Nanoindentation of Ag/Ni multilayered thin films

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Nanoindentation was used to study the mechanical properties of Ag/Ni multilayered thin films. Both the hardness and the elastic modulus of the multilayered thin films had values between those for homogeneous Ag and Ni thin films. The trend in the hardness with layer repeat length can be explained by the effects of both the stress and the microstructure. No evidence for interfacial effects on hardness was found. A decrease in modulus at the smallest repeat lengths was compared with literature data on the elastic constants of Ag/Ni multilayers.

I. INTRODUCTION

Nanoindentation is a versatile technique for studying the mechanical properties of thin films on substrates.[1] The hardness, an elastic modulus,[2] and the strain rate sensitivity[3] can be measured. The commercially available nanoindenter has a load resolution of 0.2 μN and a depth resolution of 0.2 nm, which make it possible to obtain accurate measurements on small samples (e.g., precipitates and particles in a matrix) and thin films.

The nanoindenter continuously measures the load and displacement of a three-sided pyramidal diamond indenter as it is pushed into the sample. The hardness is obtained as a function of indent depth during loading by dividing the applied load by the projected contact area of the indenter tip. The film compliance is determined from the slope of the load-displacement curve upon unloading when the material recovers elastically. An elastic modulus can be determined approximately from the compliance by using a relationship derived from the indentation of an isotropic elastic material and providing a value for the Poisson ratio.[2]

The mechanical properties of multilayered thin films are of clear technological interest. In particular, increases in the yield strength and elastic modulus have been predicted[4,5] and measured.[6,7] The hardness and elastic modulus of some metal/metal multilayered thin films have been measured with nanoindentation. When the bilayer repeat lengths became smaller than 3.0 nm, the hardness was observed to increase (Cu/Ni),[8] to decrease (Mo/V, Nb/Ta),[9] and to remain constant (Au/Ni).[10] In all of these cases the elastic modulus showed no dependence on repeat length and had a value intermediate between that of the two constituents. In this paper we present nanoindentation measurements on Ag/Ni multilayers. The system was chosen because of the unusually low mutual solubility of Ag and Ni, which produces sharp interfaces, and because of the additional advantage that Ag and Ni do not form any intermetallic compounds. We observed a decrease in hardness and modulus at small bilayer repeat lengths. We can account for the trend in hardness by stress and microstructure effects, and we compare the modulus data to literature values of the elastic constants of Ag/Ni multilayers.

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II. EXPERIMENTS

Ag/Ni multilayers with equal layer thicknesses and with bilayer repeat lengths between 1.3 and 23.0 nm were deposited using ion beam sputtering.[11] Ag (99.9% pure) and Ni (99.95% pure) targets were mounted on adjacent faces of a target block that was rotated periodically to produce the layering. The substrates were 360-μm-thick (100) Si wafers that were cooled with liquid nitrogen during deposition to minimize the roughness of the layer interfaces[12] which was around 0.3 nm as determined by x-ray diffraction.[13]

The bilayer repeat length λ of the multilayers was determined from the positions of satellite peaks about the (000) reflection in a θ-2θ x-ray scan.[14] Examples of scans for Ag/Ni multilayers with various repeat lengths are presented in Fig. 1. The total film thickness was found by multiplying the number of bilayers by the bilayer repeat length. Most films had thicknesses between 0.94 and 1.13 μm, but two samples had thicknesses of 1.29 and 1.40 μm. The thicknesses of the Ag and Ni layers in a sample were determined from the relative atomic compositions of Ag and Ni in the film, which was measured with the electron microprobe. The layer thicknesses were equal to within 4%. High-angle x-ray diffraction showed that the films had a strong $\langle 111 \rangle$ texture.

The indentation measurements were made with a commercial nanoindenter with a Berkovitch diamond indenter tip at the Los Alamos National Laboratory. Nine indents spaced 50 μm apart in a square array were made in each sample. Before the start of each indentation set, the displacement of the indenter tip was measured as it rested on the sample surface until the thermal variations were acceptable. There were five stages in each indentation sequence. During the loading segment the tip entered the sample at a rate of 40 μN/s to a total depth of roughly 100 nm. The indenter was held at that depth for 60 s and unloaded at a rate of 40 μN/s until the load was at 20% of the maximum. The indenter was

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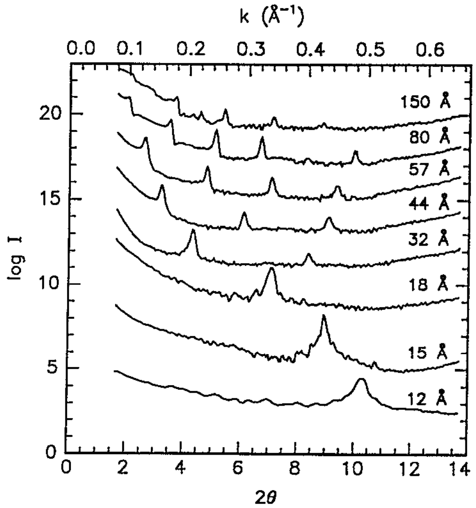

FIG. 1. θ-2θ x-ray diffraction scans, offset vertically, for Ag/Ni multilayers with different bilayer repeat lengths as indicated. Cr K_α radiation was used.

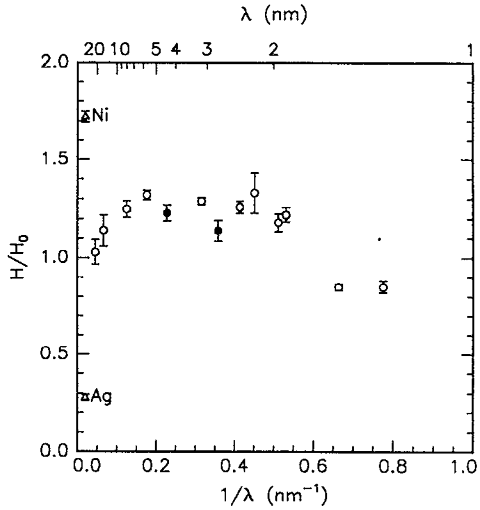

FIG. 3. Hardness vs inverse bilayer repeat length for Ag/Ni multilayered thin films (circles). The hardness was measured at a plastic depth of 65 nm. H_0 is the hardness calculated from the rule of mixtures from the hardness of the pure Ag and Ni films (triangles). The filled symobls correspond to samples prepared at an earlier time.

held at that load for 100 s to determine the thermal drift and then withdrawn from the sample. Examples of load versus displacement curves measured in that manner are given in Fig. 2 for homogeneous Ag and Ni films and multilayered Ag/Ni films with different bilayer repeat lengths.

We measured the stress in the Ag/Ni multilayers from substrate curvature using a laser scanning device.[15] Stresses were measured immediately after deposition. There was significant stress relaxation at room temperature. Since the nanoindentation measurements were made about two months after the films were deposited, the actual stress in the films was no longer at the deposition stress, but the relative order of the stresses remained the same. The stress was measured in another set of Ag/Ni multilayers[16] for 25 h after deposition, and over that time the stress in all films reduced by about the same amount (about 100 MPa).

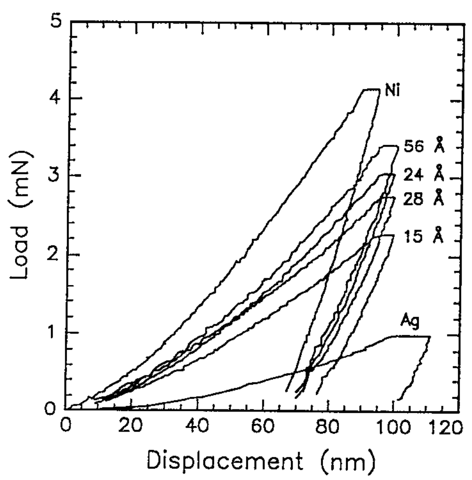

FIG. 2. Load vs total displacement measured by nanoindentation for homogeneous Ag and Ni films and Ag/Ni multilayered thin films with different bilayer repeat lengths as indicated.

III. RESULTS

The hardness and elastic modulus measured at a plastic depth of 65 nm are plotted in Figs. 3 and 4 as a function of inverse bilayer repeat length. The data were normalized by H_0 and E_0, respectively, the hardness and elastic modulus values obtained from the rule of mixtures

$$H_0 = V_{Ag}H_{Ag} + V_{Ni}H_{Ni}, \tag{1}$$

$$E_0 = V_{Ag}E_{Ag} + V_{Ni}E_{Ni}, \tag{2}$$

where H_{Ag} and H_{Ni} are the measured hardnesses of homogeneous Ag and Ni thin films, E_{Ag} and E_{Ni} are the measured elastic moduli of homogeneous Ag and Ni films, and V_{Ag} and

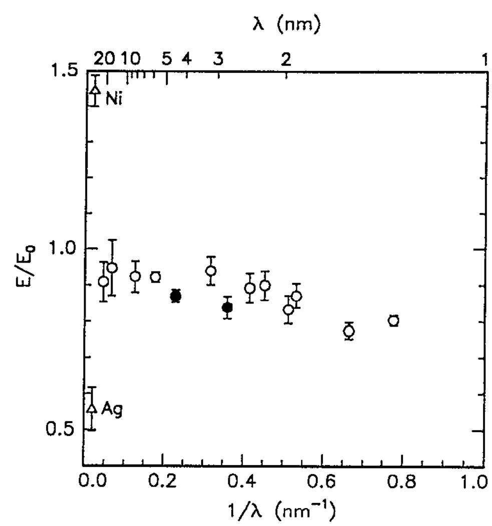

FIG. 4. Modulus vs inverse bilayer repeat length for Ag/Ni multilayered thin films (circles). The modulus was measured at a plastic depth of 65 nm. E_0 is the hardness calculated from the rule of mixtures from the hardnesses of the pure Ag and Ni films (triangles). The filled symbols correspond to samples prepared at an earlier time.

V_{Ni} are the measured volume fractions of Ag and Ni in the multilayers. The error bars represent the standard deviations of the measurements from nine indents.

The hardness is equal to the value from the rule of mixtures at the largest repeat length (15.0 nm), increases by about 30% as the repeat length is decreased to a repeat length of 5.7 nm, and remains constant until it decreases sharply at repeat lengths of less than 2.2 nm. The modulus is slightly lower than the value from the rule of mixtures at large repeat lengths, remains constant as the repeat length is decreased and decreases at small repeat lengths to about 80% of the rule of mixtures value.

There are two samples, the films with 2.78 and 4.38 nm repeat lengths, which have hardness and modulus values that are significantly lower than the trend. These films are different from the others in two ways. They are thicker (by 26% and 37%, respectively), and they were deposited two months before the other films. The film thickness is important when considering the substrate effect on the indentation. It has been suggested that the indent depth should be less than 10% of the film thickness to avoid substrate effects,[17] and this condition was met for all the films. However, if somehow the hardness measured in the 1.0-μm-thick films were larger because of the effect of the Si substrate, the indents in the two thicker films could have experienced less of a substrate effect. In that case, there should be an indent depth that is shallow enough to avoid significant substrate effects for all films. However, the hardness for shallower indents was examined, and the discrepancy between the thicker and thinner films is not reduced at lower indent depths. We conclude that the effect of the substrate, if any, is the same for all the films.

The time between deposition and testing is important because the Ag/Ni multilayers were deposited onto liquid-nitrogen-cooled substrates and had large compressive deposition stresses. The stresses are caused by the difference in thermal expansion between film and substrate, by growth stresses, and by the interface stress.[16] They relax significantly at room temperature, and the two films that were deposited two months before the others should have significantly lower stresses. Stress and normalized hardness are plotted versus inverse bilayer repeat length in Fig. 5. The stress plotted in the figure was measured immediately after deposition for all films. If a compressive stress in the films affects the hardness significantly, the two films with a smaller compressive stress are expected to have a lower hardness than the trend, as we observed. A similar decrease was observed in hardness measurements of Cu thin films made three month apart,[18] and it was attributed to the relaxation of the compressive film stress with time.

IV. DISCUSSION

We consider three factors that may influence the hardness of multilayered thin films: stress, microstructure, and interfaces. The stress state of a material can affect its hardness. A compressive (tensile) stress results in an increased (decreased) hardness compared with the stress-free material. Vitovec[19] performed conventional diamond pyramid hardness testing on large-grained, bulk materials (spring steel,

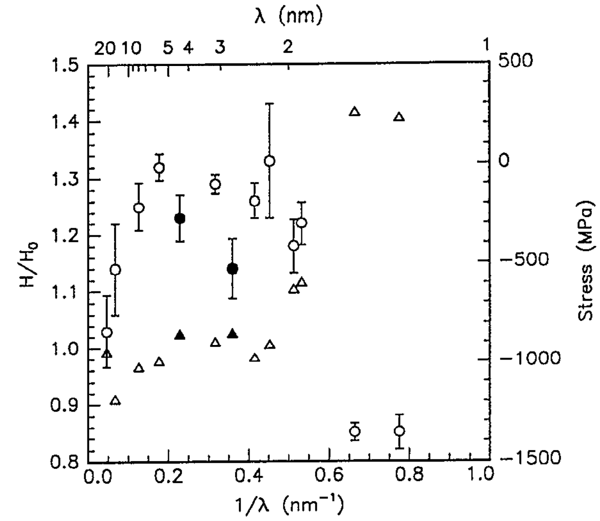

FIG. 5. Normalized hardness (circles) and film stress (triangles) vs inverse bilayer repeat length for Ag/Ni multilayered thin films. The film stress was measured immediately after deposition. The filled symbols correspond to samples prepared at an earlier time.

stainless steel, and an aluminum bronze) while the samples were held under constant uniaxial stress in a tensile tester. He observed that the hardness decreased with increasing applied tensile stress. The hardness is proportional to the yield stress of a material. Tensile preloading brings the shear stress closer to the yield point, which reduces the load from the indenter that is needed to cause yielding. When the applied tensile load was above the yield stress, the hardness increased due to work hardening. LaFontaine *et al.*[20] measured both hardness, by nanoindentation, and stress, by x-ray diffraction lattice parameter measurements, in thin Al films on Si substrates. The films were heat treated, and upon cooling, a large tensile stress was produced in the films because of the difference in film and substrate thermal expansion coefficients. The hardness and stress were measured as a function of time after heat treatment as the tensile stress decreased due to stress relaxation. The hardness was observed to increase as the stress became less tensile. It was thought that the Al grain size did not change during the heat treatment nor during stress relaxation at room temperature. Dirks *et al.*[21] measured stress and hardness as a function of alloy composition for binary alloy films containing either Ag or Au, but it is difficult to separate the effects of stress and composition on the hardness in these results.

There are three regions of interest in Fig. 5. At large repeat lengths, both stress and hardness increase with decreasing repeat length, with the exception of the largest repeat length. Where the deposition stress is relatively constant, the hardness is constant. At smaller repeat lengths, the stress becomes less compressive and eventually tensile, and the hardness decreases. The hardness in the last two regions can be explained by considering the combination of initial film stress and the stress applied by the indenter.[22] The von Mises criterion for yielding[23] is

$$\sigma_y=\frac{1}{\sqrt{2}}\ [(\sigma_1-\sigma_2)^2+(\sigma_2-\sigma_3)^2+(\sigma_3-\sigma_1)^2]^{1/2}, \quad (3)$$

where σ_y is the yield stress in uniaxial tension and σ_1, σ_2, and σ_3 are the three principal stresses. We take σ_1 and σ_2 to be stresses in the plane of the film and σ_3 to be the stress perpendicular to the film surface. Since the film stress is biaxial, $\sigma_1=\sigma_2$. The stress applied by the indenter is assumed to have a σ_3 component and to have equal σ_1 and σ_2 components. von Mises' criterion is then

$$\sigma_1^{\text{film}}+\sigma_1^{\text{indenter}}-\sigma_3^{\text{indenter}}=\pm\sigma_y, \quad (4)$$

where σ_1 and σ_3 are divided into film and indenter contributions. Since $\sigma_3^{\text{indenter}}$ is compressive, the appropriate von Mises criterion is

$$\sigma_1^{\text{indenter}}-\sigma_3^{\text{indenter}}=\sigma_y-\sigma_1^{\text{film}}. \quad (5)$$

Therefore, yielding occurs at a lower stress as the film stress becomes less compressive or more tensile. The effect of stress on hardness explains the behavior of hardness in Fig. 5 for intermediate (2.2 nm$<\lambda<$5.7 nm) and small repeat lengths ($\lambda<$2.2 nm) where the film stress is constant or becomes less compressive. It does not explain the increase in hardness with decreasing repeat length at large repeat lengths (5.7 nm$<\lambda$). It is possible that the standard, Hall–Petch-like decrease in the yield stress with increasing grain size or layer thickness dominates the stress effect in this regime. The effect of microstructure will be considered in more detail below.

Baker *et al.*[10] measured the stress and hardness for Au/Ni multilayers. The hardness was the same for all samples, which had repeat lengths between 1.0 and 4.0 nm. The film stress was compressive at large repeat lengths, rose to a compressive maximum at 2.0 nm, and became tensile at small repeat lengths. If the effect of stress on hardness dominates in these films, the hardness should decrease when the film stress becomes tensile. However, since the mutual solubilities of Au and Ni are substantial, there could also be hardening due to alloying at the interfaces. The two effects may have combined to produce an unchanged hardness. Stresses were not measured in Cu/Ni multilayers.[8] In addition, Cu and Ni form a solid solution, and significant intermixing is expected during deposition. Since the critical shear stress of a CuNi alloy is greater than that of either Cu or Ni,[24] the increased hardness can also be explained by the increased volume fraction of the alloy at smaller repeat lengths, if the thickness of the mixed interfacial layer is constant.

Microstructural characteristics of a material, in particular the grain size, affect hardness. It is well known that the hardness H and yield stress of large-grained materials often exhibit a dependence on grain size that follows a Hall–Petch relationship:

$$H=H_0+kD^{-1/2},$$

where D is the average grain diameter, and H_0 and k are constants.[25–28] In thin metal films the grain size is often of the same order as the film thickness.[29] In these multilayered films, we expect that the grain size is about equal to the layer thickness, which is less than 11.5 nm. Dependence of hardness on grain size for nanocrystalline materials with grain sizes smaller than about 10 nm is not well understood.[30] As the grain size decreased, hardness was observed to increase [mechanically alloyed Fe (Ref. 31) and Nb_3Sn (Ref. 32)], decrease [electrodeposited NiP (Ref. 33) and gas-condensed Pd and Cu (Ref. 34)], and remain constant [gas-condensed Pd (Ref. 35)]. It is predicted that there is a critical grain size below which the Hall–Petch strengthening mechanism ceases to operate. For example, there is a minimum grain size that will support a dislocation pileup.[36] Another theory suggests that the volume fraction of triple junctions strongly affects the mechanical properties of fine-grained materials.[37]

Another interpretation of the trend of hardness with layer thickness in the Ag/Ni multilayers is that the hardness is limited by deformation of the Ni or Ag grains within the layers. For the large repeat lengths ($\lambda>$5.7 nm), a Hall–Petch-like trend of hardness with layer thickness is observed. For a layer thickness smaller than 2.8 nm, the critical grain size has been reached and a deformation mechanism other than Hall–Petch is the dominant mechanism. For the Ag/Ni multilayers the hardness remains constant with layer thickness below the critical grain size, except for the two smallest bilayer repeat lengths, which show a 30% decrease in the hardness. One estimate of the critical grain size in Ni is 2.5 nm,[34] which agrees with the layer thickness at which we observe departure from Hall–Petch behavior. We know of no hardness measurements on pure Ni with such small grain sizes. However, hardness measurements on larger-grained electrodeposited Ni give conflicting results. One study shows a departure from Hall–Petch behavior for a grain size of 17.7 nm,[38] and another study reports no departure for grain sizes as small as 12 nm.[39]

The two samples with the smallest bilayer repeat lengths (1.29 and 1.51 nm) may have a different microstructure than the other films. While these two samples show strong x-ray satellites about the (000) peak (see Fig. 1), they do not exhibit high angle Bragg peaks in either reflection or transmission θ-2θ x-ray diffraction scans, which may indicate a very small crystal size, possibly from incomplete layering. Cross-sectional transmission electron microscopy (TEM) showed continuous layering for multilayers with repeat lengths as small as 1.85 nm, but no samples with repeat lengths smaller than that were examined. There is little convincing evidence in the literature to suggest that, in general, hardness decreases at very small grain sizes. The most likely explanation for the low hardness in these samples therefore remains the effect of the large tensile stress as discussed above.

The explanation based on microstructural effects suggests that the hardness is dominated by the grain size of the Ni layer. Nanoindentation measurements of Au/Ni multilayers exhibit a constant hardness for Ni layer thicknesses below 2.0 nm,[10] similar to our measurements. Cu/Ni multilayers are reported to exhibit a Hall–Petch-like dependence for films with layer thicknesses between 0.8 and 5.6 nm.[8] However, with the exception of the smallest repeat length sample, the hardness is constant within the error bars.

The interface between two materials with different elastic constants can serve as a barrier to dislocations and cause the hardness of the composite to exceed the hardness of either of the constituents, provided the layer thickness is small enough to inhibit dislocation sources.[4] The interface effect was used to explain the increase in yield stress measured in Al/Cu multilayers.[6] Doerner showed that at very small bilayer repeat lengths a softening can occur, which was observed in Mo/V and Nb/Ta multilayered thin films.[9] She calculated the total image force on a dislocation in the more compliant layer that resists its movement across the interface into the stiffer layer and showed that the image force decreases with decreasing bilayer repeat length. Her model predicts the onset of the reduced hardness in both the Mo/V and Nb/Ta systems, but the predicted average hardness is too high. Doerner attributes that to intermixing at the interfaces which reduces the effectiveness of the interfaces as dislocation barriers and to the hardness model being based on the motion of single dislocations which does not fully represent the complex stress state and dislocation below the indenter tip.

Even though our Ag/Ni multilayers had very sharp interfaces, the hardness of the multilayers was intermediate between that of the hardnesses of Ag and Ni thin films. We did observe a softening at small bilayer repeat lengths, but an application of Doerner's model to Ag/Ni multilayers predicts the softening to occur at a much smaller repeat length than we observe. In both Cu/Ni (Ref. 8) and Au/Ni (Ref. 10) multilayers the hardness was between the hardness of the constituents and no softening was observed at small repeat lengths. None of the three systems showed evidence of the effect of interfaces on hardness.

The compliance of a thin film is measured by nanoindentation using the slope of the load-displacement curve upon unloading. Doerner *et al.*[2] used the model of a flat circular punch indenting an isotropic elastic material to define the relationship between the slope and an elastic modulus. Since multilayered thin films are not isotropic, the moduli obtained from nanoindentation are at best only useful as relative values. Furthermore, since the indentation process destroys the layering, it is not clear that the effect of layering on the modulus can be measured with this method.[8] We observed a 15% decrease in the modulus at small bilayer repeat lengths as seen in Fig. 4. No change in modulus with repeat length has been observed for other metal/metal multilayered thin films,[8–10] but a 15% decrease in the modulus of epitaxial $TiN/(V_{0.6}Nb_{0.4})N$ multilayers at short repeat lengths was measured by nanoindentation.[40]

Other elastic property measurements have been performed on Ag/Ni multilayered thin films. Young's modulus, measured by tensile testing, and the elastic compliance C_{11} measured by Brillouin scattering, were observed to be constant for unsupported Ag/Ni multilayers with bilayer repeat lengths between 1.3 and 17.6 nm.[41] However, the elastic compliance C_{44} measured by Brillouin scattering, showed a continuous decrease with decreasing bilayer repeat length down to a softening of 40% for a 1.77 nm repeat length Ag/Ni multilayer.[12] It might be thought that since the shear associated with C_{44} is aligned along the film normal it should give a large contribution to the modulus measured by nanoindentation. However, Brillouin scattering measurements of C_{44} in $TiN/(V_{0.6}Nb_{0.4})N$ multilayers show that it is independent of bilayer repeat length,[42] but the modulus measured by indentation shows a decrease at small repeat lengths.[40]

V. CONCLUSION

The hardness and a modulus were measured in Ag/Ni multilayered thin films using nanoindentation. A decrease in both quantities was observed at small bilayer repeat lengths. A combination of microstructural and stress effects on hardness can account for the trends observed in the data. No evidence for interfacial effects on hardness was found. The decrease in modulus at small repeat lengths was similar to that observed for $TiN/(V_{0.6}Nb_{0.4})N$ multilayers[40] and it is similar to the softening of the C_{44} elastic compliance measured in other Ag/Ni multilayers.[12] No anomalous elastic properties were observed.

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