Two-dimensional (2D) materials are known to have low-friction interfaces by reducing the energy dissipated by sliding contacts. While this is often attributed to van der Waals (vdW) bonding of 2D materials, nanoscale and quantum confinement effects can also act to modify the atomic interactions of a 2D material, producing unique interfacial properties. Here, we demonstrate the low-friction behavior of magnetene, a non-vdW 2D material obtained via the exfoliation of magnetite, showing statistically similar friction to benchmark vdW 2D materials. We find that this low friction is due to 2D confinement effects of minimized potential energy surface corrugation, lowered valence states reducing surface adsorbates, and forbidden low-damping phonon modes, all of which contribute to producing a low-friction 2D material.

INTRODUCTION

Two-dimensional (2D) materials demonstrate distinct properties from their bulk forms owing to their confinement in the third dimension, producing forbidden phonon modes, confined electron waveforms, mechanical anisotropy, and high volumes of asymmetrically bonded surface atoms among other unique effects (1). As a result, a variety of application-favorable characteristics such as low defect densities (2), supercapacitance (3), high in-plane strength (4), and superlubricity (5) can be found in 2D materials. The vast majority of 2D materials are produced by cleavage of weak van der Waals (vdW) bonds between layers. However, a series of non-vdW–bonded 2D materials from transition metal oxides have been recently exfoliated (6–12). Of particular note, the iron oxide materials including hematene (Fe$_2$O$_3$), magnetene (Fe$_3$O$_4$), and chromiteen (FeCr$_2$O$_4$) are 2D versions of bulk ores, which present thicknesses down to a monolayer despite featuring covalent bonding out of plane (6–8). These 2D materials have exhibited distinct magnetic and photocatalytic properties from their bulk counterparts due to preferred magnetic spin moments by planar confinement and increased bandgap by confined spin polarization, respectively (6–8).

While confinement in these non-vdW materials have produced enhanced properties owing to their 2D electron waveforms, one application that remains dominated by vdW materials is lubricity. Graphene, MoS$_2$, and other vdW 2D materials can reduce the contact friction by several orders of magnitude compared to conventional lubricants (13). This is attributed to their weak vdW bonding between adjacent 2D layers in the (001) direction, which facilitate incommensurate contacts between adjacent layers and layer-by-layer sliding when subjected to shear forces (14). However, monolayer graphene still exhibits phenomenal lubricating behavior despite, by definition, not exhibiting this vdW bonding between 2D layers (5,13). This indicates that other signatures of 2D materials, such as the previously suggested quantum confinement states (15), low interfacial shear strength and roughness (16), minimized residual stresses (17), and uniform defect-free surfaces (18), could be applicable to a wider classification of 2D materials to produce similarly low friction. We demonstrate through nanoscale friction studies the appearance of low friction for magnetene, a non-vdW 2D iron oxide, upon exfoliation to its 2D form. We further note the minimized potential energy landscape of the exfoliated plane, internal shift of valence states that reduces surface adsorbate, and emergence of low-damping forbidden phonon modes present in magnetene. These three phenomena all contribute to the low friction of magnetene due to the unique 2D signature of this non-vdW material.

RESULTS

Non-vdW 2D iron oxides

Exfoliation of non-vdW 2D iron oxides has been recently demonstrated; hematene (Fe$_2$O$_3$; Fig. 1, A, D, G, and H) (6), magnetene (Fe$_3$O$_4$; Fig. 1, B, E, H, and I) (7), and chromiteen (FeCr$_2$O$_4$; Fig. 1, C, F, I, and L) (8) are 2D iron oxide nanomaterials that can be cleaved by liquid sonication exfoliation along their respective weak planes, producing uniform atomic layers. The flakes are produced from bulk iron oxide ores in a sonication medium of N,N-dimethylformamide (DMF); full details on non-vdW exfoliation are described in previous studies (6–8). The resulting exfoliated flakes show a lateral size on the order of several micrometers (Fig. 1, A to C, and Fig. S1) and are commonly few-layer thicknesses on the order of a few nanometers (Fig. 1, D to F, and Fig. S2). The liquid exfoliation is found to split the basal planes without introducing substantial defects or disrupting the lattice structure in-plane as seen in Fig. 1 (G to I). Molecular schematics of the most prevalently exfoliated basal lattice structures, (001) rhombohedral for hematene (6), (110) inverse spinel for magnetene (7, 19), and (111) spinel for chromiteen (8, 20), are shown in Fig. 1 (I to L). 2D confinement of the three materials can be noted by peak broadening in the x-ray diffraction (XRD) patterns (fig. S3) (21) and blue shifts in the ultraviolet (UV)/visible absorbance
The stoichiometries of the three non-vdW materials are confirmed to be preserved during exfoliation by energy dispersive spectroscopy and electron probe microanalysis (fig. S5 and table S1).

**Nanoscale friction of non-vdW 2D materials**

Friction was measured along the basal planes of the three non-vdW materials as well as two common vdW materials, graphene and MoS₂, by friction force microscopy (FFM) with a diamond-tipped cantilever. The average friction signal from at least 12 flakes of each sample is shown for the five 2D materials as a function of normal load in Fig. 2A. FFM was additionally performed on the bulk ores of the non-vdW materials (several millimeters in thickness, fig. S6), and comparative friction forces for the 2D and 3D forms are shown in Fig. 2B. Representative topographic and friction maps of the 2D materials can be seen in Fig. 2 (C to L, respectively). All of the 2D flakes were selected to be 2 nm < t < 8 nm in thickness, which is thin enough to exhibit confinement effects (23, 24) while being thick enough to avoid the frictional layer dependence that occurs within the one- to five-layer regime (13) as noted in fig. S7. In addition, the contact conditions were confirmed to be consistent across samples by examining the diamond-tipped cantilever and the friction of the SiO₂ substrate in the FFM images across sequential tests (figs. S8A and S9). We found that the non-vdW material magnetene exhibits statistically similar low friction to multilayer graphene and MoS₂ with a near-zero slope and friction coefficients for the 16-nN loads of \( \mu_{\text{Mag}} = 0.08 \pm 0.01, \mu_{\text{Gra}} = 0.06 \pm 0.02, \) and \( \mu_{\text{MoS}_2} = 0.07 \pm 0.03 \) (Fig. 2A). This is contrary to hematene and chromiteen, which show higher friction forces that increase with normal load and coefficients of friction for the 16-nN load of \( \mu_{\text{Hem}} = 0.29 \pm 0.04 \) and \( \mu_{\text{Chr}} = 0.49 \pm 0.07, \) respectively. The statistically similar friction behavior of graphene and magnetene was also observed under an inert low...
humidity argon environment, suggesting that the environmental influence is not the dominant mechanism (fig. S10). It should be noted that, while surface roughness plays a prominent role in nanoscale friction, the surface roughness of the five 2D materials and the 3D materials was not found to correlate with friction (figs. S11 and S12).

The low-friction behavior of magnetene is markedly different from the other two non-vdW materials as well as its bulk form, magnetite. Despite sharing a similar composition to hematene (Fe₂O₃ and Fe₃O₄) and an isostructural spinel structure with chromiteen, 2D magnetene is the only non-vdW material tested to exhibit this lubricating characteristic. This behavior is especially notable as the low friction is statistically similar to the two vdW materials despite being unable to achieve the vdW sliding that is commonly credited for ultralow friction of graphene and MoS₂.

Evidently, the mechanisms typically associated with macroscopic friction are not dominant in the low friction of 2D magnetene. This is further exemplified by considering intermediate length-scale magnetene and seeing that the friction only begins to decrease when the thickness approaches <15 nm (fig. S13). As such, we instead consider the mechanisms unique to 2D materials including predominant exfoliation planes, quantum confinement, and asymmetric surface bonding. We therefore examine the potential energy corrugation of the exfoliated planes, modified valence states by electron confinement in a quantum well, and forbidden phonon modes by asymmetric surface bonding.

**Potential energy surface corrugation**

The interaction of the diamond atomic force microscopy (AFM) tip on the exfoliated 2D material is determined by the short- and long-range atomic forces of the two materials. The attractive adhesion energy between surfaces can act to increase the contact normal load, which, in turn, increases friction. However, we find no correlation of adhesion energy (fig. S14) or charge transfer (fig. S15) with friction force aside from the notably higher adhesion energy of chromiteen. Instead, we consider the interaction of the tip and 2D material during lateral translation that more closely mimics FFM. Lateral sliding across an atomic lattice can be considered as a series of interferences between atoms spaced apart by gaps between lattice positions. This creates a landscape of localized potential energy peaks and wells.
best known for producing the atomic slip-stick effect (30). The series of peaks and wells for a given material is determined by the surface chemistry, structure, and valence states and is termed the potential energy corrugation of the surface-counter surface contact (31). The potential energy corrugation is an element of the nanoscale friction as related by the Prandtl-Tomlinson model (32) describing the ideal friction at absolute zero and no normal load as seen in Eq. 1

$$\eta = \frac{4\pi^2 U}{k a^2}$$  

(1)

This dimensionless friction parameter ($\eta$) is proportional to the maximum amplitude of the potential energy corrugation ($U$) and inversely proportional to the periodicity ($a$) of peak-peak or well-well. The spring constant, $k$, is determined by three springs in series for the contact, tip, and cantilever stiffnesses (32).

To quantify the potential energy corrugation, density functional theory (DFT) first principles calculations of a diamond lattice sliding across the five nanomaterials can be seen in Fig. 3. The most prevalent basal lattice structures and energetically favorable termination configurations for the 2D materials were determined as (001) for hematene (6), (110)B termination for magnetene (7, 19), (111) Cr/Fe termination for chromiteen (8, 20), and (001) for graphene and MoS$_2$, as shown in Fig. 3A. The potential energy landscape of at least one period for each material is shown in Fig. 3B where the amplitude of surface energy ($U = U_{\text{peak}} - U_{\text{well}}$) and the periodicity of corrugation ($a =$ lateral distance peak-peak) can be identified.

Using the amplitude and periodicity as determined by the potential energy corrugation, we can therefore determine $\eta$ for each material as an idealized atomic surface friction without load using Eq. 1. The calculated values of $\eta$ along with the experimental friction forces at 0 nN load are shown in Fig. 3C and show considerable agreement in their relative magnitudes. We further note two predominant considerations for the interpretation of these potential energy landscapes. First, these atomic-scale interactions only become relevant in the range of forces used for nanoscale contacts and between two atomically flat lattices as per the configuration in FFM of exfoliated 2D materials. Second, the surface lattices that are used in the present calculations are unique to the 2D forms of these materials based on the energetically favorable planes that are exposed during the exfoliation process. For magnetene, the exposed plane is (110) B-terminated spinel as per Fig. 1H, while bulk magnetite shows predominantly (311) and (440) planes (cf. fig. S3), which exhibit a greater potential energy corrugation (fig. S16). Therefore, the low potential energy corrugation of magnetene is unique to the exfoliated plane in its 2D form.

**Quantum well surface chemistry**

The five 2D materials measure <8 nm in thickness, which is the typical threshold where quantum well effects begin to become relevant as the thickness approaches the de Broglie wavelength (23, 24). A
typical feature of quantum wells is altered valence states of the materials due to electron waveform confinement and asymmetric surface bonding (23, 33). Examining the changes to the chemical signature, we perform x-ray photoelectron spectroscopy (XPS) measurements of the 2D materials and their bulk counterparts, as shown in Fig. 4 (A and B, respectively). The Fe2p, O1s, and Cr2p peaks have been indexed to their respective valence states (cf. Materials and Methods) as seen in Fig. 4.

Comparing the 2D and 3D versions of these non-vdW iron oxides, we note two clear differences for the exfoliation of magnetene as compared to chromiteen and hematene. First, the Fe2p spectra of hematene and chromiteen are largely similar to their bulk forms, while magnetene shows a transition from a divalent to trivalent Fe2p state as the Fe$^{3+}$ band is weakened and two strong Fe$^{2+}$ bands are raised. Correspondingly, the magnetene O1s peak confirms this reduced valence showing a predominance of Fe$^{2+}$-O, while the Fe$^{3+}$-O peak is diminished. This indicates that, upon confinement, the collective valence state is reduced from the bulk Fe$^{2.48+}$ to Fe$^{2.19+}$, which is in agreement with previous DFT calculations that showed a decrease from +1.25 $|\epsilon|$ at the Fe octahedral site (mixed Fe$^{2+}$ and Fe$^{3+}$) to +1.1 $|\epsilon|$ (7). The second predominant difference that can be noted by the XPS spectra is the O1s signal. Bulk hematite and chromite show a predominance of the Fe/Cr$^{3+}$-O band at 528 to 529.5 eV in the O1s signal but, upon exfoliation to hematene and chromiteen, the spectra are dominated by the OH$^-$ /O$^-$ defect band at 532 eV. This is rational as the prevalence of highly reactive asymmetric surface bonds created during exfoliation in the DMF are passivated by OH$^-$ /O$^-$ groups and makes up a substantial portion of the surface atomic bonds. However, this effect is reduced for magnetene, which shows O1s spectra dominated by the Fe$^{2+}$-O peak upon exfoliation with the OH$^-$/O$^-$ peak being secondary in intensity.

The exfoliation and quantum well confinement of magnetene therefore produces a material with modified valence states, resulting in a surface with fewer dangling bonds as compared to the other non-vdW 2D materials. As a result, magnetene presents 25 and 34% reduced surface adsorbate bond density as compared to hematene and chromiteen, respectively (table S2). Adsorbate O$^-$ and OH$^-$ passivation defects are known to increase the friction of 2D materials through localized increases in potential energy corrugation, phonon mode mismatch, and modified interaction potentials with the counter surface (34–36). As such, the decreased Fe valence states of magnetene upon exfoliation contribute to preserving the low potential energy surface and low friction of magnetene by reducing adsorbate density.

**Phononic friction damping**

Upon confinement of a material in at least one dimension, it is common for nanomaterials to take on unique forbidden phonon modes because of their predominance of asymmetric and softer bonding of surface atoms (37). We consider the phonon modes by Raman spectroscopy for the three non-vdW materials as compared to their bulk 3D forms (Fig. 5A, index cf. Materials and Methods) and note that the difference in spectra is drastic for magnetene as compared to magnetite, while chromiteen and hematene show similar spectra to their bulk counterparts. Specifically, magnetene shows the promotion of three forbidden Raman modes ($E_u$, $A_1g$, and $2A_1g$) and a marked decrease in $T_{2g}$ relative intensities, while chromiteen and hematene show only minor forbidden modes and intensity variations (tables S3 to S5). The dominant phonon modes for magnetene per the molecular model of a spinel crystal as proposed by Waldron (38) and expanded upon by Verble (39) are shown in Fig. 5B.

According to the phononic damping theorem proposed by Persson (40, 41), the friction can be directly related to the energy transferred...
and damped by phonon modes in the contact material. Sliding energy is transferred from the tip to the 2D material by momentum in atomic collisions that excite the 2D material phonon modes (Fig. 5C). This energy is dissipated through the 2D material phonons, which results in an exponential damping of energy in the tip-surface system. The phononic friction force is thus

\[ F_F = -m_{\text{tip}} \xi v \]  \hspace{1cm} (2)

where \( m_{\text{tip}} \), \( \xi \), and \( v \) are the mass of the sliding tip, average phononic damping coefficient, and sliding velocity, respectively (40, 41). The phononic damping coefficient is determined by the rate of phonon mode propagation in the transverse (parallel to the direction of sliding) and longitudinal (perpendicular to the direction of sliding) directions as seen in Fig. 5D and Eqs. 3 and 4, respectively

\[ \xi_\parallel = \frac{m \omega_\parallel^2 n}{\rho c_T} \]  \hspace{1cm} (3)

\[ \xi_\perp = \frac{m \omega_\perp^2 n}{\rho c_L} \]  \hspace{1cm} (4)

In Eqs. 3 and 4, \( m \) is the mass of the surface atom, \( \omega \) is the excited phonon frequency, \( n \) is the number of atoms per unit area, \( \rho \) is the density, and \( c \) is the sound velocity within the material. It can be further noted that the sound velocity in the longitudinal direction is approximately double that of the transverse direction for most materials (\( c_L \approx 2c_T \)) (42), and as a result, the frictional damping by phonon modes parallel to the direction of sliding is greater than perpendicular modes.

We note that upon exfoliation of magnetene, the phonon modes that appear or increase in relative intensity correlate with nonplanar phonon modes (\( E_u, A_{1g}, \) and \( E_g \)); meanwhile, the relative intensities of the planar phonon modes (\( T_{2g}^1 \) and \( T_{2g}^2 \)) are diminished. For the (110) plane of a spinel crystal as per the exfoliation of magnetene, the planar phonon modes coincide parallel with the direction of sliding (transverse), while the nonplanar modes have wave vectors that are nonorthogonal and contribute to both transverse and longitudinal directions. As the phononic friction force of Eq. 2 is due to the average damping coefficient across all phonon modes, the reduction in planar \( T_{2g}^1 \) and \( T_{2g}^2 \) phonon modes relative to other modes due to the asymmetric soft surface bonding of magnetene therefore produces a material with reduced phononic damping and friction behavior.

**DISCUSSION**

This study demonstrates that the lubricating characteristics that are typical of graphene and other vDW 2D materials can be achieved in...
magnetene, a non-vdW–bonded 2D iron oxide upon exfoliation. We find that this low friction can be attributed to the low potential energy corrugation of the (110)B exfoliation plane, reduced iron valence states and adsorbate density by electron confinement in the 2D material, and nonplanar forbidden phonon modes by asymmetric surface bonding. These three mechanisms all contribute to creating a nanomaterial with low-friction properties, which does not rely on the vdW nature of bonding. We encourage investigation into these materials further to elucidate these fundamental phenomena, which allow the engineering of 2D material properties to create controlled interfaces and frictional contacts.

**MATERIALS AND METHODS**

**Material synthesis**

Magnetite, hematite, and chromite ore samples are ground to fine powder using mortar and pestle, then dispersed in DMF solvent (0.25 mg/ml), and subjected to prolonged ultrasonication (approximately 50 hours). The obtained solution is ultracentrifuged at a relative centrifugal force of 10,000g. The supernatant containing ultrathin 2D sheets is collected, neglecting the thicker sediments, and is vacuum-filtered using membranes of suitable pore size. The filtrate thus obtained is dried under vacuum at room temperature and used for further characterizations.

The 2D flakes were then dispersed in isopropyl alcohol and further bath-sonicated for 45 min and centrifuged at 3000 rpm for 10 min before drop casting for AFM analysis. The supernatant was pulled from the middle of the solution and drop-cast on silicon wafers that were treated by O₂ plasma for 10 min to improve adhesion.

**Spectroscopy and characterization**

Transmission electron microscopy (TEM) measurements were carried out using Titan Themis (Thermo Fisher Scientific, Electron Microscopy Solutions) aberration-corrected electron microscope furnished with Energy dispersive spectroscopy (EDS) probes. XRD was obtained using a Rigaku SmartLab X-ray Diffractometer (Rigaku Corporation, Tokyo, Japan) and was indexed to the corresponding International Centre for Diffraction Data (ICDD) database. The diffraction pattern of hematene, magnetene, and chromiteen can be matched with their bulk crystal structures: hematite (ICDD card 33-664, a = 5.035 Å and c = 13.74 Å), magnetite [Fd-3 m, choice-2 space group (ID: 01-084-2782), a = 8.397 Å; Z = 8], and chromite (ICDD file no. 04-006-2807). UV/Visible Absorbance Spectra were obtained using a Shimadzu 2450 UV-visible spectrophotometer (Shimadzu Corp., Kyoto, Japan). Electron probe microanalysis of the samples was carried out on a field emission Jeol 41 JXA 8530F Hyperprobe (Jeol Ltd., Kyoto, Japan), using five wavelength dispersive spectrometers wavelength dispersive spectroscopy (WDS) and ensured the sample purity of the ores. Raman spectroscopy measurements were carried out using a Renishaw inVia Raman microscope (Renishaw plc, Wotton-under-Edge, UK) with a 50× objective using a He-Ne laser of wavelength of 632.8 nm and with a spot size of 1 μm. The peaks were indexed according to previous studies on bulk ores (43–45).

The Raman spectra of hematene were matched to hematite including two major peaks of A₁g and E₂g modes at 223 and 288 cm⁻¹ (43) and an additional weak mode at 663 cm⁻¹, which is assigned to E₃g, a forbidden vibrational mode (44). The Raman spectra of magnetite are interpreted in terms of the symmetries of the observed modes A₁g, E₂g, and T₂g (45). Three forbidden vibration modes are observed; the E₄g mode at 401 cm⁻¹, the shoulder on A₁g at 610 cm⁻¹, and a new reflected 2A₁g peak at 1290 cm⁻¹. Chromiteen’s spectra are matched to chromite with two prominent peaks at 714 and 570 cm⁻¹ (46).

The chemical environments of the iron, chromium, and oxygen in the samples were interpreted using XPS using a PHI Quantera x-ray photoelectron spectrometer (Physical Electronics Inc., Chanhassen, USA). The Fe₂p peaks for the three materials can be identified as two Fe²⁺ peaks at ~709 and ~712 eV and the Fe³⁺ at ~710 eV, all ±1 eV based on experimental error (47–49). For the O1s peak, three subpeaks can be identified as lattice Fe/Cr²⁺–O and Fe²⁺–O at ~528 and ~530 eV, respectively, and defect-passivated Fe/Cr-OH at ~532 eV ±1 eV due to experimental error (50, 51). Chromium is found to be exclusively in the Cr³⁺ valence with Cr₂p peaks at 576 and 586 eV (48).

**FFM and AFM**

AFM imaging was performed through tapping and contact mode imaging using Cypher and MFP-3D atomic force microscopes (Asylum Research, Oxford Instruments, Goleta, USA). Sharp diamond-tipped cantilevers were used throughout all of the present experiments (K-TEK Nanotechnology, Wilsonville, USA) with either a low stiffness (D10, K = 30 mN/m) for imaging and friction or higher stiffness (D160, K = 2 N/m) for adhesion testing (cf. fig. S8). FFM was performed using the normal and lateral signals of the AFM for which the cantilever spring constants were calibrated using the normal (52) and lateral (53) Sader’s methods and lateral sensitivity as per the test probe method (54). The same cantilever was used for an entire series of tests (cf. fig. S9), which showed no evidence of wear of the cantilever tip or 2D materials. Tests under an inert environment were performed under flowing 99.9% argon using a custom environmental control box within the AFM enclosure.

**Density functional theory**

Plane wave–based DFT simulations were carried out using the Vienna ab initio simulation package (VASP) software package. Interactions between the valence electrons and the ionic core were represented by the projector augmented wave method with Perdew-Burke-Ernzerhof formulation. Kinetic energy cutoffs of 400 eV were used for the wave functions. All calculations were spin-polarized and used DFT+U to account for highly correlated d electrons commonly found in ferrous materials with U = 4 eV for Fe and Cr (55, 56). Brillouin zone integrations were performed over the Γ point. A vacuum layer of 15 Å, based on iterative testing, was added to avoid interaction between periodic surface images. Partial atomic charges were assigned using the Bader charge analysis method implemented by Henkelman’s group (57, 58).

All material systems were modeled periodically, where the surfaces were brought to a separation of 2.54 Å and the interface was allowed to relax in the out-of-plane direction (normal to each surface) for the most stable interfacial separation distance. The diamond tip was cut from a 5-nm radius hemisphere, while the actual contact between the two surfaces was localized to the scale of angstroms due to system size constraints and led to an essentially flat tip surface. The diamond tip was then moved in the in-plane direction by a 0.33-Å step displacement (Δd) and again relaxed in the out-of-plane direction for multiple steps. This simulates a pseudo-sliding motion where the surfaces are allowed to reach equilibrium separation distances at discrete points along the sliding path. Note that this does not simulate a dynamic process such as found in kinetic friction measurements or those with applied loads (no load is applied in the simulations, and the systems are relaxed at absolute zero in a vacuum).
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