

# Tribological Properties of Low-cost Titanium Alloys Reinforced by Rice Husk

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**Abstract:** Wear performance and mechanism of Ti alloys strengthened by solid solute atoms Si and O from rice husk were studied from the viewpoint of wear rate, worn surface morphology and wear debris analysis. Results show that the hardness of Ti alloys increases while the volume wear rate decreases significantly with the addition of SiO<sub>2</sub>. There are three changes of the worn surface with the increase in SiO<sub>2</sub> content. One is the transfer of deep grooves to shallow scratches, second is the decrease of plastic deformation degree and the third is the reduction in size and amount of adhesive debris. The decrease of debris size can be attributed to the embrittlement of wear dust since the content of hard and brittle counter grinding GCr15 steel increases in the wear dust and the hardness of Ti alloys increases with the increase in SiO<sub>2</sub> content. Wear mechanism also transfers from adhesive and abrasive wear of pure Ti to abrasive and oxidation wear with the increase in SiO<sub>2</sub> content. The results provides a cheap method to produce Ti alloys with excellent comprehensive properties by agricultural waste rice husk, which is also benefit for the environmental protection.

**Key words:** low-cost Ti alloys; rice husk SiO<sub>2</sub>; tribology performance; wear mechanism

Ti alloys have been considered as one of the most promising materials due to their excellent properties, such as low density, high strength, good resistance in corrosion and oxidation<sup>[1]</sup>. However, there are still some intrinsic disadvantages baffling the widespread applications of Ti materials, especially the low friction and wear resistance as well as high production cost<sup>[2,3]</sup>. Therefore, it is an indispensable research domain to explore Ti alloys with high strength and wear performance by cheap methods.

Although there are many factors that can significantly decrease the manufacturing costs of Ti and its alloys, two methods are considered as the most important and effective ones. The first is using cheap raw materials, including alloying elements and raw Ti resource. For example, using Fe, Mn, Al, Si, etc, to replace V, Mo, Ta and Nb, and using low cost hydride-dehydride (HDH) Ti powder, spongy Ti and even

recycled machining chips, instead of atomized Ti alloying powder<sup>[4-6]</sup>. The second is using other methods to replace traditional ingot metallurgy processing crafts, which are of great expense due to the high requirement in vacuum<sup>[7]</sup>. Typically, powder metallurgy is one of the most popular methods to produce low-cost Ti materials owing to its lower fabrication temperature, higher usage of materials (net-shape or near-net-shape), and lower requirement for vacuum of equipment<sup>[8]</sup>.

Recently, environmental pollution is one of the most serious questions all over the world, and thus the usage of waste is meaningful from the viewpoint of economy or environmental protection. In China, Japan, Indian and Thailand, rice is the most important food, and millions of tons of rice husk are produced as agriculture waste<sup>[9,10]</sup>. Rice husk is rich in SiO<sub>2</sub>, which can also be picked up by a simple method<sup>[11]</sup>. Therefore,

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a lot of work now has been focused on the re-usage of rice husks<sup>[12, 13]</sup>. In previous work<sup>[14]</sup>, a low-cost method to produce Ti alloys by powder metallurgy method using rice husk as SiO<sub>2</sub> resource was reported, and the hardness was enhanced significantly by the solid solution of Si and O atoms decomposed from SiO<sub>2</sub>. However, the wear performance and mechanism still remain unknown.

In this study, dry sliding test was used to investigate the wear properties of Ti alloys strengthened by SiO<sub>2</sub>, the wear rate, worn morphologies and wear debris were systematically studied, and the wear mechanism as well as the influence of SiO<sub>2</sub> were discussed.

## 1 Experiment

Ti solid-solution alloys strengthened by Si and O atoms were prepared from pure Ti powder and SiO<sub>2</sub> powder by powder metallurgy method, where SiO<sub>2</sub> powder was produced by combusting rice husks and the contents were 0.25 wt%, 0.5 wt% and 1 wt%, respectively. For comparison, pure Ti block was also prepared by the identical procedure. The detailed information on the sample preparation can be found in our previous work<sup>[14]</sup>.

Dry sliding wear tests were carried out by ball-on-disc mode on a rotary friction and wear tester. Pure Ti and Ti alloys were machined to 40 mm in diameter and 3 mm in height, and used as disc samples. GCr15 bearing steel balls with diameter of 4.7 mm were used as ball samples. The experimental parameters were follows: friction radius 6 mm, rotating speed

of disc 600 r/min, axial load 15 N, and friction time 90 min. Mass loss was measured by an electrical analytical balance with a precision of 0.1 mg, and repeat three times to get an average value. Wear resistance was characterized by volume wear rate<sup>[15]</sup>, which was calculated by the following equation.

$$V_m = \frac{\Delta m}{NS\rho_v} \quad (1)$$

where  $\Delta m$  is the mass loss (g),  $N$  is the load (N),  $S$  is the sliding distance (m), and  $\rho_v$  is the density of the sample (g/cm<sup>3</sup>).

Microstructure and morphology of worn surface and wear debris were observed by scanning electron microscope (SEM). Phase composition was investigated by X-ray diffractometer (XRD), and EDS equipped with SEM was used to study the chemical composition of the wear debris. Profile of worn surface was analyzed by 3D laser scanning confocal microscope (LSCM) and then the width and depth of the worn groove were obtained. Hardness was tested by Vickers hardness tester with a load of 20 kg and duration time of 30 s. Five measurements were repeated to get an average hardness value.

## 2 Results and Discussion

### 2.1 Microstructure and phase composition

Fig.1 shows the result of microstructure characterization on Ti alloys with 0 wt%, 0.25 wt%, 0.5 wt% and 1.0 wt% rice husk ashes (RHAs) SiO<sub>2</sub> addition. It can be found that the addition of SiO<sub>2</sub> does not change the microstructure of pure Ti significantly, and all the four Ti alloys consist of equiaxed grains (Fig.1a~1d). With the increase in SiO<sub>2</sub> content, there

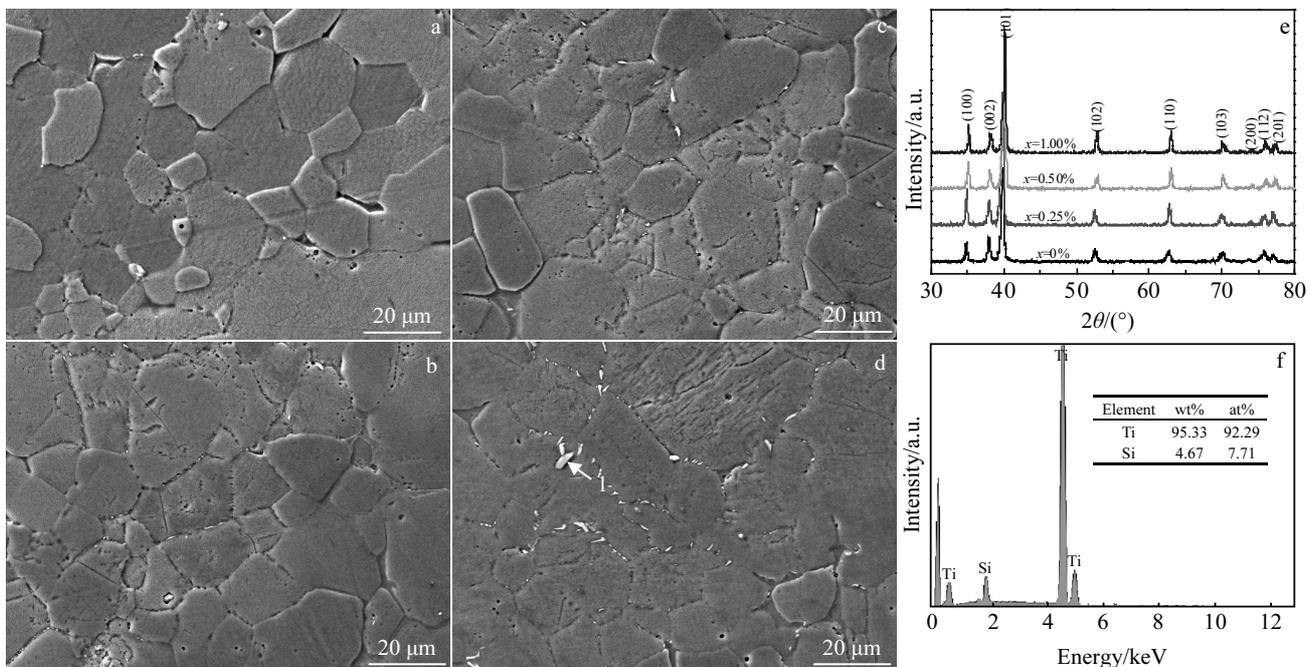


Fig.1 Microstructures of Ti alloys with different content of RHAs SiO<sub>2</sub> addition: (a) 0 wt%, (b) 0.25 wt%, (c) 0.5 wt% and (d) 1.0 wt%; XRD patterns (e) and EDS result (f) of the white particle marked by arrow "1" in Fig.1d

are a small number of white particles at the grain boundary of Ti grains. XRD analysis indicates that no new phase appears after the addition of SiO<sub>2</sub>, and all the diffraction patterns correspond to  $\alpha$ -Ti phase (Fig.1e). The small white particle in Fig.1d marked by arrow "1" contains 95.33 wt% Ti and 4.67 wt% Si, which are much higher than the nominal content of Si content (0.468 wt% converted from 1.0 wt% SiO<sub>2</sub>). Combined with Ti-Si binary phase diagram and Zhu's work<sup>[16]</sup>, it can be conjectured that these white particles are Ti<sub>5</sub>Si<sub>3</sub>. Although the phase constitution is not confirmed exactly enough, the results suggest that most RHAs SiO<sub>2</sub> can dissolve into Ti matrix, resulting in Si and O solution-strengthened Ti alloys. It should be mentioned that, since Si and O atoms lead to negative and positive lattice distortion of Ti matrix, respectively<sup>[14]</sup>, there is no obvious shifting of the diffraction peaks corresponding to  $\alpha$ -Ti phase.

## 2.2 Hardness test

It is well known that the friction and wear performance of materials closely depend on hardness, and thus the hardness was tested and result is shown in Fig.2. It can be seen that the addition of RHAs SiO<sub>2</sub> can significantly improve the hardness of Ti alloys. With the increase in SiO<sub>2</sub> content, the increasing tendency decreases gradually, which may be caused by the formation of Ti-Si intermetallic (Fig.1d). Furthermore, linear fitting is done only for the first three points since most Si and O dissolve into the Ti matrix rather than form Ti-Si compounds, and the result suggests that the increase amplitude is about 3142.7 MPa per 1 wt% SiO<sub>2</sub> addition, which is much higher than the reported strengthening effect of single O element<sup>[17]</sup>. Obviously, it is successful to prepare high strength Ti alloys using rice husk as SiO<sub>2</sub> resource, which can also avoid environmental pollution.

## 2.3 Wear performance

In order to evaluate the wear performance of Ti alloys with different contents of RHAs SiO<sub>2</sub> addition, mass loss and 3D LSCM analysis were carried out, and the results are shown in Fig.3. It can be observed that, with the addition of SiO<sub>2</sub>, the wear rate decreases obviously, which decreases by about 40% per 1 wt% SiO<sub>2</sub> addition. Combined with the variation of

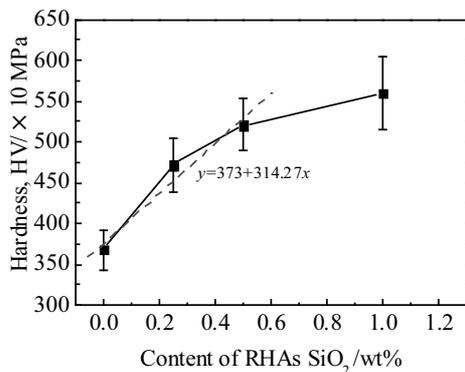


Fig.2 Hardness of Ti alloys with different RHAs SiO<sub>2</sub> contents

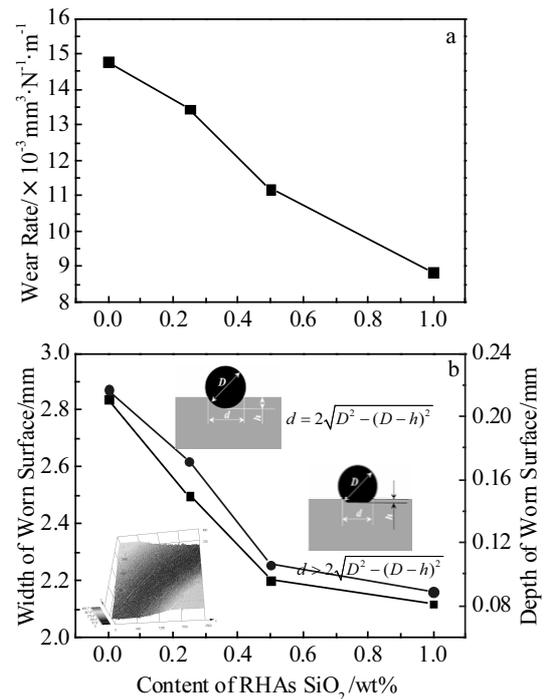


Fig.3 Volume wear rate (a) and geometric size (b) of worn groove of Ti alloys with different RHAs SiO<sub>2</sub> addition (3D LSCM profile of the worn surface is inserted in Fig.3b)

hardness of Ti alloys (Fig.2), it can be confirmed that the higher the hardness of Ti alloys, the lower the worn mass loss, suggesting that hardness is an importance factor for the wear performance of materials. In addition, 3D measurements show that both the width and depth of the wear grooves decrease with the increase in RHAs SiO<sub>2</sub> content, further demonstrating that the wear resistance is improved by RHAs SiO<sub>2</sub> addition (Fig.3). Moreover, when the SiO<sub>2</sub> content is less than 0.5 wt%, the relationship between the width and depth corresponds well with the geometrical shape of GCr15 steel ball used in this study, meaning that the wear mostly occurs on the disc sample (viz Ti alloys) rather than on the grinding ball. However, when the SiO<sub>2</sub> content is higher than 0.5 wt%, the measured width is larger than that calculated from the mathematic formulation, suggesting that both the ball and Ti alloys are worn. Apparently, these results demonstrate that the improvement of hardness caused by RHAs SiO<sub>2</sub> addition is beneficial for the wear performance of Ti alloys.

## 2.4 Wear mechanism

Though Fig.3 summarize the wear rate of Ti alloys, there is still no specific information about the worn surface, and the wear mechanism remains unknown. In order to clarify this, detailed information of the worn surface was studied by SEM and the results are shown in Fig.4. It can be seen from the worn surface of pure Ti (Fig.4a) that, there are numerous of deep grooves parallel to the sliding direction and serious plastic

deformation around the grooves. The grooves suggest that abrasive wear exists, and the plastic deformation, delamination areas and adhered debris mean that adhesive wear exists at the same time. Hence, the wear mechanism of pure Ti materials may be a mixture of adhesive and abrasive wear. With the increase in RHAs SiO<sub>2</sub> content, the deep grooves convert to shallow scratches, the amount of adhesive debris reduces, and the degree of plastic deformation also decreases significantly, as

shown in Fig.4b~4d. This phenomenon indicates that the plastic deformation and adhesive wear are abated, agreeing well with the hardness of Ti alloys shown in Fig.2.

SEM morphology and EDS analysis of wear debris of pure Ti and Ti alloy with 0.5 wt% SiO<sub>2</sub> addition are shown in Fig.5. It can be seen that, there are several large plates beside many small particles in the wear debris of pure Ti, but there are uniform small particles in wear debris of Ti alloy with 0.5 wt% SiO<sub>2</sub>,

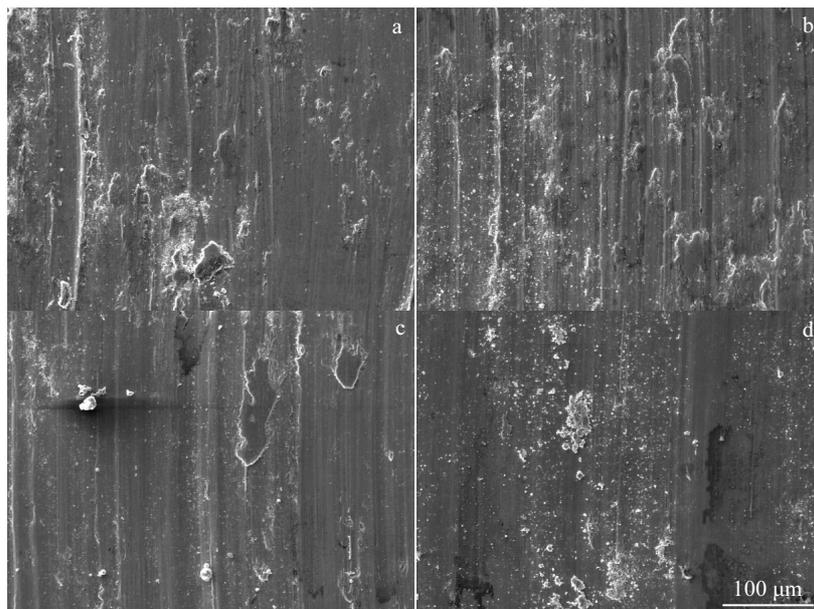


Fig.4 Worn surface of Ti alloys with different RHAs SiO<sub>2</sub> addition: (a) 0 wt%, (b) 0.25 wt%, (c) 0.5 wt%, and (d) 1.0 wt%

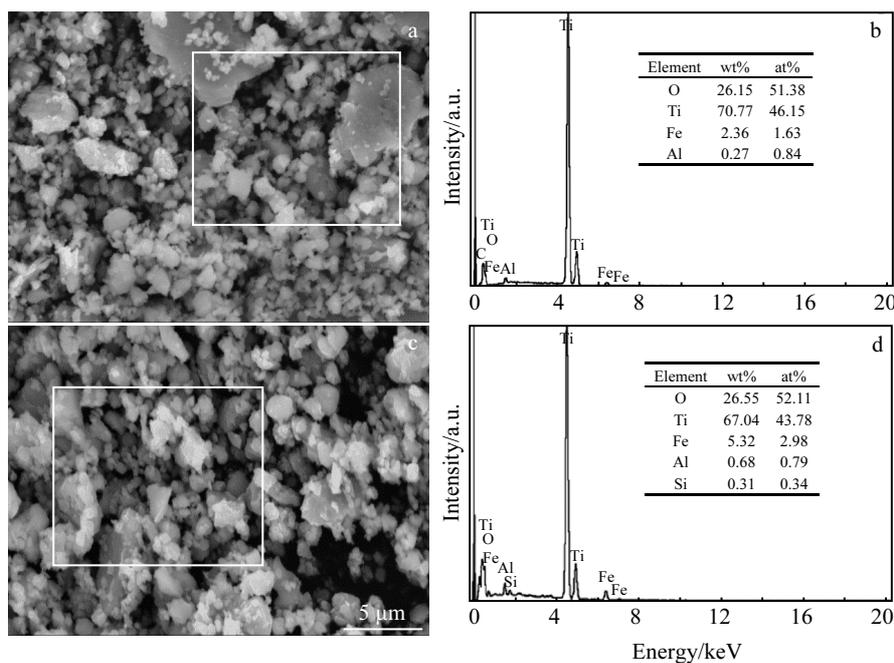


Fig.5 Morphologies (a, c) and EDS results (b, d) of wear debris of pure Ti (a, b) and Ti alloys with 0.5 wt% RHAs SiO<sub>2</sub> (c, d)

which also suggests that the plastic deformation and adhesive wear are abated. In addition, EDS analysis suggests that oxidation occurs in the friction and wear process due to the heat generated by high sliding speed<sup>[18]</sup>. Fe content in Ti alloy with 0.5 wt% SiO<sub>2</sub> is higher than that in pure Ti, meaning that grinding GCr15 ball is also worn, as illustrated in Fig.4d. Due to oxidation, hard and brittle GCr15 dust mixed into wear debris as well as the high hardness of Ti matrix caused by SiO<sub>2</sub> addition (Fig.2), the wear debris becomes brittle, and then the adhesive debris becomes less and smaller, as shown in Fig.4. As a consequence, the dominant wear mechanism transfers to abrasive and oxidation wear.

### 3 Conclusions

1) Ti alloys strengthened by solid solution atoms Si and O can be prepared using agriculture waste rice husks as SiO<sub>2</sub> resource.

2) Wear rate of Ti alloys with SiO<sub>2</sub> addition is obviously lower than that of pure Ti under dry sliding condition. When SiO<sub>2</sub> content is lower than 0.5 wt%, the wear mainly occurs on Ti alloys, but the friction pair GCr15 ball is also worn when SiO<sub>2</sub> content is higher than 0.5 wt%.

3) With the addition of SiO<sub>2</sub>, the deep grooves on worn surface change to shallow scratches, large wear debris breaks up to small one, and plastic deformation decreases, suggesting that the wear mechanism transfers from adhesive and abrasive wear of pure Ti to abrasive and oxidation wear.

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## 以稻壳为强化原料制备低成本钛合金的摩擦学性能

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**摘要:** 以稻壳热解产物为 SiO<sub>2</sub> 源, 通过粉末冶金法制备了 Si 和 O 固溶强化的低成本钛合金, 并从磨损率、磨损表面形貌和磨屑成分等角度研究了其摩擦学性能和磨损机理。结果表明, 随着 SiO<sub>2</sub> 加入量的增加, Ti 合金的体积磨损率显著降低, 与硬度的变化规律吻合良好, 同时磨损表面有三处变化, 即犁沟的深度变浅、塑性变形程度降低和磨屑的尺寸变小且数量减少。SiO<sub>2</sub> 的加入使磨损机制从纯 Ti 的粘着磨损和磨料磨损转变为磨料磨损和氧化磨损, 而磨屑尺寸的减小可归因于 GCr15 球和钛基体反复摩擦, 导致磨屑的脆化。本研究提供了一种使用农业废稻壳生产具有优良综合性能的钛合金方法, 在降低钛合金成本的同时也有益于生态环境的保护。

**关键词:** 低成本钛合金; 稻壳分解物 SiO<sub>2</sub>; 摩擦学性能; 磨损机制

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