The search for new hard materials is often challenging, but strongly motivated by the vast application potential such materials hold. Ti$_3$Au exhibits high hardness values (about four times those of pure Ti and most steel alloys), reduced coefficient of friction and wear rates, and biocompatibility, all of which are optimal traits for orthopedic, dental, and prosthetic applications. In addition, the ability of this compound to adhere to ceramic parts can reduce both the weight and the cost of medical components. The fourfold increase in the hardness of Ti$_3$Au compared to other Ti–Au alloys and compounds can be attributed to the elevated valence electron density, the reduced bond length, and the pseudogap formation. Understanding the origin of hardness in this intermetallic compound provides an avenue toward designing superior biocompatible, hard materials.

INTRODUCTION

In addition to numerous applications in the industrial, automotive, and aerospace fields, Ti has been widely used for implant devices that replace patients’ hard tissues (1, 2). A number of in vivo and in vitro experiments with various grades of Ti concluded that commercially pure Ti is a highly biocompatible material due to the spontaneous buildup of an inert and stable oxide layer (1, 3). Additional properties that make Ti suitable for biomedical applications include its high strength-to-weight ratio (4, 5) and low ion formation levels in aqueous environments (1). Moreover, Ti is one of a few materials capable of osseointegration—the mechanical retention of the implant by the host bone tissue—which stabilizes the implant without any soft tissue layers between the two (6). These properties enable the wide use of Ti for devices, such as artificial knee and hip joints, screws and shunts for fracture fixation, bone plates, pacemakers, and cardiac valve prostheses (7, 8). Not surprisingly, the dental applications of Ti are just as common, including implants and their components, such as inlays, crowns, overdentures, and bridges (1, 9–12).

However, pure Ti is not strong enough for a number of medical devices (13, 14), thus necessitating the development of superior alloys (15–19). Although hardness can be improved by alloying Ti with another element (1), care must be taken to preserve biocompatibility. Previously, a twofold increase in hardness has been achieved by alloying Ti with Cu or Ag (19–22). The use of an alloying element with the same valence as Cu and Ag, but with higher mass density, should result in a higher valence electron density (VED), which would likely lead to higher bond strength and, consequently, enhanced hardness (23, 24). This finding suggests that Au is a suitable alloying candidate to increase the hardness in Ti binary alloys, given its nearly twofold density increase over Cu or Ag (19–22). The current wide use of Au-based implant devices (25–28) is testament to its biocompatibility and corrosion resistance (10).

Biomedical applications of both Ti-rich and Au-rich alloys have been previously explored in detail (19, 25, 29, 30). Although hardness values showed modest increase in both these regimes of the Ti–Au solution [Fig. 1, open circles, reproduced from previous studies (25, 29, 30)], the hardness was comparable to that of Ti–Ag and Ti–Cu alloys (19–22). Here, we present evidence that the hardness varies nonmonotonously in the Ti–Au alloys, and a drastic increase is registered at an intermediate composition, for the cubic compound β-Ti$_3$Au (Fig. 1, full circles). Further evidence from wear experiments reveal that this enhanced hardness is associated with a low coefficient of friction (COF). Experimental observations and theoretical calculations point to three main factors that contribute to the high hardness in β-Ti$_3$Au: the cubic structure with short Ti–Au bonds, the high VED, and a pseudogap evident in the electronic density of states (DOS).

RESULTS AND DISCUSSION

Hardness measurements of the Ti$_{1-x}$Au$_x$ alloys reveal a nonmonotonous change with x (Fig. 1, circles), with hardness values in the composition range 0.22 ≤ x ≤ 0.35, which are about three to four times higher than the hardness value of pure Ti. The maximum hardness of ≈800 HV (Vickers hardness) is reached for x ≈ 0.25, for which the cubic compound Ti$_3$Au forms, in two distinct phases, α and β, with the latter stabilized by the presence of small amounts of carbon, nitrogen, or oxygen (31). This maximum hardness value exceeds that of most biocompatible materials [and even the maximum hardness value of some structural materials, such as pearlitic steels (32, 33)] and is similar to that of both drawn pearlitic and high-carbon martensitic steels (34). Although other metallic alloys and compounds, such as WC, BN, and high-carbon steels show higher hardness values (35–41), they are often not desirable for medical applications because of their high toxicity (42, 43). However, both Ti and Au are biocompatible and have high resistance to in vivo corrosion, suggesting that the resulting alloys would be suitable for biomedical applications (1, 3, 10). Not surprisingly, biocompatibility and corrosion resistance have been confirmed in the Ti$_{1-x}$Au$_x$ alloys for x ≤ 0.40 (Fig. 1, open circles) (44). Here, relative cell viability was examined, as described in Methods. Remarkably, the relative cell viability values 98.7% (for x ≈ 0.25) and 95.9% (for x ≈ 0.50) were found to be much higher than 33.8% in the case of pure Ti. The exceptional biocompatibility of these Ti$_{1-x}$Au$_x$ alloys makes them particularly well suited for a variety of medical applications. To compare the mechanical properties of the


Ti_{1-x}Au alloys with materials typically used for medical applications, we added a hardness–versus–mass density diagram (45) in Fig. 1 (bottom axis). Although Ti_{0.75}Au_{0.25} (or Ti3Au) displays high hardness, its mass density is comparable to that of other commonly used implant materials [Fig. 1, squares (37, 46)]. Moreover, among intermetallic compounds (47–54), Ti3Al has significantly enhanced hardness while preserving its biocompatibility. The only other intermetallic compound with a similar hardness value is Ti3Ir, the biocompatible properties of which remain unknown.

Among the Ti–Au binary compounds, Ti3Au is the only cubic one, which is consistent with high mechanical stability and, therefore, high hardness. A three-dimensional bonding network (similar to that in cubic compounds) is deemed one of the most important parameters enhancing hardness (55, 56). This correlation between the crystal structure and hardness is evident from several known Ti-based compounds: Cubic Ti3Au and Ti3Ir (14-fold coordinated Ti) are the hardest, whereas cubic Ti3Sn (12-fold coordinated Ti) and hexagonal Ti3Al exhibit hardness values comparable to that of pure Ti (Fig. 1, circles). Notably, Ti3Au forms in two cubic crystal structures: α-Ti3Au (Pm3m, Cu3Au type; Fig. 2A) and β-Ti3Au (Pm3n, Cr3Si type; Fig. 2B). β-Ti3Au is expected to have higher hardness, considering the Ti coordination (14) and its Ti–Au bond length $d_{\text{Ti–Au}} = 2.84$ Å (57) being shorter than that of the α-Ti3Au phase [$d_{\text{Ti–Au}} = 2.93$ Å (31)].

A full theoretical understanding of hardness remains challenging because of the inherently complex relationship between elasticity and toughness that defines hardness (58). In alloys, in particular, hardness mostly depends on the underlying crystal structure, atomic bonding, and microstructure (23), making the structural analysis of paramount importance (59, 60). Powder x-ray diffraction (XRD) analysis (Fig. 2C) reveals that Ti_{0.75}Au_{0.25} consists of a majority phase β-Ti3Au (Pm3n; Fig. 2B) (57) along with minute amounts of α-Ti3Au (Pm3m; Fig. 2A) (less than 0.6%) (57) and α-Ti (less than 4%). The majority phase β-Ti3Au has a lattice constant $a = 5.1$ Å, whereas the minority phase β-Ti3Au has a smaller lattice constant $a = 4.1$ Å (31). Because the formation energy of a Burgers vector (a vector denoting the magnitude and direction of the lattice distortion resulting from a dislocation) is proportional to the unit cell parameter, the compound with the larger unit cell parameter β-Ti3Au is expected to have a higher hardness. Furthermore, the two phases differ profoundly in the atomic environment types (AETs) of Au and Ti. In the α-Ti3Au phase (Fig. 2A), both Ti and Au are 12-fold coordinated with a cuboctahedron AET; Au is surrounded by 12 Ti atoms, whereas Ti is surrounded by 8 Ti and 4 Au atoms. By contrast, in the β-Ti3Au phase (Fig. 2B), the two atoms have very different AETs, Au is still 12-fold coordinated but now with an icosahedron AET formed by Ti (Fig. 2B, bottom left), which implies shorter bond lengths. However, Ti is 14-fold coordinated with a 14-vertex Frank-Kasper polyhedron composed of 4 Au and 10 Ti atoms (Fig. 2B, bottom right). As mentioned above, the key differences in the AETs of Au and Ti (coordination number, bond lengths) are consistent with the higher hardness of β-Ti3Au compared to α-Ti3Au. Oftentimes, hardness strongly depends on plastic deformation resulting from the creation and motion of dislocations and defects (23, 61). In α-Ti3Au, both atoms have the same AETs, and it is therefore reasonable to assume that this gives rise to low shear strength, stemming from the low energy needed to create and move dislocations and defects (23). In the β-Ti3Au phase, on the other hand, the different
AETs of Au and Ti introduce higher energy barriers for any dislocation movement; that is, atoms cannot easily slide and break as well as reform bonds, resulting in increased hardness. One may argue further that the presence of small amounts of minority phases $\alpha$-Ti$_{3}$Au and $\alpha$-Ti may favorably interact with $\beta$-Ti$_{3}$Au to further inhibit dislocation formation and dislocation motion, thus increasing the hardness and toughness of the majority phase $\beta$-Ti$_{3}$Au.

To investigate the microstructure of the Ti$_{0.75}$Au$_{0.25}$ sample and its consistency with the x-ray analysis, HRTEM and SAD were performed for different crystallographic orientations. The HRTEM images along [111] (Fig. 2D) and [100] (Fig. 2E), together with the SAD images for the [111] (Fig. 2F) and [102] (Fig. 2G) orientations, are shown in Fig. 2 (F and G) and reveal the atomic arrangement in both real space (HRTEM) and reciprocal space (SAD) in $\beta$-Ti$_{3}$Au. Similar TEM and XRD analyses were performed on several other Ti$_{1-x}$Au$_{x}$ alloys, with the results summarized in Table 1. For all compositions, the main crystallographic phase is confirmed by both XRD and TEM data.

In addition to the crystal structure, two other factors point to the high hardness in Ti$_{0.75}$Au$_{0.25}$: high VED and pseudogap formation. In metallic materials, strong atomic bonding has been suggested to improve phase stability and hardness (23, 24), and this can be quantified by examining the value of the VED. The VED value for $\beta$-Ti$_{3}$Au is 0.20 Å$^{-3}$, which is higher than that of any other Ti-$\alpha$ binary (Table 2). This is consistent with $\beta$-Ti$_{3}$Au being the hardest among the Ti-Au alloys. Another possible origin of the high hardness of $\beta$-Ti$_{3}$Au is the reduction in the DOS at the Fermi level $E_{F}$, which has been referred to as a pseudogap (62, 63). It has been suggested that the pseudogap formation stabilizes the phase and, consequently, improves the hardness (63). Typically, to significantly affect the crystallographic phase, the pseudogap must have a width $W$ of 0.5 to 1.5 eV and a relative height ratio $H/H_{0}$ of greater than 0.5, where $H$ and $H_{0}$ represent the DOS at the top and bottom of the pseudogap, respectively (63). $\beta$-TiAu, TiAu$_{2}$, and $\beta$-Ti$_{3}$Au all exhibit pseudogap-like features around the Fermi level ($E = 0$ in Fig. 3). The pseudogap of $\beta$-Ti$_{3}$Au is the most pronounced of all the studied Ti-Au compounds (Fig. 3, inset), with $W \approx 1$ eV and $H/H_{0} \approx 4$, a result that reinforces the experimental observation of the highest hardness in $\beta$-Ti$_{3}$Au.

Together with hardness, the lifetime of a material has to be considered in determining a material’s applicability toward medical components. The lifetime of a component in vivo is determined partially by its component’s wear rate. In particular, knee and hip replacements currently only last approximately a decade, making additional component replacements necessary (45). A comparison of the time-dependent COF in Ti$_{1-x}$Au$_{x}$ (x = 0, 0.25, 0.30, and 0.50) alloys is shown in Fig. 4A. The reference sample is Ti (blue, $x = 0$), with an average COF close to 0.35, which persists for about 700 s. The three Ti$_{1-x}$Au$_{x}$ alloys, however, show

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### Table 1. Composition and wear test summary for the Ti$_{1-x}$Au$_{x}$ alloys.

| x       | Phase-determined from TEM | Phase-determined from XRD | Ti$_{1-x}$Au$_{x}$ disc | Diamond-SiC disc |
|---------|---------------------------|---------------------------|------------------------|------------------|
|         |                           |                           | Wear volume (mm$^{3}$) | Wear type        |
|         |                           |                           |                        | Wear volume (mm$^{3}$) | Wear type |
| 0       | Reference (commercial specimen) |                           | 0.038 | Abrasive, adhesive | 0.183 | Adhesive |
| 0.25    | $\beta$-Ti$_{3}$Au | $\beta$-Ti$_{3}$Au $+$ $\alpha$-Ti$_{3}$Au $+$ $\alpha$-Ti | 0.012 | Abrasive, adhesive | 0.163 | Abrasive, adhesive |
| 0.33    | $\beta$-Ti$_{3}$Au $+$ $\beta$-TiAu | $\alpha$-Ti$_{3}$Au $+$ $\beta$-Ti$_{3}$Au $+$ $\beta$-TiAu | 0.010 | Abrasive, adhesive | 0.153 | Adhesive |
| 0.50    | $\beta$-Ti$_{3}$Au $+$ $\alpha$-Ti | $\beta$-Ti$_{3}$Au $+$ $\alpha$-Ti $+$ $\alpha$-Ti$_{3}$Au | 0.017 | Adhesive | 0.093 | Adhesive |

### Table 2. Summary of crystallographic and electronic parameters of the Ti–Au phases.

|         | Space group | $d_{Ti-Au}$ (Å) | VED (Å$^{-3}$) | Pseudogap $W$ (eV) | $H/H_{0}$ |
|---------|-------------|----------------|---------------|-------------------|-----------|
| $\alpha$-Ti$_{3}$Au | Pm3m | 2.93237 (31) | 0.18 | — | — |
| $\beta$-Ti$_{3}$Au | Pm3n | 2.84478 (57) | 0.20 | 1 | 4 |
| $\beta$-TiAu | Pmma | 2.79248 (71) | 0.15 | 1 | 0.4 |
| TiAu$_{2}$ | I4/mmm | 2.80056 (72) | 0.12 | 0.2 | 2 |
| TiAu$_{4}$ | I4/m | 2.85050 (73) | 0.10 | — | — |

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**Fig. 3.** DOS of Ti–Au stoichiometric compounds. DOS (solid lines) as a function of energy for $\beta$-TiAu (black), TiAu (yellow), TiAu$_{2}$ (blue), and $\beta$-Ti$_{3}$Au (red), with the pronounced valley around the Fermi energy in $\beta$-Ti$_{3}$Au marked by a dotted line and highlighted in yellow (inset).
The mechanical properties of the intermetallic compound $\beta$-Ti$_3$Au suggest that this material is well suited for medical applications where Ti is already used, with some examples including replacement parts and components (both permanent and temporary), dental prosthetics, and implants. The fourfold increase in hardness, as compared with pure Ti, renders $\beta$-Ti$_3$Au as the hardest known biocompatible intermetallic compound. The wear properties of $\beta$-Ti$_3$Au indicate that this compound has a COF that is four times less than that of Ti, resulting in the reduction of the wear volume by 70%, which will ensure longer component lifetime and less debris accumulation. Moreover, the ability to adhere to a ceramic surface will result in reducing both the cost and the weight of these components.

The high hardness in $\beta$-Ti$_3$Au can be attributed to three main factors: (i) the cubic crystal structure with inherently short Ti–Au bonds and high (14) Ti atomic coordination, (ii) the high VED, and (iii) the pseudogap formation. Between the two cubic Ti$_3$Au compounds, the Ti–Au bond length is smaller for the $\beta$ phase ($d_{\text{Ti–Au}} = 2.84478\,\text{Å}$) compared to the $\alpha$ phase ($d_{\text{Ti–Au}} = 2.87224\,\text{Å}$), indicating that the wear in these alloys is reduced compared to that in pure Ti ($x = 0$). These results demonstrate that the addition of Au is an effective route to reducing the friction of Ti. To identify the wear modes, scanning electron microscopy (SEM) analysis was conducted, and the results are shown in Fig. 5, with the wear images of the Ti$_{1-x}$Au$_x$ pins (left panels) compared to those of the diamond-SiC disc (right panels). The bright and groove-like features correspond to abrasion from diamond grits, whereas the darker spots indicate adhesion wear. The Ti$_{1-x}$Au$_x$ alloys have some areas that seem to be worn less, corresponding to regions with elevated hardness. These features are most pronounced in the Ti$_{0.75}$Au$_{0.25}$ sample, consistent with its lower wear rates. The wear modes and volumes of the Ti$_{1-x}$Au$_x$ alloys are given in Table 1.
compared to the α phase ($d_{Ti-Au} = 2.93237 \text{ Å}$). Together with the more complex crystallographic environments of both Ti and Au in $\beta$-Ti$_2$Au, this inhibits dislocations and results in high hardness in this particular compound. Understanding the factors that influence the hardness of $\beta$-Ti$_2$Au provides insights for improving the existing biocompatible alloys and designing new biocompatible materials with superior mechanical properties.

**METHODS**

Alloys of Ti$_{1-x}$Au$_x$ were prepared by arc melting Ti (Cerac, 99.99%) and Au (Cerac, 99.99%) in stoichiometric ratios, with mass losses of no more than 0.3%. To ensure homogeneity, the samples were remelted several times. Given that Ti alloys are frequently heat-treated to improve both hardness and ductility, annealing studies were carried out for the Ti–Au system. However, due to the different annealing cycles, similar to those used for other Ti-based alloys (65), resulted in minimal changes in the hardness compared to the as-cast samples. This might be caused by variation in microstructure homogeneity, which can mask the true annealing effects. The hardness of the arc-melted samples made it virtually impossible to grind these samples, which rendered powder XRD experiments difficult. Therefore, XRD data were collected at room temperature off the cross section (about 3 mm in diameter) of cut and polished samples using a custom four-circle Huber diffractometer with a focusing graphite monochromator and analyzer in a nondispersive geometry. CuK$_\alpha$ radiation was used in a Tukon 2100 microhardness tester, equipped with a Vickers diamond pyramid indenter. The microhardness tests were performed on a polished sample surface of about 3 mm in diameter. Multiple tests were conducted for all samples to maintain repeatability, using a 300-g load, with a duration of 10 s.

Tribological experiments were conducted using a pin-on-disc tribometer (CSM Instruments) with a total of 40,000 cycles. The diamond-SiC disc was selected for its durability. The ingot samples of Ti$_{1-x}$Au$_x$ ($x = 0.25, 0.3$, and $0.50$) were used as a pin, with a Ti ingot used as a reference. An example of the Ti$_{0.75}$Au$_{0.25}$ sample used for wear tests is shown in the inset of Fig. 4A. To simulate wear during walking, a linear reciprocal motion was used with a sliding speed of 3.15 cm s$^{-1}$ and an applied load of 2 N. The sliding distance of wear tests was set at 4 mm per stroke, with a total of 40,000 cycles. A synthetic body fluid was used as the test medium. The total wear is presented as volume loss. Details regarding the wear of the diamond-SiC disc have been previously reported (66–68).

The MTS assay was used to assess the cytotoxicity of the samples. For the study, 293T cells were cultured in Dulbecco’s modified Eagle’s medium supplemented with 10% fetal bovine serum, penicillin, and streptomycin. Five thousand cells were seeded in a 24-well plate along with the samples. The cells with the samples were incubated at 37°C. After 3 days of incubation, 150 μl per well of MTS reagent was added. It was further incubated for an hour and then the optical density was measured using a microplate reader. The sample of pure titanium had very poor cytocompatibility. The cells were observed to be strained and rounded, whereas the alloys did not show any significant effects.

Samples for HRTEM analysis were prepared via grinding and ion milling. The HRTEM image of the $x = 0.25$ sample was performed using a JEOL 2100 field emission gun transmission electron microscope. The microstructures of the $x = 0.33$ and $x = 0.50$ samples were investigated by a probe aberration–corrected JEOL JEM-ARM200FC at 200 kV.

Band-structure calculations were performed using the full-potential linearized plane-wave method implemented in the WIEN2k package (69, 70). The Perdew-Burke-Ernzerhof generalized gradient approximation was used as the exchange correlation potential, and a $10 \times 10 \times 10$ grid was used to sample the k-points in the Brillouin zone.

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