Achieving Ultralow Wear with Stable Nanocrystalline Metals

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Recent work suggests that thermally stable nanocrystallinity in metals is achievable in several binary alloys by modifying grain boundary energies via solute segregation. The remarkable thermal stability of these alloys has been demonstrated in recent reports, with many alloys exhibiting negligible grain growth during prolonged exposure to near-melting temperatures. Pt–Au, a proposed stable alloy consisting of two noble metals, is shown to exhibit extraordinary resistance to wear. Ultralow wear rates, less than a monolayer of material removed per sliding pass, are measured for Pt–Au thin films at a maximum Hertz contact stress of up to 1.1 GPa. This is the first instance of an all-metallic material exhibiting a specific wear rate on the order of \(10^{-9}\) mm\(^3\) N\(^{-1}\) m\(^{-1}\), comparable to diamond-like carbon (DLC) and sapphire. Remarkably, the wear rate of sapphire and silicon nitride probes used in wear experiments are either higher or comparable to that of the Pt–Au alloy, despite the substantially higher hardness of the ceramic probe materials. High-resolution microscopy shows negligible surface microstructural evolution in the wear tracks after 100k sliding passes. Mitigation of fatigue-driven delamination enables a transition to wear by atomic attrition, a regime previously limited to highly wear-resistant materials such as DLC.

Materials exhibiting ultralow wear rates are generally defined as having a rate of attrition on the order of a monolayer per sliding pass,[1] translating to a specific or volumetric wear rate less than \(10^{-8}\) mm\(^3\) N\(^{-1}\) m\(^{-1}\). Very few materials exhibit this behavior, including diamond-like carbon (DLC),[2,3] graphite,[4–6] metal dichalcogenides like MoS\(_2\) and WS\(_2\),[7–10] metal nitrides,[11] and carbides widely used in bearings and coatings such as Si\(_3\)N\(_4\) and WC, and even semiconductors like GaN.[11] More recently, ultralow wear has been achieved in polymer nanocomposites such as PTFE–alumina.[12,13] As a material class, metals have remained a notable exception to this list.[12,14] Some of these materials, like graphite and metal dichalcogenides, depend on the formation of highly ordered low shear strength lamellar surface films to achieve low friction and high wear resistance,[9,15] and can only accomplish this in specific environments.[16] For metals, surface microstructure evolution—specifically refinement—has been linked to improved friction and wear resistance,[17–20] implying that alloys with grain structures that can resist thermal and mechanically driven grain growth should exhibit lower friction and higher wear resistance than conventional alloys. The potential practical impacts of ultralow wear alloys are significant, perhaps most notably in electrical contacts, where bare metal contacts remain an intrinsic requirement for maintaining electrical conductivity across sliding and rolling interfaces.[21,22]

Achieving ultralow wear rates with metals requires the mitigation of deformation resulting from cyclic applied contact stresses, which can lead to evolution of the microstructure, defect nucleation, and eventual emission of wear particles. Hall–Petch strengthening via reduction in grain size is a primary mechanism to achieve higher hardness in metals,[23] which has historically been correlated, though not causally, to increased wear resistance for a wide range of materials.[24] When applied surface stresses do not regularly exceed the flow stress of metals in sliding contact—as can be the case with nanocrystalline metals, even accounting for surface roughness—wear becomes primarily a fatigue-dominated process,[25–27] Fatigue wear depends on the formation of cracks through repeated cycling of contact stresses, leading to the formation of wear particles via cohesive failure. This regime is typically categorized as the “delamination wear regime.” While most coarse-grained metals undergo dislocation-mediated plasticity, cell formation, and grain refinement under cyclic loading, nanocrystalline metals with unstable grain boundaries (GBs) can undergo grain growth that facilitates cracking.[28] Our motivation was to

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demonstrate that improving the thermo-mechanical stability of nanocrystalline alloys can mitigate stress-driven microstructure evolution at elevated contact stresses, and ultimately suppress delamination wear.

Alloying is a classic approach to stabilizing grain structures in metals via solute drag to retard motion of GBs or Zener pinning through formation of second-phase precipitates. In contrast to these methods of kinetically limiting grain growth, recent models suggest new thermodynamic routes for achieving stable nanocrystallinity through alloying.

In accordance with the Gibbs adsorption equation, the preferential segregation of solute to GBs (when the chemical potential is increased at a fixed temperature and pressure) leads to a reduction of the boundary energy, and thus the driving force for grain growth. If the energy advantage from segregation is strong enough, substantial reduction or even elimination of the thermodynamic driving force for grain growth can be achieved. This will lead to either a slowing of the grain growth kinetics or the thermodynamic stabilization of the nanocrystalline grain structure. The effect will be greatest in alloys with strong boundary segregation, causing segregated GBs to be energetically preferable to solid solutions or second phases.

Platinum and gold alloys were proposed as one of several binary metal alloys theoretically exhibiting the thermodynamic features needed for stable nanocrystallinity. These alloys have the advantage that the constituent materials are available in high purity, and their shared nobility obviates the added complexity of oxide formation on the analysis of GB stability. In addition to the benefits for electrical contact applications where oxidation is problematic, previous studies on non-noble nanocrystalline systems have shown oxygen uptake as a key confounding factor in the interpretation of stability. The role of lattice strain on segregation behavior in this alloy is also minimal, with a relatively small elastic mismatch of 4% between Pt and Au. Here we present evidence that a solid solution alloy of Pt and Au (Pt$_{90}$Au$_{10}$, hereafter denoted Pt–Au) to highlight the solution-state of Au in Pt, predicted to have unusually high nanocrystalline thermal stability, exhibits ultralow wear rates. This suggests that the primary wear mechanism found with traditional nanocrystalline alloys having specific wear rates as low as $10^{-6}$ mm$^3$ N$^{-1}$ m$^{-1}$, i.e., fatigue-driven delamination wear, can be effectively inhibited or greatly suppressed in metals. As such, Pt–Au alloys exhibit wear rates comparable to the most wear-resistant materials in nature.

We deposited films of Pt–Au by magnetron-deposition of a mixed Pt and Au target (see below). Representative scanning TEM high-angle annular dark-field (STEM-HAADF) image for a Pt–Au film in cross section, and STEM energy-dispersive X-ray spectroscopy (STEM-EDS) plan-view composition map and line scan are shown in Figure 1A,B. The Pt–Au films exhibit a columnar structure with large grain aspect ratios, typical of metal films produced by magnetron sputter-deposition. As such, reported grain sizes are the average columnar grain diameters ($=40$ nm), based on plan-view images of films sectioned near the middle of the film thickness. Solute enrichment at boundaries and triple junctions was observed, as well as solute-denuded zones that appeared in bulk grains adjacent to the segregated boundaries. In these films, it was observed that Au segregated to GBs and free surfaces, with highly heterogeneous distributions. A comprehensive thermal stability investigation on these films is reported elsewhere, but we note here that these films were shown to have extraordinarily stable nanocrystallinity, with no evidence of grain size evolution after annealing as much as 1 week at temperatures as high as 500 °C. Abdeljawad et al. utilized a diffuse interface modeling framework to examine GB segregation in Pt–Au, and generally in immiscible nanocrystalline alloys. Their work provides additional insights about the combined effects of GB solute segregation and bulk phase separation, and the relative role of each on the microstructural evolution of nanocrystalline alloys.

While the thermal stability of these alloys has been demonstrated, their stability under mechanical stress has not been previously investigated. We show in the following that our Pt–Au nanocrystalline films are remarkably resistant to

Figure 1. A) STEM-HAADF images of a cross-sectional view of as-deposited Pt–Au films. B) Plan-view EDS composition map showing heterogeneous Au segregation. Also shown is a quantitative line scan of Au concentration across the dashed line showing preferential segregation (i.e., above the global mean composition) to grain boundaries.
growth under cyclic applied shear stress. Nanocrystalline mechanical stability is most clearly evident in the friction and wear behavior of these films, exhibiting wear rates of $3 \times 10^{-9} \pm 1 \times 10^{-9}$ mm$^3$ N$^{-1}$ m$^{-1}$ and friction coefficients of $\mu = 0.2-0.3$; these results are discussed in more detail below. Details of this method for determining wear rates are provided elsewhere.[31] Notably, the Pt–Au films even outperformed the harder sapphire and Si$_3$N$_4$ counterparts. In all wear experiments, 3.2 mm diameter spheres were loaded in reciprocating contact in ambient laboratory air (varying in the range of 12–17% over each week-long experiment, 20°C) against a ~2 µm thick Pt–Au film deposited on a 440PH stainless steel substrate using a stroke length of 1 mm, in bidirectional sliding at 1 mm s$^{-1}$, for $10^5$ sliding passes ($5 \times 10^5$ bidirectional cycles). A constant normal force of 1 N was applied to the contacts, corresponding to Hertzian maximum stresses of 1.1 GPa for both sapphire and Si$_3$N$_4$ spheres.

To demonstrate the combined thermo-mechanical stability of Pt–Au, a specimen was annealed in ultrahigh vacuum for 1 day at 500 °C, followed by friction and wear experiments over the same number of cycles. The cycle average friction coefficient data for the annealed film as compared to the as-deposited film are plotted in Figure 2A, and show minimal differences with a sapphire ball. When a Si$_3$N$_4$ ball was used instead of sapphire, the friction progressively increased, likely a result of the much higher roughness of Si$_3$N$_4$ as compared to sapphire; the root-mean-square surface roughness of the sapphire and Si$_3$N$_4$ balls was 10 and 200 nm, respectively. A friction coefficient of $\mu \approx 0.3$ is consistent with reports of nanocrystalline metals.[17,48,52] The specific wear rates were identical for both annealed and as-deposited Pt–Au wear tracks, with a value of $K = 3 \times 10^{-9} \pm 1 \times 10^{-9}$ mm$^3$ N$^{-1}$ m$^{-1}$, low enough to identify atomic attrition as the likely dominant wear mechanism.[16] Specific wear rates and wear rate uncertainty were based on a volumetric material loss method,[53] where a scanning white light interferometer (Bruker Contour GT) was used to measure the entirety of each wear track. The average cross-sectional area of the wear track ($A$), wear track length ($L$), total sliding passes ($N$), and contact force ($F_n$) were used to calculate the equivalent volumetric specific wear rate using the expression $K = \frac{A \cdot L}{F_n \cdot N \cdot L}$; an established method described in greater detail by Erickson et al.[54] Friction results were reproducible across multiple specimens and wear tracks, but quantification of such low wear rates required extremely long tests and therefore was not performed on all samples.

A comparison of the friction and wear performance of Pt–Au with respect to state-of-the-art tribological materials is presented in Figure 2B, including conventional (i.e., not stabilized) nanocrystalline metals and nonconductive polymer and ionic solid nanocomposites.[2,9,12,14,49,55,56] It is worth mentioning that while many composites require environmental control to achieve the lowest reported wear rates, such as ultrahigh vacuum for MoS$_2$ and various environments for DLC films,[9,56] the remarkably low wear rates of these Pt–Au films were obtained in laboratory air.

In studies assessing wear of materials, harder counterparts are generally used so that wear is predominantly due to the material or coating of interest. Interestingly, for these Pt–Au thin films, we found that the specific wear rates (i.e., aggregate volume lost) were lower than that of the much harder sapphire sphere (Figure 3). Neither cross-sectional STEM (Figure 3D) nor plan-view SEM (Figure 3E) of the wear track surface suggested the presence of surface contaminants, and molecularly thin films that are non-load-bearing would not significantly affect stress-driven fatigue wear of the load-bearing metal film. Even in cases where vapor-phase, thin-film lubrication was implemented in sliding metal contacts, the lowest reported wear rates were about 100x higher than what we report here, and there was consistent evidence of wear debris and surface morphologies associated with high-cycle fatigue and delamination wear.[14,26,57]

Similar experiments against Si$_3$N$_4$ spheres are shown in Figure 4. While the wear rates for both Pt–Au and Si$_3$N$_4$ were substantially higher than the sapphire case, with evidence of plastic deformation and ploughing, Figure 4 shows that the wear rate of the Pt–Au was nearly an order of magnitude lower than the Si$_3$N$_4$. In Figure 2A, where we show the friction coefficient for the case of the Si$_3$N$_4$ sphere, the gradual increase in friction coefficient can be associated with transfer and embedding of Si$_3$N$_4$ wear particles into the Pt–Au film, resulting in higher
roughness and a transition to sliding of Si$_3$N$_4$ against Si$_3$N$_4$ debris. SEM and EDS surface analysis (Figure 4D,E) reveal significant transfer and embedding of the Si$_3$N$_4$ into the Pt–Au film.

The maximum Hertzian contact stresses of about 1.1 GPa in friction experiments are approximately equal to, or just below, the fatigue endurance limit of these Pt–Au films, and well below the measured nanoindentation hardness of 7.1 ± 0.4 GPa; methods and additional results are provided in Supporting Information. By comparison, typical hardness values for Si$_3$N$_4$ and sapphire are 15 and 25 GPa, respectively.[58,59] The endurance limit for Pt–Au was found using 5 µm free-standing films having microstructures equivalent to those of the films used in the friction experiments. Nanoindentation hardness was measured on the same Pt–Au films used in friction experiments, in the as-deposited and annealed states. We hypothesize that because the sapphire balls were relatively smooth, contact stresses at asperities remained below the fatigue endurance limit of Pt–Au. As such, crack nucleation/propagation, stress-driven microstructure evolution, and grain growth were inhibited, suppressing the fatigue-driven wear. We hypothesize that the higher measured wear rate of Si$_3$N$_4$ was due to higher fatigue wear of the ceramic ball, leading to the observed transfer of material and overall volume gain inside the Pt–Au wear track (Figure 4E).

We note that an ultralow wear rate is not necessarily concomitant with a low friction coefficient,[2] as wear is largely a property of the bulk while friction coefficient is a property of the shear layer, which in instances of ultralow wear is molecularly thin. In instances of ultralow wear, friction coefficients are often largely dependent on the presence of adsorbates on the film surface, which can significantly affect the shear strength of the sliding interface. While an increase
in contact shear strength due to a thin film of adventitious carbon adsorbates can certainly enhance wear rate by amplifying stress, in this case, any such adsorbates did not compromise the ultralow wear of the load-bearing Pt–Au film, which is a further testament to the impressive wear resistance of this alloy.

Ultralow wear rates on the order of $10^{-9}$ mm$^3$ N$^{-1}$ m$^{-1}$ were measured for highly stable nanocrystalline Pt–Au thin films in lab air. These results suggest important and interesting directions for future research. Technologically, the demonstration of a noble metal alloy with an extremely low wear rate in nonlubricated sliding and extraordinary thermo-mechanical stability has far-reaching implications, particularly in an electrical contact industry driven by increasing demand for greater efficiency in the use, storage, and transmission of energy. An additional benefit for electrical contacts is the electrical resistivity of these coatings (measured to be approximately 30 nΩ m; see Supporting Information), which was only 1.8x higher than that of coarse grained pure Cu. One noteworthy example of an application where these coatings may have high immediate impact potential is in the developing field of nano- and micro-electromechanical devices. The proliferation of gigahertz nano-/micro-switches remains fundamentally limited by stress- and temperature-driven grain growth, oxidation, and the formation of insulating organic films. A highly stable nanocrystalline noble metal alloy could address persistent roadblocks to widespread adoption of this technology. This demonstration of fatigue resistance and high strength of a stable nanocrystalline alloy also suggests promising opportunities for this class of alloys as structural material.

Figure 4. Surface topographical area scans of: A) the worn Si$_3$N$_4$ ball and B) the Pt–Au wear track near the end of the track after 100k sliding passes. C) Representative cross-sectional wear depth plots of the ball and Pt–Au wear track. D) Plan-view BSD and E) EDS of the wear track surface.
Experimental Section

Materials Synthesis: Pt-10 at% Au thin films (i.e., Pt–Au) were deposited by direct current magnetron sputtering using a cryopumped vacuum system with a base pressure of 2 × 10^{-7} Torr. A single, high purity Pt–Au target (Refining Systems) was used for all depositions. This target was pre-sputtered for ∼5 min prior to the start of each deposition to avoid incorporation of adventitious carbon or other impurities in the deposited films. Ultrahigh purity Ar (99.999%) was used for all sputter processes, and substrates did not exceed 50 °C during deposition. A quartz crystal monitor was used to determine film thickness with Angstrom-level precision. Thickness calibrations conducted prior to depositions utilized a calibrated DEKTAK 3 (Veeco; Woodbury, NY) surface profilometer and cross-sectional TEM.

Film composition was verified by wavelength-dispersive spectroscopy. A JEOL model JXA-8530F Microprobe operating at 7.0 keV incident energy (20 nA, 2 μm beam diameter) was used to determine the composition of a 1 μm thick Pt–Au film sample. The average of 50 spaced measurements demonstrated a film composition of Pt$_{90}$Au$_{10}$. Uncertainty in the measurement was ±0.5 at% with 95% confidence.

Tribological Testing: A custom linear reciprocating tribometer was used for friction and wear measurements, similar in design to the instrument described by Erickson et al.[54] This instrument relies on two pairs of leaf-springs to generate linear elastic deformation in two independent axes with negligible rotation in response to the friction and normal contact forces experienced by the spherical probe (3.2 mm diameter sapphire ball lens) as it slid back and forth on a Pt–Au–coated substrate. A piezo positioner maintained the desired contact load within an error of 1%, and a precision stepper motor linear stage (PLS-85 by Physik Instrumente) generated the sliding motion. The relative humidity (RH) was maintained in the range of 12–17%, and the ambient temperature at 20 ± 1 °C. Specific wear rates and wear rate uncertainty were based on a volumetric material loss method,[53] where a scanning white light interferometer (Bruker Contour GT) was used to measure the entirety of each wear track.

Microstructure Characterization via TEM: An FEI TitanTM G2 80–200 STEM with a Cs probe corrector and ChemiSTEM technology (X-FEGTM and SuperXTM EDS with four windowless silicon drift detectors) operated at 200 kV was used in this study. The STEM images were recorded by using a HAADF detector. EDS spectral imaging was acquired as a series of frames where the same region was scanned multiple times.[60] Au L lines and Pt L lines were used for constructing the EDS maps of Au and Pt, respectively. Since the Au L lines overlap with the Pt L lines significantly, the EDS spectra were deconvoluted pixel-by-pixel using pure spectra of Au and Pt as reference spectra before map construction. Atomic composition was calculated using the Cliff and Lorimer method.[61] Sample annealing was carried out in a tube furnace in air. The TEM samples were prepared using the focused ion beam (FIB) method.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

friction, metals, nanocrystalline, stable, wear

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