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Analysis of internal stress build-up during deposition of nanocrystalline Ni thin films using transmission electron microscopy

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Abstract

Ni thin films sputter-deposited at room temperature with varying Ar pressures were investigated with automated crystal orientation mapping in a transmission electron microscope to uncover the mechanisms controlling the internal stress build-up recorded in-situ during deposition. Large grains were found to induce behaviour similar to a stress-free nucleation layer. The measurements of grain size in most of the Ni thin films are in agreement with the island coalescence model. Low internal stress was observed at low Ar pressure and was explained by the presence of large grains. Relaxation of high internal stress was also noticed at the highest Ar pressure, which was attributed to a decrease of Σ3 twin boundary density due to a low deposition rate. The results provide insightful information to better understand the relationship between structural boundaries and the evolution of internal stress upon deposition of thin films.

Keywords: thin films, nickel, electron microscopy, internal stress, grain boundaries

1. Introduction

In the past, mechanical properties of nickel (Ni) thin films such as hydrogen-induced effects [1,2], the shift in deformation mechanisms in the Hall-Petch transition region [3] and internal stress induced during deposition [4] have been heavily investigated. The latter has been a matter of attention for close to 70 years now [4] and several mechanisms explaining the stress build-up during deposition have been suggested. Some parameters are still under heavy debate such as the role of adatoms on the strain field [5], excess vacancy (or interstitial) reduction [6,7] and the influence of dislocations [8]. On the other hand, some mechanisms are well-established like lattice mismatch, capillarity stress [9], or as will be applied in this work, the island coalescence model [10]. By approximation, the exterior of growing grains can be considered as rounded surfaces. During contact with adjacent islands, the system can lower its net free energy by reducing the high surface energy and by creating an interface with a relatively low interfacial energy [11]. This interface will take on the physical characteristics of an elastic crack. During the closing of this crack (this mechanism is commonly referred to as 'zipping'), the islands involved become elastically strained, contributing to the internal stress measurement. Furthermore, it is assumed that the zipping mechanism occurs on a relatively short time in comparison to the required time needed for

45 the islands' elastic relaxation to occur [11], thus inducing a permanent residual strain in the
46 film. If the film would be deposited uniformly, no internal stress would build up since the
47 film can grow in an energetically favourable condition, leading to a single-crystal structure. It
48 can occur that even though the initial deposition starts uniformly, islands can still start to
49 form from a certain thickness; the uniform layer is referred to as a nucleation layer.
50
51 On top of the uncertainty on the nucleation mechanism(s), another interesting aspect is the
52 resulting texture and grain size in thin films as it strongly influences the mechanical
53 behaviour of the material. For example, the Hall-Petch curve is depending on the combined
54 contribution of the orientation texture and grain boundary distribution [12]. X-ray diffraction
55 (XRD) [13] and electron backscatter diffraction (EBSD) are typically used for the examination
56 of these characteristics, although the first method does not provide local information and
57 the latter is limited to microscaled samples [12]. For nanoscaled films, in which the thickness
58 usually does not exceed ~100 nm, more advanced transmission electron microscopy (TEM)
59 techniques are required. In this research, nanocrystalline Ni thin films were sputter-
60 deposited on a Si substrate during which the stress was measured in-situ. The stress-
61 thickness behaviour observed during deposition is explained using microstructural
62 information extracted from automatic crystallographic orientation mapping in TEM (ACOM-
63 TEM) [14–16]. The results provide information on the relationship between structural
64 boundaries and the internal stress evolution in nanocrystalline Ni thin films. Similar results
65 can be achieved using transmission kikuchi diffraction (TKD) as shown by Kacher *et al*.[17,18]
66 who used this technique to display the formation of twin boundaries in annealed
67 nanocrystalline Ni thin films. They were able to prove through TKD the importance of the
68 formed boundaries for the equilibrium state of their samples and were able to resolve
69 features as small as 5 nm twins. The most suited transmission orientation mapping
70 technique depends on the requirements of the sample; TKD yields a higher angular
71 resolution due to the presence of the kikuchi lines while ACOM-TEM is more fitting for
72 beam-sensitive samples [19].
73 Knowledge on the microstructure can be useful to explain results observed at the
74 macroscale. For example, the discussed Ni thin films were also part of the work performed
75 by Delvaux *et al*. [20] who studied the hydriding behaviour of these films through water
76 electrolysis, showing a connection between the solubility of hydrogen and the film's average
77 grain size.
78

79 ## 2. Experimental details

80
81 A double-sided polished, 380 µm thick silicon (Si) film was used as a substrate. To prepare
82 the deposition procedure, the Si substrate was cleaned in a H_2SO_4/H_2O_2 bath (5:2 in volume)
83 for 10 minutes at 80°C. Next, in order to etch away intrinsic SiO_2, the substrate was
84 submerged for 20 seconds in 2% HF followed by rinsing for 10 minutes in ultra-pure H_2O
85 [20]. The substrate is then immediately transferred to an Orion 5 multi-target sputtering
86 chamber from AJA International where the substrate was coated with a 4 nm amorphous
87 TiO_2 layer by magnetron sputter-deposition from a TiO_2 target at a rate of 0.3 nm/min. The
88 base chamber pressure prior to deposition was 1.10^{-5} Pa. The thin amorphous TiO_2 coating
89 (shown in Figure A1 of the appendix) acts as a passivation layer to protect the Si from
90 oxidation and to ensure the adhesion of the Ni layer. On top of this TiO_2 layer, without

91 breaking the vacuum, nanocrystalline Ni thin films were magnetron sputter-deposited from
92 a pure Ni target (K.J. Lester Co.) at room temperature with an argon (Ar) plasma pressure
93 ranging between 0.27 Pa and 1.07 Pa until a thickness of 100-120 nm was reached. The
94 amount of scattering is proportional with the Ar pressure, thus the deposition rate and time
95 were proportionally decreased and increased respectively to compensate for this effect;
96 detailed values are provided in Table 1. The internal stress was measured in-situ using a
97 high-resolution curvature measurement setup aimed at the substrate in the deposition
98 chamber [17]. Using multiple laser beams directed towards the deposited film, the curvature
99 of the film can be quantified based on the position of the reflected beams [22].
100 After complete dissolution in a 50-50 $\%_{wt}$ HCl-HNO_3 acid solution, the final thickness of the
101 Ni layer was measured through Inductively Coupled Plasma – Optical Emission Spectrometry
102 (Agilent Technologies 5100 ICP-OES). The thickness resolution was of the scale of 0.1 nm as a
103 result of a chemical detection limit of ~0.01 ppm for Ni. This ICP-OES method was also
104 applied to investigate the purity of these Ni films; the concentration of contaminants
105 remained under 10 ppm. The purity levels of the Ni and TiO_2 targets were respectively
106 99.995% and 99.99%. Cross-sectional TEM samples were prepared from films deposited
107 under different conditions with a focused ion beam (FIB) using the "lift-out" procedure
108 (more details can be found in the appendix). To improve the reliability of the ACOM-TEM
109 results, careful thinning was performed with FIB to minimize the amount of overlapping
110 grains.
111
112 A Tecnai G2 microscope (FEG, 200 kV) from Thermo Fisher Scientific equipped with the
113 ASTAR system from Nanomegas was used to perform high resolution TEM (HR-TEM) and
114 ACOM-TEM [23]. Using electron precession with an angle of 0.4°, the dynamical scattering
115 effects were minimized which will facilitate the automatic indexation of the diffraction
116 patterns during post-treatment [24]; the resulting electron probe size was ~2 nm. Using the
117 software package from Nanomegas, pre-calculated electron diffraction patterns were
118 generated and cross-correlated to every obtained pattern [23]. The orientation resolution of
119 this database was 0.5°. Once all patterns are indexed, further processing is performed with
120 the Orientation Imaging Microscopy analysis software from EDAX Inc. This includes noise
121 reduction, statistical analysis and interface mapping. Non-indexed and mis-indexed points
122 were corrected using a clean-up method commonly used on electron backscatter data [25].
123

124 3. Results and Discussions

125

126 The stress-thickness data displaying the internal stress build-up are shown in Figure 1a.
127 Every curve can be divided in three sequential characteristic sections. Firstly, the stress
128 remains stable at a constant value, which could be explained by the formation of a single
129 crystal nucleation layer without or with just a few grain boundaries (GBs) or an amorphous
130 structure. For the Ni thin film prepared at 0.27 Pa, we observed that the stress remained
131 almost zero during the entire deposition. All other samples show a sudden increase,
132 indicating typically the end of the nucleation layer [26] and the start of the formation of GBs.
133 For 0.40 Pa and 0.53 Pa, the expected nucleation layer thickness is respectively 50 nm and
134 30 nm, as measured from the stress-thickness data in Figure 1a. For the samples deposited
135 with 0.67 Pa and 1.07 Pa, the nucleation layer ends at around 10 nm.
136

137 Secondly, the initial increasing slope can be used as an indication for the grain size based on
138 the 'zipping mechanism' as suggested by Nix and Clemens [10], where the tensile stress
139 increase is inversely proportional to the grain size. It needs to be restated here that every Ni
140 film was deposited at a different deposition rate, which can be proportional to the tensile
141 stress as was proven by Hearne *et al*. [27] and Kongstein *et al*. [28] for electrodepositing Ni
142 or Cu, respectively. However, since each film also had an Ar pressure in the deposition
143 chamber inversely proportional to the used deposition rate, comparable deposition
144 conditions are induced in all samples. Aside from 0.27 Pa, all samples show a similar slope,
145 thus the grain size is expected to be similar. At any point on the curve, the stress contributed
146 by the incremental layer of film deposited can be quantified by taking the derivative at that
147 thickness [29]; an incremental internal stress of ~1.1 GPa was estimated from the initial
148 slope. Finally, for the samples prepared at the highest Ar pressures, 0.67 Pa and 1.07 Pa, a
149 downward bending of the curve is seen. This effect together with all predictions described in
150 this section will be cross-correlated with the TEM results. To improve readability, the colour
151 labelling applied in Figure 1a will also be used in the succeeding figures.
152 Representative ACOM-TEM maps of the different samples are shown in Figure 1b. Several
153 ACOM-TEM maps were recorded per sample to improve statistical analysis and yielding a
154 total of 99 grains for the sample prepared at 0.27 Pa and between 400 and 1000 grains for
155 the other samples. The deposition direction is horizontal in each map, growing from left to
156 right (i.e., the Si/TiO_2 substrate is for every map on the left hand side, out of the frame). An
157 example of a high resolution TEM (HR-TEM) image showing the Si/TiO2/Ni interface is
158 provided in the appendix.
159 The viewing direction in these figures is the normal direction (ND) to the FIB sample. The
160 used reference system with ND, rolling direction (RD) and transverse direction (TD) is shown
161 in Figure 1b. Starting from the 0.27 Pa sample, we can observe that the thin film mostly
162 consists of large grains spanning the entire deposition width with only a few high angle GBs
163 (HAGBs), which are represented in the maps as black lines and include possible twin
164 boundaries. This is in agreement with the conclusion obtained from Figure 1a. For the 0.40
165 Pa and 0.53 Pa samples, a respective nucleation layer of 50 nm and 30 nm was anticipated
166 from the observations made in Figure 1a. Instead of the expected single-crystal nucleation
167 layer, several large grains originating on the TiO_2 side are observed in these films. It has
168 already been shown in the literature that an inverse correlation exists between grain size
169 and internal stress in electrodeposited Ni thin films [12,30]. Aside from the uniformly
170 distributed films (0.67 and 1.07 Pa), the largest grains in these examined films mostly
171 agglomerate on the TiO_2 side, explaining the delay in stress build-up during deposition.
172
173 A detailed grain size distribution is provided in Figure 2, showing that the average grain size
174 and spread decreases with increasing Ar pressure. Table 2 displays the average grain size
175 derived from Figure 2. The mean stress per film is also provided in Table 2, extracted from
176 Figure 1a by dividing the final stress.thickness value by the total film thickness.
177
178 It is worth noting that the films used in the present work have no strong (111) texture along
179 the growth direction, although this is commonly expected in fcc thin films [31]. This was
180 proven through (111) pole figures which show the texture parallel with the growth direction;
181 these figures are provided in Figure 3. Generally, the (111) orientation along the growth
182 direction is preferred in fcc metals due to the low interfacial energy of this plane [31]. The
183 expected texture can change because of, for example, impurities that can influence the

184 interfacial energies, the nucleation sites and/or surface diffusivities [31]. Perpendicular to
185 the growing direction, as shown in Figure 1b, it seems that at higher Ar pressure the films
186 have a distinct {110} texture. This is also displayed in the inverse pole figures in Figure 4.
187
188 So far, all TEM observations are consistent with the predictions from the stress-thickness
189 data. The delay in stress-build up has been explained by the agglomeration of large grains on
190 the substrate side, while the comparable increasing stress.thickness slope has been clarified
191 by a similarity in grain size. Regarding the downwards bending slope, it was noticed that
192 even though the samples deposited with 0.67 Pa and 1.07 Pa have a similar texture (Figure
193 1b) and grain size distribution (Figure 2), the curve of the downwards bending slope of the
194 internal stress is clearly stronger in the latter (Figure 1a). An explanation can be found using
195 the GBs and Σ3 twin boundaries (TBs).
196
197 In this respect, Figure 5a shows the same maps as Figure 1b, but with the orientation colour
198 overlay removed. Some of the present high angle GBs (HAGBs) are recognized as Σ3 TBs
199 (blue lines in Figure 5). These are identified based on a misorientation plane of 60°±9° and a
200 common plane between the neighbouring grains, which are the conditions according to the
201 Brandon criteria for Σ3 {111} coincident site lattice boundaries [28, 29]. In this work, we
202 show that Σ3 TBs are abundantly present in the films, even though Ni has a high stacking
203 fault energy (~120-130 $mJ.m^{-2}$) [33]. This is not a unique phenomenon as growth twins were
204 also observed in other materials with a high SFE such as in electron beam evaporated Pd thin
205 films [34] and electrodeposited Al [35]. In the maps, some Σ3 boundaries appear to be
206 curved which can due to the limitations from the ACOM-TEM or by the presence of Σ3 {112}
207 ITBs. A TB density can be calculated per sample based on the total sum of the lengths of
208 these Σ3 TBs divided by the total surface area of the accompanying sample; this is displayed
209 in Figure 5b. It shows that the 0.27, 0.40 and 1.07 Pa films have a relatively low TB density,
210 while 0.53 and 0.67 Pa are comparably high.
211
212 Next, from Figure 1a, the mean internal stress per sample can be extracted from each curve
213 (Figure 5c). This value is determined by dividing the final stress from the tail-end of that
214 curve by the total deposition thickness. Cross-correlating this stress behaviour with the TB
215 density shown in Figure 5b displays a clear connection. In the 0.27, 0.40, 0.53 and 0.67 Pa
216 samples, the increase of the mean stress observed in Figure 5c is attributed to the increase
217 of the density of GBs. When the internal elastic energy (IEE) reaches high levels such as in
218 the 0.53 and 0.67 Pa samples, the local stress could reach values higher than the yield
219 strength leading to the formation of dislocations. However, because of the high density of
220 GBs and TBs in these samples, the internal stress cannot be relaxed since both GBs [36] and
221 TBs [37–41] act as barriers for dislocation motion. Other mechanisms involving GB mediated
222 processes could also play a role. In this case, twin boundaries, being more stable, could
223 stabilize the grain structure and decrease the influence of grain boundary mediated stress
224 relaxation. For the 1.07 Pa sample, however, the mean stress is significantly lower in
225 comparison to the 0.67 Pa sample even though the HAGB density is similar. The difference
226 can be found in the Σ3 TB density as shown in Figure 5b, implying that TBs have a significant
227 influence on dislocation motion. Indeed, because of the smaller density of TBs in the 1.07 Pa
228 sample, dislocations can escape more easily leading to the relaxation of the internal stress.
229 The low TB density in the 1.07 Pa sample can be attributed to the deposition rate used.
230 Indeed, Amin-Ahmadi *et al*. observed a proportional correlation between the density of

growth twins in nanocrystalline Pd thin films deposited with electron beam evaporation and the used deposition rate [16,34]. On the other hand, the average twin spacing and grain size remained approximately unchanged. Considering that in our work the deposition rate was decreased with increasing Ar pressure and that the average grain size remained consistent at higher pressure, we can conclude that only the twin boundary density will be affected, as shown in this study.

4. Conclusions

The origin of internal stress build-up in sputter-deposited Ni thin films was explained based on ACOM-TEM analysis. It was shown that there is a clear correlation between the thickness from which the internal stress starts building up and the presence of large grains close to the substrate in agreement with the 'nucleation layer' model. The similarity between the films in internal stress increase in the beginning of the growth is correlated with a comparable grain size, which is in accordance with the island coalescence model. The low internal stress at low Ar pressure is justified by the small amount of grain/twin boundaries, induced by the low amount of scattering during deposition. With increasing pressure, even though the deposition rate is adjusted, the scattering induces smaller grains which increase the internal stress of the film. At the highest Ar pressure, this effect is counterbalanced by a decreased Σ3 TB density due to the low deposition rate. This allows more relaxation of the internal stress by reducing the confinement of dislocations in grains in which local plasticity can be activated.

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264 # 6. Appendix
265

266 ## 6.1. Focused ion beam preparation details
267

268 Since we are working with nanocrystalline films and to avoid complete amorphization, the
269 thickness of the FIB foil is aimed to lie between 20-40 nm, which is for our case ideal to
270 minimize the amount of overlapping grains. The starting thickness of the samples cut by FIB
271 from the films is usually ~3 µm. During thinning, while the thickness is still higher than 100
272 nm, the FIB is operated at 30 kV and the current is decreased proportional to the remaining
273 thickness (ranging from 0.79 nA to 0.08 nA). Below 100 nm, the voltage and current are first
274 lowered to 8 kV and 66 pA, and final thinning is performed at 2 kV with 23 pA.

275

276 # 7. References
277

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396 ## 8. List of figure and table captions
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398 Table 1: Deposition rate and deposition time used at each Ar pressure.

399 Figure 1: (a) Stress-thickness curves showing the stress build-up in the Ni thin film deposited at
400 different Ar pressures. (b) ACOM-TEM maps of Ni thin films deposited with varying Ar pressure,
401 viewed normal to the FIB sample. Showing from left to right: 0.27 Pa (black contour), 0.40 Pa (red),
402 0.53 Pa (blue), 0.67 Pa (green) and 1.07 Pa (orange). The indexation colour scaling is included as inset
403 figure. The grains are separated by black lines representing HAGBs. The reference system is provided
404 at the bottom.

405 Figure 2: (a) Grain size distribution of the 0.27 Pa, (b) 0.40 Pa (c) 0.53 Pa (d) 0.67 Pa and (e) 1.07 Pa
406 samples.

407 Table 1: Average grain size and mean stress per film. The numbers in between brackets represent the
408 standard deviation.

409 Figure 3: (a) (111) pole figure viewed parallel to the growth direction for the 0.27 Pa sample. (b)
410 same for the 0.40 Pa (c) 0.53 Pa (d) 0.67 Pa and (e) 1.07 Pa samples. (ND = normal direction to the
411 FIB; RD = rolling direction inside the film parallel to the substrate).

412 Figure 4: Inverse pole figures showing the distribution of texture in the FIB samples. (a) 0.27 Pa (b)
413 0.40 Pa (c) 0.53 Pa (d) 0.67 Pa and (e) 1.07 Pa.

414 Figure 5: Blank ACOM-TEM maps highlighting the HAGBs. The Σ3 TBs are marked as blue lines. (b) Σ3
415 TB density as a function of deposition pressure. Error bars are added based on the Σ3 TB density
416 spread from ACOM-TEM maps of different regions. (c) Mean internal stress as a function of
417 deposition pressure, obtained from the stress-thickness curves of Figure 1a.

418 Figure A1: HR-TEM image showing the Si/TiO_2/Ni interface with the amorphous TiO_2 passivation
419 layer.