is reached during the same time and for the same value of $F$ at $T_h = 0.22$ (i.e., $T_h = 298$ K), and merely $\approx$35% is reached for the lowest values $F = 0.015$ nm/s and $T_h = 0.22$. The $F$-dependence of the tensile rise kinetics for the Ag/a-C system shows the same trend, however, less pronounced (see Fig. S2 in the supplementary material).

Previous studies on postdeposition stress evolution attributed tensile rise within hundreds of seconds to surface-diffusion based processes, while grain growth is a slower process involving bulk rearrangements. Flötotto et al. found faster tensile rise with increasing $F$ and for larger grain sizes. They explained this trend in light of different shapes of GB grooves during and after deposition: (i) during deposition, the driving force for adatom diffusion to the GB is large, allowing adatoms to overcome the accumulation of steps close to GB (i.e., Zeno effect), which results in GB grooves that are more shallow than the equilibrium surface profile; (ii) while after deposition, the driving force for atom incorporation into GB decreases dramatically, the grooves deepen as adatoms attach to steps, and a shape closer to the equilibrium surface profile is attained. This postdeposition deepening of GB grooves releases compressive stress and is more pronounced for high $T_h$, which is consistent with our data.

Earlier in the present section, we have discussed the influence of incorporation of adsorbed gas molecules on the magnitude of the tensile rise of Cu films at low values of $T_h$ and $F$. The presence of such impurities in GBs may delay the out-diffusion of atoms postdeposition, resulting in the slow tensile rise seen for these deposition conditions. Moreover, during the discussion of the stress evolution in continuous Cu films deposited with $F = 0.015$ nm/s (see Sec. III C), we have identified grain growth as a potential explanation for the trend toward tensile stress formation in Cu films. If grain growth is active after deposition, its long time scale may account for the much slower tensile rise seen for $F = 0.015$ nm/s in Fig. 9. The above discussed mechanisms (i.e., grain growth and impurity incorporation) are seemingly not relevant for Ag film stress generation and evolution, as mentioned in Sec. III C, which may explain the overall faster postdeposition stress evolution kinetics as compared to Cu.

IV. SUMMARY AND OUTLOOK

We have studied real-time stress evolution during and after growth of Ag and Cu films that are deposited by magnetron sputtering on amorphous carbon substrates, while systematically changing substrate temperature between 298 and 413 K (corresponding to homologous temperatures $0.22 \rightarrow 0.31$) and deposition rate between 0.015 and 1.27 nm/s. All films show a compressive-tensile-compressive stress evolution as a function of film thickness. Increasing substrate temperature results in smaller compressive stress due to increasing grain size (i.e., lower grain-boundary length), and weaker deposition rate-dependence of the stress as adatom surface diffusion is promoted. With increasing deposition rate, less compressive stress is formed during the deposition of Ag. This is an indication that incorporation of atoms into grain boundaries is inhibited owing to the higher density of adatoms on the surface with increasing deposition rate, and consequently shorter diffusion lengths. This trend can also be seen for Cu films deposited with deposition rates $> 0.102$ nm/s, whereas for deposition rates $< 0.102$ nm/s, a tendency toward tensile stress formation is observed. From chemical microanalysis with x-ray photoelectron spectroscopy, we find increasing incorporation of oxygen from the background pressure with decreasing deposition rate, which inhibits atom incorporation into grain boundaries, and such that less compressive stress is formed. The grain-boundary number density determines the strength of the postdeposition
tensile rise, while high substrate temperature and deposition rate promote the kinetics of the postdeposition stress evolution. This trend is especially pronounced for Cu, where the presence of gas molecules seemingly slows the diffusion out of grain-boundaries.

SUPPLEMENTARY MATERIAL

See the supplementary material for (i) calculation of the deposition rate-dependent thermal stress that builds up after the deposition is stopped; (ii) incremental compressive stress during the initial growth stages of Ag and Cu film growth; (iii) atomic force microscopy images that were used to analyze the surface morphology of Ag and Cu films; (iv) normalized stress-thickness vs time curves for Ag thin films after deposition stop; and (v) x-ray photoelectron spectroscopy measurements of Ag and Cu thin films.

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