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Designing hard, low-refractive-index lossy materials for super wear-resistant absorbers

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ABSTRACT

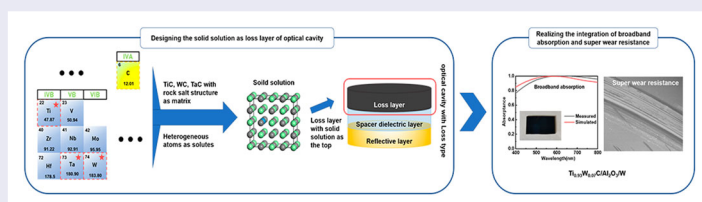
Super wear-resistant absorbers are important for next-generation high-stability optical splitters. However, traditional absorbers cannot obtain ideal wear resistance because of the limitation of hardness. We show that solid solutions of transition metal carbides are new hard, low-refractive-index lossy materials (HLLM). Super wear-resistant absorbers were obtained by constructing HLLM-based optical cavities. As a proof-of-concept, the absorptance of our fabricated $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ -based optical cavity was 95.6% in 400–800 nm wavelength range. In addition, its wear resistance was 23.3 times greater than that of traditional absorbers. Through the experiments and theoretical calculations, we proposed the design principles of HLLM and presented the materials map.

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Hardness; refractive index; lossy materials; wear resistance; absorber



IMPACT STATEMENT

We obtained the super wear-resistant absorbers for the first time through designing hard, low-refractive-index lossy materials, and constructing an optical cavity with the lossy materials as the top layer.

1. Introduction

Super wear-resistant absorbers (SWA) are functional films with high broadband absorption performance and high wear resistance. There are many applications of SWA in next-generation high-stability optical splitters devices and light-collection systems. For example, SWA is used on the surface of mechanical parts in splitters devices of optical-communication systems. It not only provides broadband absorption performance to eliminate stray light [1,2], but also effectively reduces the damage to the

absorber caused by repeated scratches and impacts from hard objects during assembly [3,4]. When SWA is coated on the surface of mechanical parts of solar collectors [5,6], it can absorb light and resist the impact of sand particles [7,8]. At present, SWA has attracted much attention because of its potential applications in the civil & military industries.

The lossy optical cavities with Salisbury screen structure [9–11] have both anti-reflection and high absorption loss. It is one of the most effective ways to achieve

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broadband absorption. Lossy optical cavities have been widely used in solar cells [12], concentrated solar power [13,14], photodetectors [15], structure color generation [10,16] and pyroelectric thermal detector [17]. Traditionally, the loss layer is mainly made of high extinction coefficient metals such as W, Ti, Cr, etc. [1,15,18]. However, the hardness of this kind of metal is less than 5 GPa [19], and the wear resistance and scratch resistance are not good. To increase wear resistance and reduce surface reflection, researchers coated the top surface of metals with low-refractive-index oxides, such as amorphous Al_2O_3 and SiO_2 [20]. However, because the hardness of these amorphous oxides was only 8–12 GPa [21], the improvement of the wear resistance of the obtained absorbers $\text{Al}_2\text{O}_3/\text{W}/\text{Al}_2\text{O}_3/\text{W}$ was very limited [22]. It is challenging to construct a lossy optical cavity for the integration of broadband absorption and super wear resistance.

In this letter, we proposed an optical cavity with Salisbury screen structure, and realized the integration of super wear resistance and broadband absorption properties for the first time. In this optical cavity, the top loss layer is no longer composed of a double-layer oxide/metal composite, but a single-layer hard, low-refractive-index lossy material (HLLM). It is called a HLLM-based optical cavity. $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ solid solution is a HLLM that has never been reported before. The absorptance of the HLLM-based optical cavity ($\text{Ti}_{0.93}\text{W}_{0.07}\text{C}/\text{Al}_2\text{O}_3/\text{W}$) was 95.6% in the wavelength range of 400–800 nm. The wear resistance was 23.3 times greater than that of traditional absorbers ($\text{Al}_2\text{O}_3/\text{W}/\text{Al}_2\text{O}_3/\text{W}$). We outline the map of HLLM candidates based on the new insight into the relationship between the microstructure of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ solid solution and its performance. The results show that the performance of solid solutions such as $\text{Ti}_{0.74}\text{Zr}_{0.26}\text{C}$ and $\text{Ti}_{0.5}\text{Hf}_{0.5}\text{C}$ is better than that of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$. Therefore, this letter provides a new way for designing SWA by introducing HLLM. This is possible to solve the bottleneck problem on the integration of super wear resistance and broadband absorption.

2. Experiments and calculations

In this work, all films were obtained by magnetron sputtering. The detailed information on sample preparation, characterization, simulation, first-principles calculations [23] and structure information of optical cavity were presented in the supplementary information.

3. Results and discussion

3.1. Formation of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ solid solution

The XPS results (Section S1 of SI) show that the atomic ratio of Ti:W:C in the W-doped TiC film was 0.93:0.07:1,

named $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$. In Figure 1(a), the (111), (200) and (220) diffraction peaks of the rock-salt phase simultaneously appear in the XRD spectra of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ and TiC films. Compared with that of TiC, the diffraction peak of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ film was shifted to a greater angle. These results indicate that the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ film has the same rock-salt structure as TiC. The replacement of a large Ti atom with the atomic radius of 0.146 nm by a small W atom with the atomic radius of 0.137 nm leads lattice shrinkage. The films were studied by Selected Area Electron Diffraction (SAED) and High Resolution Transmission Electron Microscope (HRTEM) to further characterize the structure. Both $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ and TiC films show (111), (200) and (220) diffraction rings in Figure 1(b, c). After the introduction of W, the interplanar spacing of the (111) and (200) crystal planes of the TiC film were decreased from 0.25 and 0.23 nm to 0.24 and 0.21 nm, respectively, as shown in Figure 1(d, e). Therefore, the results of SAED, HRTEM and XRD are in good agreement. This proves that the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ substitutional solid solution is formed after the introduction of W into the TiC film.

3.2. Design, preparation and optical performance of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ -based optical cavity

We designed and prepared three optical cavities for the integration of high wear resistance and high broadband absorption. They are named as $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity, TiC optical cavity and $\text{Al}_2\text{O}_3/\text{W}$ optical cavity, respectively. We compared a traditional $\text{Al}_2\text{O}_3/\text{W}$ optical cavity with $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity. The theoretical absorption spectra of the three optical cavities are shown in Figure 2(a–c), respectively. All optical cavities show high broadband absorption.

We used RF magnetron sputtering to prepare three optical cavities and characterized their optical properties to verify our designs. Because the broadband absorption of the designed optical cavities depends on the thickness of each layer, we measured the thickness of each layer for three times by a surface profiler, and showed the standard error bars of the thickness. The error is calculated in the way of ‘Mean $\pm$ S.D.’ [24]. The results show that the thickness of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$, Al_2O_3 and W layers in $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity were 14.2 ± 0.1 , 45.2 ± 0.1 and 152.0 ± 2.9 nm, respectively. The thickness of other optical cavities and their standard error are listed in Table S2.

In Figure 2(a–c), the experimental absorption spectra of the three optical cavities are consistent with the theoretical absorption spectra. This indicates that the electromagnetic simulation is completely reliable. The spectral curves of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ and TiC optical cavity are in good agreement. This shows that the introduction of

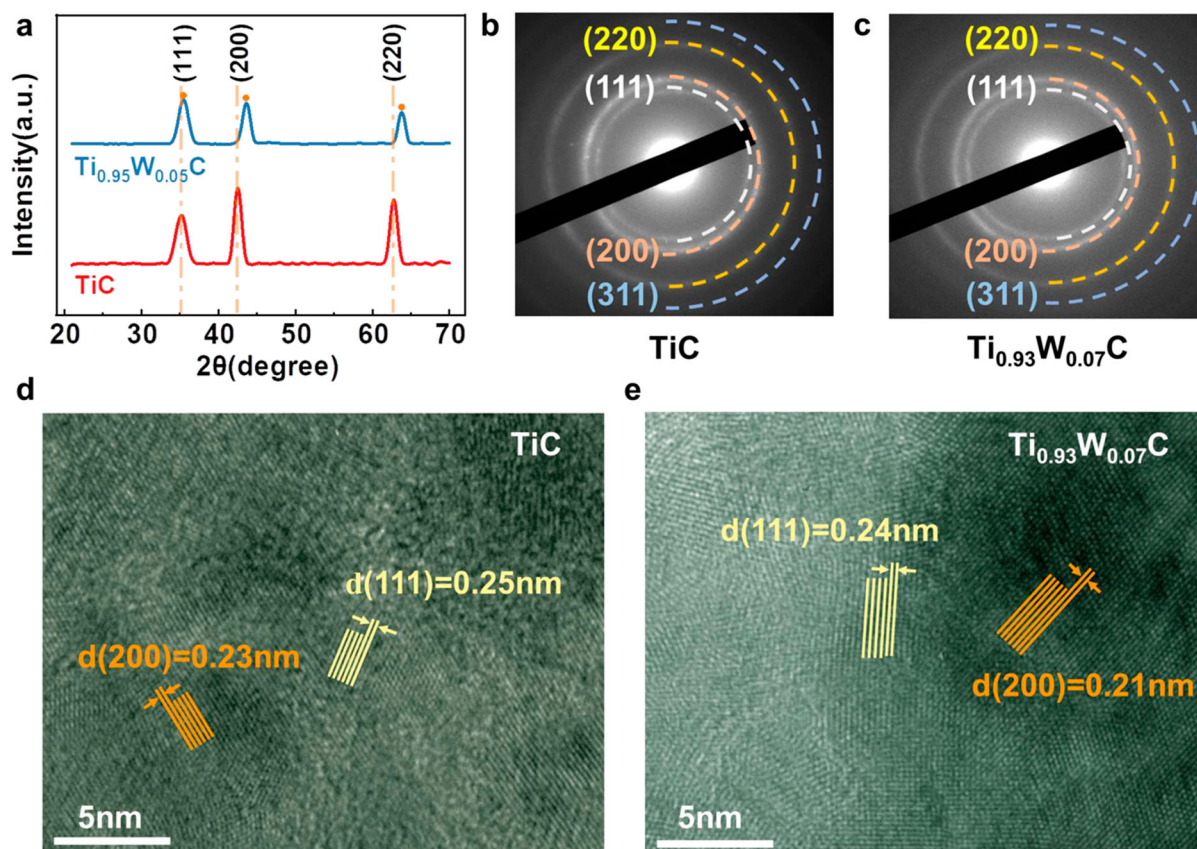


Figure 1. (a) XRD pattern of TiC and $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ films. SAED patterns of (b) TiC and (c) $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ films. HRTEM images of (d) TiC and (e) $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ films.

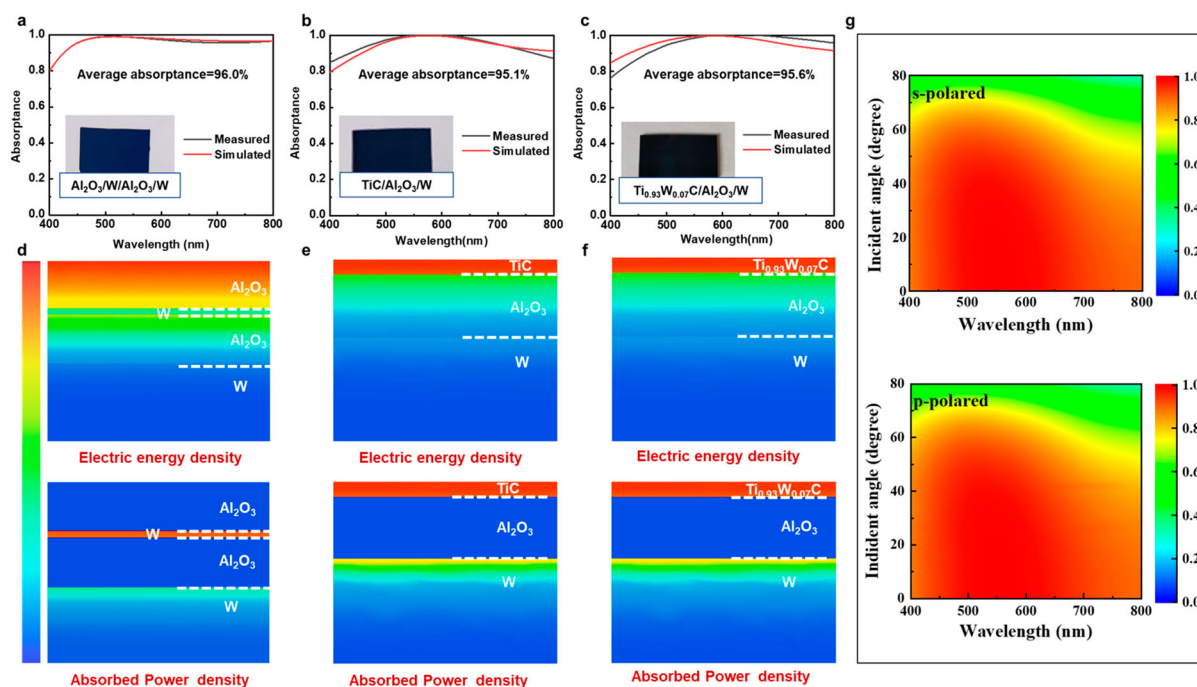


Figure 2. Theoretical and experimental absorption spectra, electric energy density and absorbed power density of (a, d) $\text{Al}_2\text{O}_3/\text{W}/\text{Al}_2\text{O}_3/\text{W}$ film, (b, e) $\text{TiC}/\text{Al}_2\text{O}_3/\text{W}$ film and (c, f) $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}/\text{Al}_2\text{O}_3/\text{W}$ film. (g) The absorption spectrum of s-polarized and p-polarized light incident at different angles in the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity.

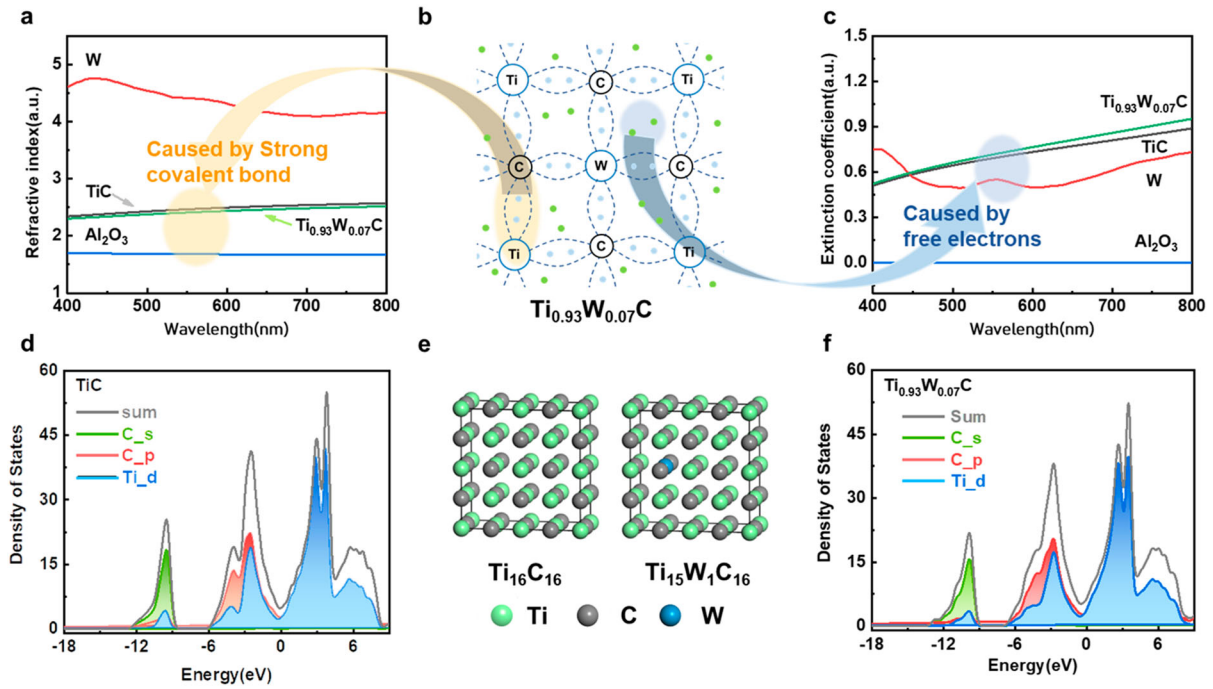


Figure 3. The (a) refractive index and (c) extinction coefficient of W, Al_2O_3 , TiC and $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ films. (b) A local structure diagram of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ films. Green balls represent free electrons and blue balls represent localized electrons forming covalent bonds. DOS and unit cell of (d, e) $\text{Ti}_{16}\text{C}_{16}$ and (f, e) $\text{Ti}_{15}\text{W}_1\text{C}_{16}$ obtained by first-principles calculations.

W and the formation of solid solution do not change the absorption properties of the optical cavity. In addition, although the curve shapes are slightly different, the average absorptance of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity (95.6%) and $\text{Al}_2\text{O}_3/\text{W}$ optical cavity (96.0%) are very close. The average absorptance is the integral area of the absorption curve and the x -axis (the details are given in SI). This showed that the single-layer $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ can act as a two-layer $\text{Al}_2\text{O}_3/\text{W}$ composite. It has the functions of anti-reflection and loss absorbing.

We analyzed the electric energy density and absorbed power density inside the optical cavity to explain the high broadband absorption of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity, as shown in Figure 2(d–f). In $\text{Al}_2\text{O}_3/\text{W}$ optical cavity, the electric energy was concentrated on the Al_2O_3 layer, and absorbed power was concentrated on the W layer. This indicates that anti-reflection and loss absorbing occur in the Al_2O_3 and W layers, respectively. In contrast, the electric energy and absorbed power in $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ and TiC optical cavities were concentrated in the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ and TiC layers. This indicates that $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ and TiC play the roles of anti-reflection and loss absorbing at the same time. To further explain effects of antireflection and light loss of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$, we showed the refractive index and extinction coefficient of TiC and $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ (Figure 3(a,c)) from fitting the reflection spectrum. This is consistent with that from ellipsometry (Figure S5). According to the Fresnel formula [25], the decrease in

the refractive index of the top layer leads to the increase of absorptance (detailed in Section 5 of SI). We simulated the absorption spectra of the optical cavities with different refractive indexes to verify the assumption. The results show that the absorptance of the optical cavity increases with the increase of n_3 (Figure S3). Similarly, it can be shown that the increase in k_3 will also lead to the increase in absorptance. Therefore, $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ and TiC, as low refractive index loss materials, are more conducive to high broadband absorption.

We constructed a unit cell of $\text{Ti}_{15}\text{W}_1\text{C}_{16}$ (Figure 3(e)) for first-principles calculations to study the low refractive index and high extinction coefficient of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$. It was the closest to $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$. The $\text{Ti}_{16}\text{C}_{16}$ unit cell was also constructed and calculated for further comparison. The density of states (DOS) of $\text{Ti}_{15}\text{W}_1\text{C}_{16}$ and $\text{Ti}_{16}\text{C}_{16}$ are shown in Figure 3(d, f). The two spectral lines are highly consistent in shape and peak position. The result indicates that the introduction of a small amount of W does not significantly change the electronic structure of $\text{Ti}_{16}\text{C}_{16}$. This is the reason why the refractive indices and extinction coefficients of $\text{Ti}_{15}\text{W}_1\text{C}_{16}$ and $\text{Ti}_{16}\text{C}_{16}$ are similar (Figure 3(a, c)). There is strong hybridization between the C-p orbitals and W, Ti-d orbitals from -9 eV to the Fermi level region. This indicates that there are significant covalent bond characteristics in Ti-C and W-C. The higher electron DOS near the Fermi level indicates the existence of free electrons,

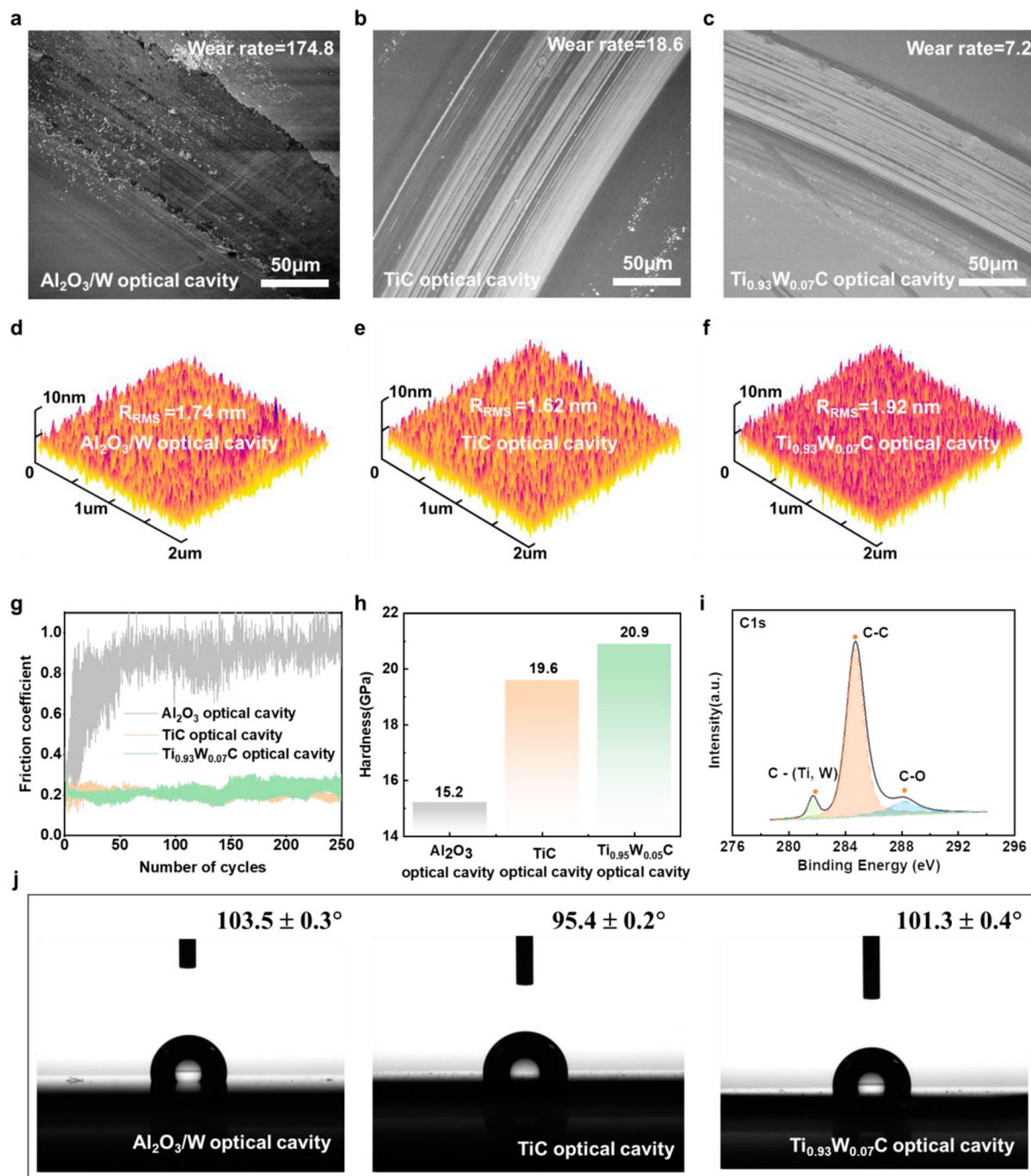


Figure 4. The SEM images on the wear tracks after the ball-on-disk wear tests for (a) $\text{Al}_2\text{O}_3/\text{W}$, (b) TiC and (c) $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavities. AFM images (d–f), friction coefficients curves (g) and hardness (h) for the same optical cavities. (i) The core level spectra of XPS C1s performed on the wear tracks for $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity. (j) The WCA of $\text{Al}_2\text{O}_3/\text{W}$, TiC and $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavities.

which are mainly from the unbonded d-orbital electrons of Ti and W. Therefore, the low refractive index of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ is attributed to the strong covalent bond network (Figure 3(b)), and the extinction coefficient of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ is attributed to the high carrier absorption (Figure 3(b)).

We show the absorption spectra of s-polarized and p-polarized light incident at different angles to study the angular performance of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity (Figure 2(g)). The average absorptance of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity was always greater than 90% as the incident

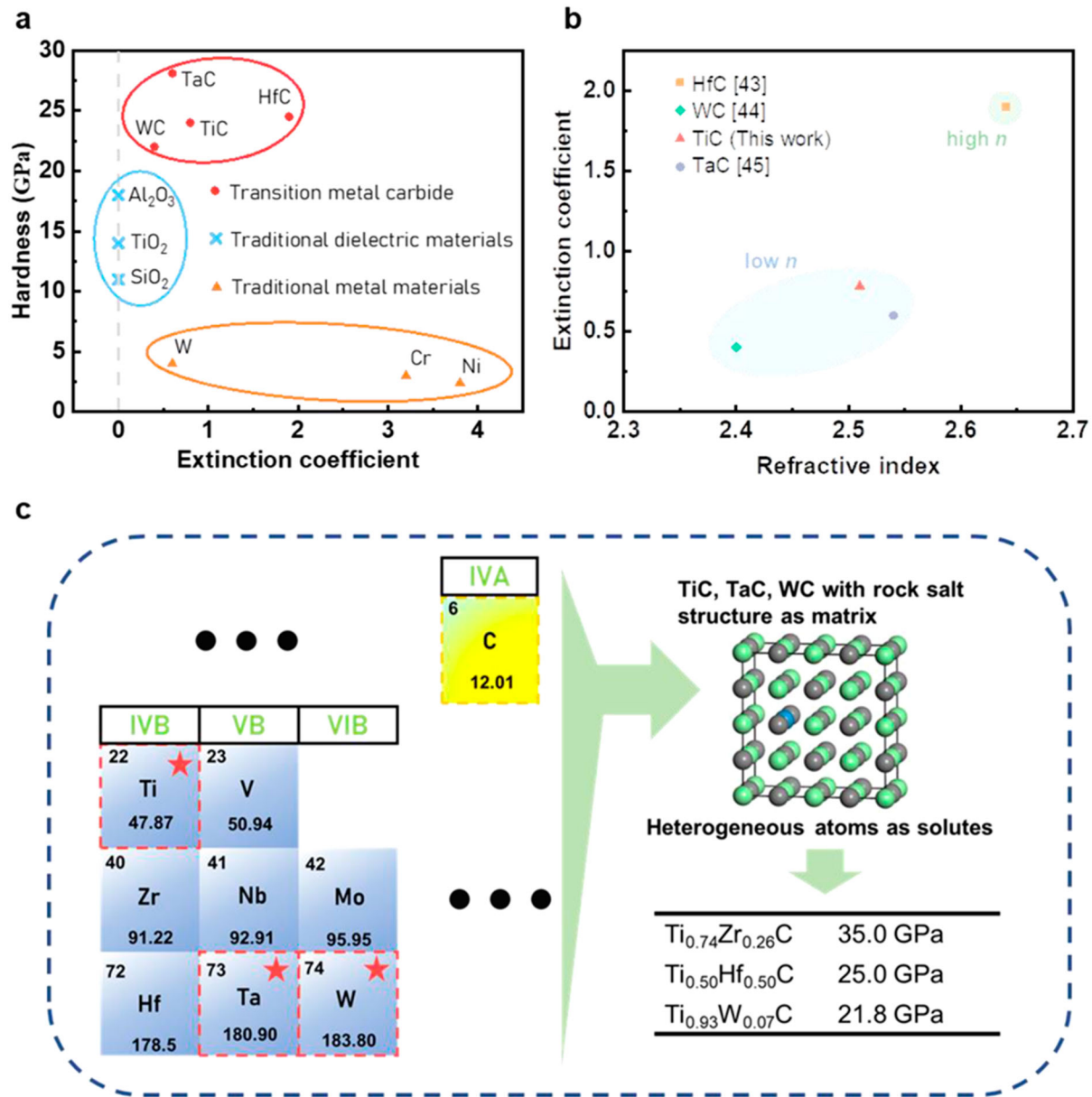


Figure 5. (a) The hardness of typical transition metal carbides, traditional dielectric materials and pure metals and their average extinction coefficient. (b) Average refractive index and extinction coefficient of typical transition metal carbides. (c) Element composition of HLLM.

angle changes from 0 to 50°. Therefore, the broad-band absorption of Ti_{0.93}W_{0.07}C optical cavity does not strongly depend on the incident angle.

3.3. Wear resistance of Ti_{0.93}W_{0.07}C-based optical cavity

The SEM images of the wear track of Ti_{0.93}W_{0.07}C, TiC and Al₂O₃/W optical cavities are shown in Figure 4(a–c). There are many wear debris and significant ploughing grooves on the surface of the film in the SEM image of the Al₂O₃/W optical cavity (Figure 4(a)). In addition, debris in the form of tiny particles accumulated on the

edge of the wear track. These phenomena are consistent with the typical abrasive wear mechanism. However, when Ti_{0.93}W_{0.07}C or TiC replaced Al₂O₃/W, only some fine ploughing grooves are shown in the wear surface. The wear surface and the edge of the track are smooth (Figure 4(b,c)). The wear rate of the Al₂O₃/W optical cavity was $174.8 \times 10^{-9} \text{ mm}^3/\text{N m}$, and the wear rate of the TiC optical cavity was reduced to $18.6 \times 10^{-9} \text{ mm}^3/\text{N m}$. The wear rate of the Ti_{0.93}W_{0.07}C optical cavity was further reduced to $7.2 \times 10^{-9} \text{ mm}^3/\text{N m}$, which was 23.3 times greater than the Al₂O₃/W optical cavity (Figure 4(a–c)). These results indicate that the replacement of Al₂O₃/W composite film by Ti_{0.93}W_{0.07}C can

significantly improve the wear resistance of the optical cavity.

We compared the friction coefficient and hardness of the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity with the other optical cavities (Figure 4(g,h)) to explain the low wear rate of the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity. After the running-in period of the $\text{Al}_2\text{O}_3/\text{W}$ optical cavity, the friction coefficient was about 0.90 (Figure 4(g)). In contrast, the friction coefficient of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity and TiC optical cavity were 0.23 and 0.22 after a very short running-in period. They were much lower than the friction coefficient of the $\text{Al}_2\text{O}_3/\text{W}$ optical cavity. In Figure 4(h), the hardness of the $\text{Al}_2\text{O}_3/\text{W}$ optical cavity was 15.2 GPa, the hardness of the TiC optical cavity was increased to 19.6 GPa. In addition, the hardness of the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity was further increased to 20.9 GPa (Figure 4(h)), which is greater than that of the $\text{Al}_2\text{O}_3/\text{W}$ optical cavity. Therefore, the super wear resistance of the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity is attributed to the low friction coefficient and high hardness.

We discussed the origin of the low friction coefficient and high hardness of the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity in the following. The AFM images of three optical cavities are shown in Figure 4(d–f). The root-mean-square of surface roughness of the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$, TiC, Al_2O_3 were 1.92, 1.62 and 1.74 nm, respectively. These are in good agreement with the results reported previously [26,27]. There are two reasons for the similar roughness. First, these films are deposited by magnetron sputtering. In the deposition process, the substrate, deposition temperature (room temperature), background vacuum (1×10^{-4} Pa), sputtering gas (Argon atmosphere) and working pressure (1 Pa) are all the same. Second, these films are high-density hard materials. Previous study has shown that hard films usually have the characteristics of low surface roughness [28]. Therefore, the surface roughness is not the main reason for the low friction coefficient of the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity. The C1s spectrum (Figure 4(i)) shows that the C on the surface of the $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity after wear was mainly in the form of sp^2 C–C bonds (located at 286.7 eV). It does not present in $\text{Al}_2\text{O}_3/\text{W}$. Therefore, the low friction coefficient of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ optical cavity is related to the formation of self-lubricating graphite-like tribofilms in the top layer material $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ [29–31]. The high hardness of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ is attributed to the formation of strong covalent bonds between Ti–C and W–C, and the solid solution strengthening effect caused by the replacement of Ti by W [32].

In addition, the surface wettability of the film is one of the important characteristics of the film surface. To characterize the surface wettability of the films, we tested the water contact angles (WCA, θ) of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$, TiC

and $\text{Al}_2\text{O}_3/\text{W}$ optical cavities. The results show that the WCA of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$, TiC and $\text{Al}_2\text{O}_3/\text{W}$ optical cavities were 101.3°, 95.4° and 103.5°, respectively (Figure 4(j)). Compared with multifunctional superamphiphobic cotton fabrics [33], superhydrophobic cellulose-based plastic [34] and hydrophobic transparent paper [35], the WCA of optical cavities was far from meeting the requirements of superhydrophobic materials for contact angles greater than 150°. Therefore, the three optical cavities have only moderate surfaces hydrophobicity.

3.4. Design criteria and material map for high-performance solid solutions

In addition to splitters devices of optical-communication systems, the wear-resistant broadband absorbers can be applied to other fields such as solar steam generation devices [36]. This involves different bands and performance requirements. We provided the design principles of HLLM, and predicted candidate materials to better ‘customize’ the properties of the film.

The results show that the δ -TiC film with rock-salt structure has strong Ti–C covalent bonds and remaining d-orbital electrons. Thus, it has high hardness and extinction coefficient. Since all transition metal carbides with rock-salt structure [37,38], δ -TMC (TM = Ti, Zr, Hf, V, Nb, Ta, Mo, W) have similar bonding structures, they have high hardness and extinction coefficient. The hardness of δ -TMC and its average extinction coefficient in the range of 400 nm–800 nm are shown in Figure 5(a). The hardness and extinction coefficient of amorphous dielectrics (SiO_2 , Al_2O_3) and metals (W, Cr, Ni) used in traditional absorbers are given for comparison. Amorphous dielectrics have relatively high hardness (8–12 GPa), but do not have high extinction coefficient ($k \approx 0$) [39]. The metals have high extinction coefficient [40] ($k \approx 1.5$), but do not have high hardness (0.9–5.0 GPa) [19]. In contrast, δ -TMC not only has a high extinction coefficient ($k \approx 1.2$), but also has a high hardness (19.8–30.2 GPa) [41,42]. Therefore, they are potential hard lossy materials. The refractive indexes of δ -TMC are shown in Figure 5(b). The refractive index of δ -HfC was as high as 2.65 [43], while the refractive indexes of δ -WC [44], δ -TiC and δ -TaC [45] were only about 2.45. Therefore, δ -WC, δ -TiC and δ -TaC are more suitable for HLLM, which can significantly reduce the light reflection on the surface of the absorbers.

The above results show that $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ substitutional solid solution can significantly improve the wear resistance without reducing the absorption performance of the TiC matrix. Therefore, the substitutional solid solution with δ -WC, δ -TiC and δ -TaC as the matrix can obtain higher wear resistance. Previous studies [46]

have shown that the group-IV, -V and -VIB transition metal carbides except chromium are rock-salt structures. In addition, the atomic radii ($R_{\text{TM}} = 122\text{--}145\text{pm}$) and electronegativity ($\chi_{\text{TM}} = 1.22\text{--}1.45$) of these transition metal elements are very similar, and these three conditions are all favorable to the formation of substitutional solid solutions. Therefore, the introduction of any heterogeneous atom of Zr, Hf, V, Nb, Mo, W, Ti and Ta into the WC, TiC and TaC matrix (Figure 5(c)) can form a substitutional solid solution. In the literatures [47,48] (Figure 5(c)), the hardness of $\text{Ti}_{0.74}\text{Zr}_{0.26}\text{C}$ and $\text{Ti}_{0.5}\text{Hf}_{0.5}\text{C}$ are 35 and 25 GPa, respectively. They are greater than the hardness of $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ (21.7 GPa). Therefore, in addition to $\text{Ti}_{0.93}\text{W}_{0.07}\text{C}$ solid solution, $\text{Ti}_{0.74}\text{Zr}_{0.26}\text{C}$ and $\text{Ti}_{0.5}\text{Hf}_{0.5}\text{C}$ are also ideal HLLMs. The optical cavities with $\text{Ti}_{0.74}\text{Zr}_{0.26}\text{C}$ and $\text{Ti}_{0.5}\text{Hf}_{0.5}\text{C}$ as the top loss layer have excellent performances. It is worthy of further in-depth study.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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