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Characterization of Metal and Metal Alloy Films as Contact Materials in MEMS Switches

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Characterization of metal and metal alloy films as contact materials in MEMS switches

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Abstract

This study presents a basic step toward the selection methodology of electric contact materials for microelectromechanical systems (MEMS) metal contact switches. This involves the interrelationship between two important parameters, resistivity and hardness, since they provide the guidelines and assessment of contact resistance, wear, deformation and adhesion characteristics of MEMS switches. For this purpose, thin film alloys of three noble metals, platinum (Pt), rhodium (Rh) and ruthenium (Ru) with gold (Au), were investigated. The interrelationship between resistivity and hardness was established for three levels of alloying of these metals with gold. Thin films of gold (Au), platinum (Pt), ruthenium (Rh) and rhodium (Ru) were also characterized to obtain their baseline data for comparison. All films were deposited on silicon substrates. When Ru, Rh and Pt are alloyed with Au, their hardness generally decreases but resistivity increases. This decrease or increase was, in general, dependent upon the amount of alloying.

1. Introduction

Recently, radio frequency (RF) microelectromechanical systems (MEMS) switches have drawn attention due to their superior switching performance in GHz range signals. Because of their miniature sizes and low power requirements, RF MEMS metal contact switches are ideally suited in many applications, such as phase shifters in phased array radar and reconfigurable antennas in satellite communication. Recent developments in micro-fabrication techniques have also helped their popularity. Two of the most important performance criteria of RF MEMS switches are low contact resistance and high reliability. It is desired to have a contact resistance of less than 1 $^{\sim}$ 2 Ω and high reliability of more than 10 8 hotswitched cycles during their practical use in most applications. Selection of the appropriate contact

material(s) for MEMS switches is therefore a paramount consideration and probably the first important step to achieve the aforementioned goals.

Low contact resistance of MEMS switches requires contact materials with low resistivity, low hardness and high chemical resistance to corrosion. Numerous materials have been tried for contact materials of macro switch, i.e. Pd, Pt, Au, Ni, Ag, etc, and their alloys. Among them, the most widely used contact material in macro switch is silver and its alloys due to their superior electrical and mechanical properties. On the other hand, silver and its alloys tend to form a nonconductive sulfide layer on the surface which should be removed for good electrical contact. This is not a big problem in macro switches, which usually apply enough contact force (N range) to break the sulfide layer. However, this sulfide surface layer is a real obstacle in MEMS switches since the contact forces of MEMS switches (NN range) are usually well short of penetrating the surface layer. Thus, gold-on-gold electric contact has been typically used in MEMS switches due to its low electric resistivity, and resistance to surface oxide. The low hardness of gold, however, leads to adhesion problems in MEMS switches. The low hardness of gold as well as the adhesive force between gold contacts also causes wear, deformation and adhesion of contacts during cycling which eventually lead to the failure of a MEMS switch such that it is stuck closed or contact resistance increases with increasing switch cycles.

Therefore, a long-term goal of MEMS designers as well as for the present study is to explore metal and/or metal alloy thin films which are able to mitigate wear, deformation and adhesion of electric contacts and are not prone to corrosion (polymerization and oxidation) with no increase or a moderate increase in resistivity. To alleviate the aforementioned shortcomings of gold-on-gold electric contact, different metals and metal alloys have been investigated to improve wear properties while maintaining low contact resistance, but no clear alternative has yet emerged. 5.8-10 The present study is a continuation of these efforts to seek appropriate contact materials for MEMS switches by exploiting various metals or their alloys with gold. Particularly, the mechanical properties of thin metal and metal alloy films were characterized by using the instrumented nanoindentation technique, so that these mechanical properties along with other considerations (e.g. contact resistance and surface contaminants) can be used as a basis for material selection in MEMS design. Nanoindentation measurement is a proven and well-established technique for measuring modulus and hardness of thin film or small-scale materials. 11,12 The major attraction of the nanoindentation technique is its simplicity. Mechanical properties can be obtained by a simple load and displacement relationship during indentation without a requirement of imaging the indentation impression. As the resolution of nanoindenter equipment has recently improved, this technique has been extended reliably at micro- or nano-scales.

2. Experiments

2.1. Materials and thin film deposition

As mentioned earlier, gold-on-gold contacts are often used in series contact switches due to the low resistivity and superior resistance to chemical attack, but they have two principal failure modes i.e. increasing contact resistance with increasing switch cycles and sticking shut (stiction) associated with cold welding or material transfer. In other words, as gold is a soft metal it is easily subjected to mechanical wear, and thereby the associated material transfer and adhesion decrease the switch reliability. Higher modulus noble metals, such as platinum (Pt), rhodium (Rh) and ruthenium (Ru), exhibit

slightly higher resistivities but provide a harder contact surface that may be less susceptible to material transfer. However, the contact resistance of platinum group metals may degrade by frictional polymerization after repeated switch cycling. Frictional polymerization is the formation of organic polymers on contacts with catalytic active metals. Gold has significantly less affinity in creating frictional polymers. Therefore, alloying gold with these noble metals may help to improve the mechanical strength (wear and erosion) without degrading the chemical resistance. However, metal alloy thin films are known to have slightly higher resistivities than pure metal films due to crystal structure and defect density. Therefore, several films for potential application in MEMS switches were analyzed in this study to observe the influence of alloying gold with three noble metals, Pt, Rh and Ru, on mechanical and electrical properties. Based on the binary alloy phase diagrams, all of the alloys of the study except Au-10%Pt are expected to have two phases. In addition, thin films of gold (Au), platinum (Pt), ruthenium (Rh) and rhodium (Ru) were characterized to obtain their baseline data for the comparison.

Thin films were deposited on silicon substrates from 99.95% pure Au, Pt, Rh and Ru targets in a Denton Vacuum Discovery 18 dc magnetron sputtering system with a base vacuum of 1.4×10^{-6} Pa. A mass flow regulated argon (Ar) sputtering pressure of 0.32 Pa was used for all depositions except for Pt depositions at 1.06 Pa. Substrate materials were 75 mm diameter and 380 µm thick (100) silicon disc wafers. Substrates were cleaned in a buffered oxide etch solution, nitrogen (N2) dried and argon (Ar) plasma cleaned for 5 min at 60 W prior to deposition. This cleaning process was found suitable for the sputtering process in the present study. Substrates were placed on a water-cooled substrate holder without external heating. Film thicknesses were either 300 or 1500 nm. Binary alloy films were fabricated by co-sputtering Au with Pt, Rh or Ru to achieve Au-30%Rh, Au-70%Rh, Au-30%Ru, Au-70%Ru, Au-10%Pt and Au-50%Pt. The procedure for co-sputtering the metal alloy films was to first characterize deposition rates for the individual alloy components and then co-sputter at the appropriate power levels. 5 Depositions were performed at 25, 50, 100 and 250 W forward cathode power and film thicknesses were measured with a Tencor P-10 surface profilometer. Decreased power/increased deposition times were used only for deposition rate calibration for alloys. The sputter powers used were needed to obtain the desired alloy combinations. With these data, deposition rate versus cathode power was plotted and curve fitted. The curve fit equations were then used to estimate cathode power level settings needed to deposit alloy films of appropriate thickness. Table 1 shows the alloys studied, deposition rates and cathode powers to achieve alloys films. A 10 nm Cr adhesion layer was used for Au, Au-30%Rh, Au-30%Ru and Au-10%Pt films. Since the chemical analysis of thin film is required to conduct elaborate surface analysis and as this study focused on mechanical properties of alloys with rather large compositional differences to explore the possible candidate materials, chemical analysis of alloy films was not performed. Therefore, the composition of the films was not confirmed in the present study. However, once the candidate material for switch with precise composition is selected, the chemical analysis of alloy would be required.

Table 1. Deposition power and rate for single metal and co-sputtered metal films.

	Au	Pt	Rh	Ru	Au-	Au-	Au-	Au-	Au-	Au-50%Pt
					30%Rh	70%Rh	30%Ru	70%Ru	10%Pt	
Power (W)	250	250	250	250	250/254	47/250	168/250	33/250	250/54	127/250
Deposition rate (nm min ⁻¹)	87	48	40	27	126	57	87	39	95	87

Note: Deposition rate for alloys is cumulative for both targets. Sputter power for alloys is noted as Au power/metal power.

2.2. Nanoindentation test

Nanoindentation tests were conducted on thin films using a dynamic contact module (DCM) option available on the Nanoindenter XP using a Berkovich indenter tip to measure Young's modulus and hardness. Nanoindenter XP is a commercially available equipment from MTS (Oak Ridge, TN) which has displacement and load resolutions of 0.01 nm and 50 nN, respectively. The DCM option offers higher precision and better resolution than the standard Nanoindenter XP such that the displacement and load resolution of DCM are 0.0002 nm and 1 nN, respectively. However, the maximum load for DCM is 10 mN which is much lower than the maximum load of Nanoindenter XP, i.e. 500 mN. No special cleaning processes were used prior to performing nanoindentation test and resistivity measurement.

A total of 15 indentations were conducted on each specimen using the continuous stiffness measurement (CSM) technique and the average values of 15 indentations are presented in the present study. The CSM allows the measurement of dynamic stiffness continuously throughout the loading segment by superimposing a small dynamic oscillating displacement on the quasi-static force by means of a frequency-specific amplifier. The oscillation displacement was 1 nm and the frequency was 75 Hz. The area function (A) of the Berkovich tip was obtained by the indentations on the fused silica in the range of 10–500 nm indentation depth, i.e. displacement as suggested by Oliver and Pharr. As per their method, the area function is represented as

(1)

$$A(h) = C_0 h^2 + C_1 h + C_2 h^{1/2} + C_3 h^{1/4} + \dots + C_8 h^{1/128},$$

where C_0, \ldots, C_8 are constants determined by curve fitting, and h is the indentation depth. Unfortunately, it was not possible to obtain an area function that reasonably covered the depth less than ~15 nm as the indentation size effect exists at these shallow depths leading to uncertainty in measurements. Thus, only modulus and hardness obtained from the indentation depth larger than 15 nm are presented in this study. Figure 1 shows the modulus of fused silica based on the area function used in this study. It can be seen that the measured elastic modulus shows almost a constant value, which is close to the expected modulus, i.e. 72 GPa, of the fused silica throughout the entire indentation depth suggesting that the area function is reasonably good at a depth larger than 15 nm.

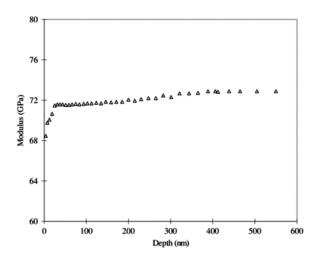


Figure 1. Modulus versus penetration depth on a fused silica standard. The elastic modulus of fused silica is 72 GPa.

2.3. Resistivity measurement

Film resistivity was calculated from the sheet resistance measured by a standard four-point probe and the film thickness measured using a profilometer. 5 At least ten resistivity measurements were obtained across each of the thin film wafer to ensure uniform material deposition as shown in table 2

Table 2. Resistivit	of metals and	metal alloys.

Resistivity	Au	Pt	Rh	Ru	Au-	Au-	Au-	Au-	Au-	Au-50%Pt
(μΩ cm)					30%Rh	70%Rh	30%Ru	70%Ru	10%Pt	
1	3.4	16.5	9.5	14.2	63.4	44.8	84.9	87.2	14.9	38.8
2	3.6	16.3	9.1	13.6	54.4	42.0	80.5	84.6	14.8	44.6
3	4.0	17.1	9.0	13.2	50.7	40.0	77.8	83.4	16.1	38.3
4	3.2	16.7	9.8	14.9	74.1	49.0	95.3	88.6	15.2	52.9
5	3.3	16.4	9.5	13.9	66.5	46.5	89.6	87.2	14.8	47.9
6	3.4	16.3	9.2	13.3	55.8	44.4	82.7	85.6	14.8	42.9
7	4.0	16.8	9.1	13.4	50.7	40.3	78.2	83.6	15.4	38.2
8	3.3	16.5	9.3	14.4	64.7	47.7	89.2	87.4	14.9	49.7
9	3.4	16.4	9.1	13.7	56.1	44.4	82.5	85.0	15.0	14.7
10	4.1	17.1	9.1	13.4	51.5	41.1	87.4	84.7	15.8	38.0
Average	3.6	16.6	9.3	13.8	58.8	44.0	83.9	85.7	15.2	43.3

3. Results and discussion

3.1. Nanoindentation measurements

A typical result of nanoindentation measurements on the silicon substrate is shown in figure 2. As expected for a monolithic material, both modulus and hardness are almost constant at 12.5 GPa and 167 GPa, respectively, over the entire indentation depth. The measured modulus and hardness are close to the previously reported values. Figure 3 compares the contact stiffness as a function of the indentation depth up to 200 nm for various metal and metal alloy thin films. The indentation depth is normalized by the film thickness, i.e. 300 nm. Since the contact stiffness is directly related to the

modulus of specimen and indenter tip as well as the contact area, it should increase linearly with increasing indentation depth for a monolithic/homogeneous material. For a thin film, on the other hand, the difference in moduli between the film and the substrate deviates the stiffness from linearity with increasing indentation depth. This is also the case in the present study as shown in figure 3. The deviation from linearity is larger when the mismatch of moduli between the thin film and the substrate material is larger, which is in the case of Ru and Au thin films as shown in figure 3. When the thin film is stiffer than the substrate, as in the case of Ru thin film, the deviation from linearity is in the negative direction while deviation in the positive direction occurs when the substrate is stiffer than the thin film, i.e. for Au thin film. However, the linearity of the contact stiffness with increasing indentation depth is maintained when the moduli of the substrate and the thin film are close to each other, i.e. for Au-50%Pt alloy.

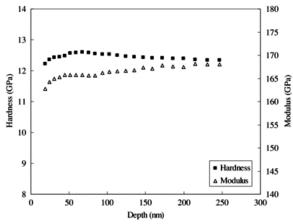


Figure 2. Hardness and modulus of the silicon substrate measured by nanoindentation.

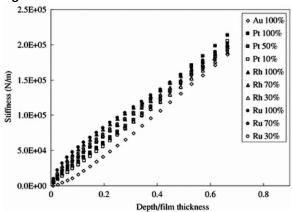


Figure 3. Contact stiffness versus indentation depth. Film thickness is 300 nm. The percentage in legend means the atomic percent of the elements in alloy with Au.

3.2. Elastic modulus and hardness of metals and metal alloys

Figures <u>4</u> and <u>5</u> are plots of the hardness and modulus of 300 nm thickness thin films as a function of the indentation depth (table <u>3</u>). A suggested rule of thumb for the hardness measurement of thin film is that the indentation depth should be less than one-tenth of the film thickness in order to exclude the effect of the substrate. However, the values of the hardness shown in figure <u>4</u> vary with increasing indentation depth even at the depth below one-tenth of the film thickness. The very small thickness of films, i.e. 300 nm, might be the reason for this since the effect of the substrate would be considerably

more on the thinner film. At shallow depth, where the effect of the substrate is smaller, Ru has the highest hardness of ~15 GPa, and Rh is the next with a hardness of ~10 GPa. Thereafter, Pt and Au have hardness of ~5 GPa and ~1 GPa, respectively. When Ru, Rh and Pt are alloyed with Au, their hardness decreases and the decrease is greater with the higher atomic percent of Au due to the lower hardness of Au.

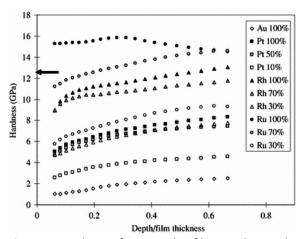


Figure 4. Hardness of various thin films on the Si substrate. The arrow mark indicates the hardness of the Si substrate. Film thickness is 300 nm.

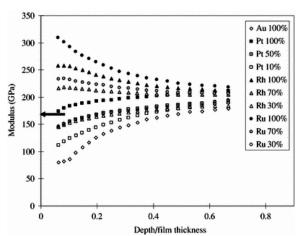


Figure 5. Modulus of various thin films on the Si substrate. The arrow mark indicates the modulus of the Si substrate. Film thickness is 300 nm.

Table 3. Elastic modulus and hardness of metals and metal alloy thin films.

	300 nm film thickness		1500 nm film thickness	
	Modulus (GPa)	Hardness (GPa)	Modulus (GPa)	Hardness (GPa)
Au	86.31	1.04	99.64	0.81
Pt	183.77	5.39	190.52	4.75

Rh	256.86	9.75	298.52	8.96
Ru	292.25	15.28	347.47	15.09
Au- 30%Rh	153.30	4.87	155.86	3.58
Au- 70%Rh	217.57	9.57	234.95	7.99
Au- 30%Ru	154.83	6.18	168.92	6.69

Note: The values were measured at 24 nm for 300 nm thickness and 120 nm for 1500 nm thickness films, respectively.

Compared to the hardness of the Si substrate, which is marked as an arrow in figure 4, only pure Ru thin film has higher hardness at an indentation depth below one-tenth of the film thickness. As the indentation depth increases, the hardness of pure Ru thin film decreases slightly but other thin film's hardness increases. It appears that this trend is quite reasonable considering the influence of the substrate as well as substrate's hardness. However, Saha and Nix¹⁷ noted that the hardness increase of soft thin films on the hard substrate with increasing indentation depth is an artifact, which is caused by inaccuracies in the determination of the contact area during indentation due to build of pile-ups at the edges of indentation. In the case of soft thin film on the hard substrate, most of the plastic deformation is confined in soft thin film until the indentation depth reaches close to the film/substrate interface, so that the effect of the substrate on the hardness of the soft thin film is minimal.¹⁷ When the thin film is harder than the substrate, the plastic deformation is not confined in thin film causing a decrease in the hardness with increasing indentation depth as in the case of pure Ru thin film (figure 4).

For the elastic modulus measurement of thin film, the one-tenth rule is not expected to apply since the elastic deformation field from the indentation can easily extend into the substrate. This is more likely to be the case with the thinner film thickness. Therefore, the effect from the substrate is generally more pronounced in the modulus measurement as compared to the hardness measurement. Figure 5 clearly shows this greater effect of the substrate on elastic modulus such that the measured modulus values of all thin films converge at ~0.5 depth/film thickness. Only at the shallow depths, the moduli of thin films separate from each other. Overall, the Ru thin film has the largest modulus, and moduli of Rh, Pt and Au films are next in the descending order. The alloys with Au of each three noble metals show smaller moduli as compared to the pure metals due to the lower modulus of Au. At a depth of ~20 nm, the measured elastic moduli were 80, 173, 275 and 309 GPa for Au, Pt, Rh and Ru thin films, respectively. These values are close to the reported elastic moduli of these metal films (i.e. 78 GPa for Au, 168 GPa for Pt, 275 GPa for Rh and 447 GPa for Ru) except for the Ru thin film. The difference in moduli between the Ru/Si system and Ru might have been due to the effect of the substrate in the indentation measurement, i.e. due to the largest difference in moduli between the thin film and the substrate.

The 300 nm thickness of the film was chosen since this film thickness was used in an actual MEMS device developed in a previous study. However, this very thin film thickness exaggerates the effect of the substrate, which restricts the indentation measurement at smaller depths in order to reduce/minimize the effects from the substrate. Moreover, the indentation measurement at a very

shallow depth/film thickness also is not preferred due to the other uncertainties (e.g. pile-up at edges of indent). Thus, the thicker films were also manufactured and subjected to the indentation measurement to minimize the effect of the substrate in order to obtain hardness and modulus data at shallow depth/film thickness. Figures 6 and 7 show the hardness and modulus data as the function of depth/film thickness for the 1500 nm thickness films. Since the maximum load of the DCM option in the nano-indenter apparatus was 10 mN, the maximum measurable depth was limited to 0.17 depth/film thickness. In spite of this limitation, the hardness is almost constant as the indentation depth increases after an initial instability. On the other hand, the measured modulus still shows the effect of the substrate as the indention depth increases, but the effect is relatively less than in the case of 300 nm thin films. For instance, the modulus of Ru at shallow depth is about 410 GPa, which is much closer to the expected value of 447 GPa as compared to the measured modulus (309 GPa) from the 300 nm thin film.

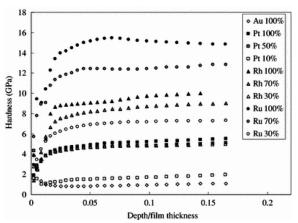


Figure 6. Hardness of thicker films (~1500 nm thickness).

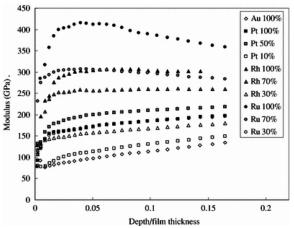


Figure 7. Modulus of thicker films (~1500 nm thickness).

3.3. Resistivity of metals and metal alloys

As mentioned earlier, low resistivity and low hardness are the desired properties of the contact material in MEMS switches to have low contact resistance. Therefore, Au is typically used as electric contacts in MEMS switches; however, it has shortcomings stemming from its low hardness that causes wear, deformation and adhesion during cycling and eventually the failure of MEMS switches. Therefore, one of the selection processes for an alternate material for electric contacts in MEMS would require at least

the examination into the interrelationship between resistivity and hardness properties besides several other factors, such as increased wear resistance, low susceptibility to oxidation and contaminant formation, etc. As stated earlier, the goal of the present study was to examine the first two factors, i.e. resistivity and hardness, as a first step for three noble metals and their alloys with gold. It should be noted here that the electrical resistivity is not a direct indication of the contact resistance, which can be influenced by other factors such as the formation of surface oxide. However, the measurement of actual contact resistance requires more complex experimental setup and therefore only the resistivity was obtained in the present study as a starting point.

Figure <u>8</u> shows the interrelationship between the measured electrical resistivity and hardness for ten metal and metal alloy films for their possible application as the contact material in a MEMS switch. This figure shows these two measured data for both 300 nm and 1500 nm thin films. The hardness values in the figure are at 24 nm (0.08 depth/film thickness) and 120 nm (0.08 depth/film thickness) indentation depths for 300 nm and 1500 nm thin films, respectively. Although there are differences in the hardness between two film thicknesses, i.e. the maximum difference is 1.01 GPa for the Rh-70%Au film system, the overall trend is similar. The alloying of all three noble metals of the present study with gold generally increases their resistivity and decreases their hardness.

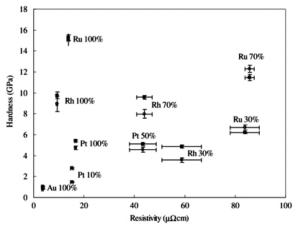


Figure 8. Electrical resistivity versus hardness for various metal films on the Si substrate. Solid squares indicate 300 nm thickness films and hollow circles are for 1500 nm thickness films.

The resistivity is one of the important factors that affects directly the contact resistance of a switch, and hardness is directly related to wear resistance in MEMS devices. The data similar to as shown in figure $\underline{8}$ would thus provide basic guidelines in the selection of electric contact materials for MEMS switches. This approach was indirectly employed in a previous study where MEMS switches were fabricated with contact materials of Au or Au-6.3%Pt alloy. The average contact resistance increased from $1.17~\Omega$ with Au to $1.87~\Omega$ with Au-6.3%Pt alloy, but there was about 2.7 times increase in 'hot-switched' life by alloying Au with Pt due to an increased hardness and wear resistance. However, the MEMS switch with Au-6.3%Pt alloy exhibited an increase in the contact resistance with increased number of switch cycles. This increase was most probably due to the formation of the contaminant film layer. Therefore, several other factors have to also be included; however, characterization of the interrelationship between hardness and resistivity similar to the present study is the first essential step in material selection methodology for electric contacts in MEMS switches.

4. Conclusions

One important requirement in the material selection processes for electric contacts in RF MEMS is the availability of a resistivity versus hardness relationship, since it provides the guidelines and assessment of contact resistance and wear/deformation/adhesion characteristics. For this purpose, thin films of three noble metals, platinum (Pt), rhodium (Rh) and ruthenium (Ru), were investigated using the nanoindentation technique to observe the influence of alloying them with gold (Au). The interrelationship between resistivity and hardness was established for three levels of alloying of these metals with gold. In addition, thin films of gold (Au), platinum (Pt), ruthenium (Rh) and rhodium (Ru) were characterized to obtain their baseline data for comparison. All films were deposited on the silicon substrate. When Ru, Rh and Pt are alloyed with Au, their hardness generally decreases but resistivity increases. This decrease and increase were, in general, dependent upon the amount of alloying. The interrelationship between resistivity and hardness, similar to that developed in the present study, could be used as a first step in the selection methodology of electric contact materials for RF MEMS switches.

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