

In-situ micro-CT investigation into wear behavior of Ti-6Al-4V: From ambient to elevated temperatures

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Abstract: Understanding the three-dimensional evolution of internal damage within contact zones during wear is critical for failure analysis, yet it remains experimentally challenging, particularly at elevated temperatures. In this study, a dedicated in-situ wear testing system compatible with computed tomography (CT) and capable of operating at up to 380 °C was developed to address this gap. Rotational wear tests were performed on Ti-6Al-4V alloy at 25 °C, 200 °C, and 350 °C, and a "wear-pause-scan" strategy was employed to capture the progressive evolution of the contact

interface. The results reveal a temperature-dependent evolution of the contact state and a corresponding shift in the dominant wear mechanism, underscoring the close coupling between interfacial structural evolution and macroscopic friction behavior. This work demonstrates the feasibility of in-situ, three-dimensional observation of the wear process from ambient to elevated temperatures for the first time, providing a versatile experimental platform that can be extended to a wide range of materials and contact configurations.

Keywords: In-situ micro-CT, Ti-6Al-4V, elevated temperature wear, contact state evolution, wear mechanism.

1 Introduction

Wear is one of the most dominant failure modes in mechanical systems and is widely encountered in critical engineering sectors such as aerospace [1], transportation [2], and energy equipment [3]. At contact interfaces with relative motion, continuous material loss not only degrades the geometric accuracy of components, but also leads to increased energy consumption, shortened service life, and even sudden malfunction [4-7]. More importantly, the wear process continuously reshapes the micro-topography and contact state of the interface, thereby directly changing its functional characteristics. In the case of sealing interfaces, for example, the wear-induced non-contact gaps can readily create interconnected leakage paths for fluids, resulting in sealing failure [8-11]. Similarly, for electrical or thermal conduction interfaces, interfacial gaps considerably increase the contact electrical or thermal resistance, severely impairing electrical or thermal conductivity [12-14]. Especially at elevated temperatures, material removal mode at contact interface during wear becomes more complicated and will cause more adverse influence on mechanical properties. Therefore, a thorough understanding of the evolution of three-dimensional topography and internal damage within the

contact zone during wear is of great engineering significance for revealing the underlying failure mechanisms and advancing the wear-resistant design of components.

For a long time, extensive experimental studies have been conducted to investigate wear issues. Firstly, the pin-on-disc test is a classic and effective method, often combined with post-mortem characterization techniques such as white light interferometry and scanning electron microscopy [15-18]. Li et al. [19] conducted dry sliding wear tests for Ti-6Al-4V alloy on a pin-on-disc wear tester, and explored the tribo-oxides and their function. Although such a method is straightforward to implement and produce intuitive results, it can only capture the initial and final states of the experiment. To obtain information over continuous timescales, some researchers have developed online monitoring techniques based on physical signals such as acoustic emission and vibration [20-24]. Wu et al. [25] investigated the ultrasound behavior of early-state wear and applied it to the measurement of early-state wear and the identification of surface damage. Feng et al. [26] established a dynamic model to estimate the gear wear profiles by updating the contact force and vibration signals. Although these techniques can indirectly reflect transitions in wear states, they cannot directly image the interior of the contact zone. Optical in-situ observation achieves real-time visual imaging of the contact zone by employing transparent materials [27-28]. Yagi et al. [29] designed an experiment to observe in situ the wear process of a point contact area, created between a rotating sapphire disc and a metal pin. This approach requires at least one of the contacting bodies to be optically transparent, which makes it almost impossible to replicate the contact conditions of real metallic friction pairs. Consequently, observing the dynamic wear process at the interface between non-transparent metallic bodies remains a persistent experimental challenge.

X-ray computed tomography (CT) offers a highly promising pathway to address the aforementioned challenge [30]. Its principle is to acquire X-ray projection images at different angles by rotating the sample, and then reconstruct a three-dimensional voxel model containing internal information using algorithms such as filtered back projection (FBP) [31]. Exploiting the ability of X-

ray to penetrate materials non-destructively, this technique can directly reveal the internal features including pores, cracks, and inclusions [32-35]. Deng et al. [36] used CT to investigate the fracture behavior of carbon fiber/epoxy composite with chopped fiber reinforcements, and revealed the damage evolution in 2D and 3D images obtained from reconstructed tomographic data. Chen et al. [37] combined in-situ plasma spark sintering (SPS) apparatus and laboratory X-ray microscopy, so as to three-dimensionally investigate the dynamic evolution process of 7055 aluminum alloy during the SPS process. In addition, CT is also suitable for the study of contact states between metallic bodies [38-40]. Wang et al. [41] used X-ray CT to measure junction growth when two aluminum alloy surfaces were in contact. Wang et al. [42] employed X-ray CT to measure non-contact clearances at metal contact interfaces, verifying their proposed multi-scale contact algorithm.

Furthermore, some researchers have attempted to apply CT technology specifically to wear studies. Alekseyev et al. [43-44] exposed the annular contacts of 6082-T6 aluminum alloy to various degrees of fretting wear, and then used synchrotron X-ray CT and diffraction to examine the extent and development of wear damage. Lin et al. [45] demonstrated the capability of X-ray micro-CT to quantitatively characterize wear progression by analyzing the volume, morphology, and evolution of wear debris. However, existing studies suffer from two main limitations. Firstly, the wear testing and CT scanning rely on physically separate setups, requiring sample transfer after wear for imaging, which inevitably disturbs the original contact state. Secondly, current work has been conducted almost exclusively at room temperature, leaving a gap in understanding wear behavior at elevated temperatures. Consequently, the dynamic wear process under in-situ, high-temperature conditions remains largely unexplored.

In this study, a dedicated in-situ wear testing system was developed that is fully compatible with micro-CT and suitable for high temperatures. Rotational wear tests were then performed on Ti-6Al-4V alloy at different temperatures. By adopting a “wear-pause-scan” strategy, high-resolution CT images of the contact zone were obtained at different wear stages, thereby achieving in-situ, quasi-

real-time, three-dimensional observation of the evolution of the contact state. Additionally, combined with real-time torque monitoring, post-wear three-dimensional surface profilometry, scanning electron microscopy and energy dispersive spectroscopy, the influence of temperature on the contact zone morphology, evolution of interfacial gaps, and underlying wear mechanisms was systematically investigated. The main contributions of the study are outlined as follows:

- (1) A dedicated in-situ wear testing system fully compatible with micro-CT and capable of operating up to 380 °C was developed. By adopting a "wear-pause-scan" strategy, the in-situ, quasi-real-time, and three-dimensional evolution of internal damage within the contact zone was captured for the first time from ambient temperature to elevated temperatures.
- (2) A temperature-dependent transition in contact state evolution was revealed for Ti-6Al-4V. At room temperature, the contact region expands initially but subsequently deteriorates, with interfacial pores first closing and then sharply regenerating. At elevated temperatures, progressive conformal contact and monotonic pore closure prevail throughout wear.
- (3) A systematic shift in the dominant wear mechanism of Ti-6Al-4V was elucidated: from a mixed mode governed by adhesive wear at 25 °C, accompanied by abrasive and fatigue wear, to an oxidation-dominated mechanism at 350 °C. The formation of a continuous, self-healing oxide layer at elevated temperatures is identified as the fundamental origin of the sustained low and stable friction.

2 Experiment

2.1 Experimental setup

This study employed micro computed tomography (CT) technology for in-situ wear testing at room temperature and high temperatures. The experiments were conducted using the XPloreVista 2000 4D micro-CT system (Weikuang Technology Co., Ltd., Suzhou, China), as shown in Fig. 1(a),

which can provide an adjustable tube voltage range of 40–160 kV and a spatial resolution of up to 2 μm .

To ensure compatibility with the micro-CT, a dedicated in-situ wear testing system was developed, as shown in Fig. 1(b). The system comprises three main components: the in-situ test rig, control cabinet, and custom test software. Serving as the core actuating unit, the in-situ test rig consists of servo motor mounted on the top, stepper motor inside the base frame, sample holders, heating coil, sensors (for temperature, pressure, and torque), along with necessary connecting and transmission parts. The servo motor drives the rotation of sample to achieve relative sliding. The stepper motor adjusts the vertical position of the sample via lead screw, enabling precise control of the normal load. The heating coil is employed to provide and maintain the target test temperature, up to approximately 380 °C. Notably, a connector made of polyether ether ketone (PEEK) was specially designed in the middle section of the rig. This material not only offers sufficient supporting stiffness at high temperatures but also exhibits low X-ray absorption to reduce imaging artifacts. Furthermore, four observation windows were incorporated into the PEEK connector to permit visual inspection and maximize X-ray transmission. The in-situ test rig is connected through signal cables to the PLC-based control cabinet and the upper computer. With the self-developed test software, integrated operations such as motor control, heating, experimental parameter setting, and real-time data acquisition can be achieved. In particular, the normal load, rotational speed, and temperature can be adjusted independently, providing flexible control over the test conditions for systematic parametric studies.

2.2 Material and sample preparation

The raw material selected for this study is Ti-6Al-4V (TC4) titanium alloy, with its chemical composition listed in Table 1. The alloy has an elastic modulus of 110 GPa and a Poisson's ratio of 0.34. Owing to its lower atomic number of Ti compared to common metals such as Fe and Cu, the

alloy exhibits lower X-ray absorption which contributes to higher spatial resolution in micro-CT imaging.

The samples were machined into custom-designed pairs, comprising upper and lower counterparts, as shown in Fig. 1(c). For both upper and lower samples, the main body diameter is 5 mm, providing sufficient stiffness during loading and wear. Both ends of each sample are tapered, with a contact tip diameter of 1 mm to minimize X-ray attenuation by the bulk material in the contact zone. The wear surfaces were treated by electrical discharge machining (EDM), which increased the R_a from less than 1 μm for the machined surface to approximately 10 μm , thereby enhancing the detectability of the contact state. The bottom of each sample features a clamping end with a shoulder to ensure alignment and stability during mounting. Additionally, a diameter hole of 1 mm was drilled into the lower sample to accommodate a thermocouple for real-time temperature monitoring during experiments.

Table 1 Chemical composition (wt.%) of Ti-6Al-4V.

Ti	Al	V	Fe	O	C
Bal.	6.0	4.0	0.3	0.2	0.08

2.3 Experimental procedure

The experimental principle and the procedure are illustrated in Fig. 1(d). First, the in-situ test rig with the mounted sample pair was fixed onto the rotation stage inside the CT chamber. It was then connected to the control cabinet and upper computer, and key experimental parameters were set through the software, including normal load, rotational speed, total number of laps, etc. The testing parameters are summarized in Table. 2. For high-temperature tests, the rig and samples needed to be fully pre-heated until thermal equilibrium, so as to eliminate the influence of thermal expansion on CT imaging quality. Once the preset number of laps was completed, the servo motor stopped. The normal loading module maintained the applied pressure, and the heating module sustained the temperature, thereby keeping the mechanical and thermal state of the contact zone between the

sample pair stationary (i.e., “frozen”) during the subsequent scanning. Subsequently, the micro-CT system was activated to perform high-resolution scan of the static contact zone. During scanning, the in-situ rig rotated continuously with the stage over 360° , collecting 1600 two-dimensional projection images. The scanning parameters were set as follows: voltage 150 kV, current 110 μA , power 16.5 W, exposure time 1.1 s. Under these conditions, each complete scan took about 63 minutes, and the real spatial resolution was 2.322 μm . This “wear–pause–scan” cycle was repeated multiple times to acquire three-dimensional data of the contact state at different stages throughout the wear test.

After each scan, the projection data were processed using three-dimensional reconstruction software based on the filtered back-projection algorithm, yielding a volumetric model of the contact zone. Further visualization and structural analysis were performed using Avizo software. Finally, after the wear tests, the worn surfaces were examined via white-light interferometry, scanning electron microscopy (SEM, ZEISS Sigma 360), and energy-dispersive spectroscopy (EDS) for topographic and compositional characterization.

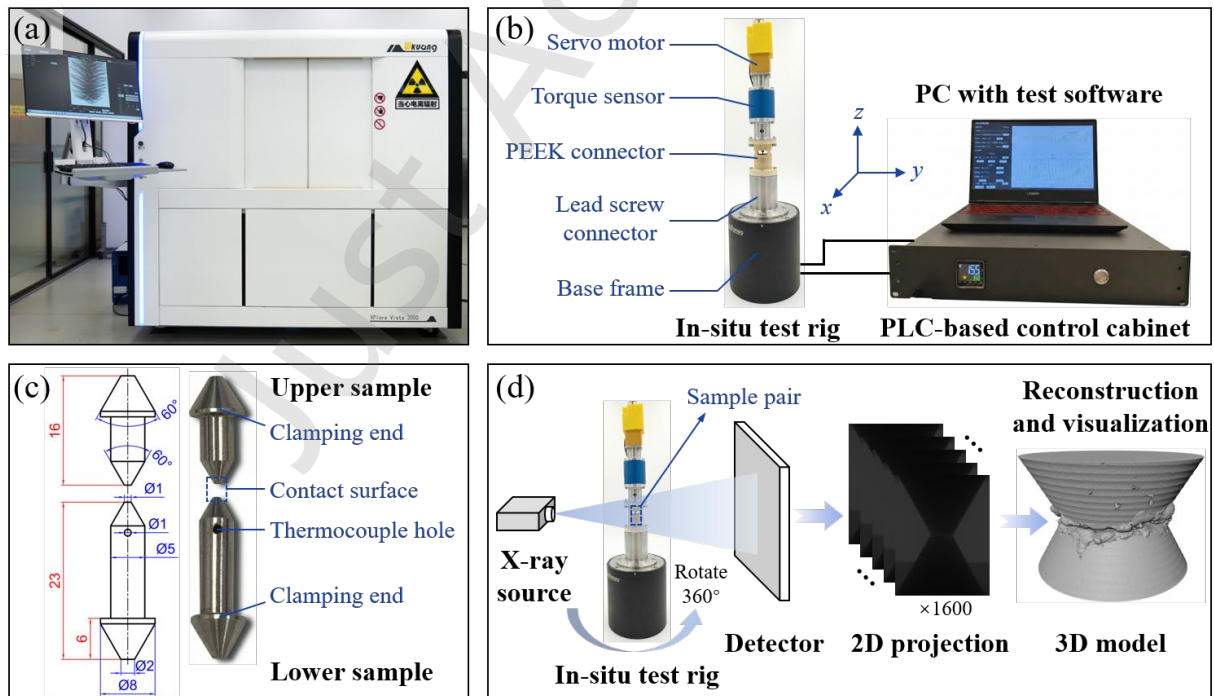


Fig. 1 Experimental setup, sample and scheme. (a) Micro-CT system; (b) in-situ wear testing system; (c) sample pair design; (d) experimental principle and procedure.

Table 2 Summary of the experimental parameters.

Parameters	Values
Temperature (°C)	25, 200, 350
Normal load (N)	100
Nominal contact stress (MPa)	127
Number of laps	0, 15, 50, 100
Rotational speed (rpm)	10
Average linear speed (mm/s, based on an average wear radius of 0.25 mm)	0.26
Cumulative sliding distance (mm)	157

3 Results and discussion

3.1 Frictional behavior and surface morphology

The coefficient of friction (COF) is one of the key indicators for evaluating the tribological performance of materials. In this study, the COF was obtained indirectly by monitoring the real-time torque between the samples during wear process. The conversion from torque to COF was based on the following assumptions: the contact surface of the sample was considered as an ideal disc with radius R ; the normal load was uniformly distributed over the disc. Selecting an infinitesimal ring element on the disk with radius r ($r < R$) and width dr ($dr \ll r$), as shown in Fig. 2(a). The normal load acting on the element is given by:

$$dF_N = \frac{F_N}{\pi R^2} 2\pi r dr = \frac{2F_N r}{R^2} dr \quad (1)$$

where F_N is the total normal load applied on the entire contact surface. Denoting the COF as μ , the friction force acting on the ring during rotation can be expressed as:

$$dF_f = \mu g dF_N = \frac{2\mu F_N r}{R^2} dr \quad (2)$$

The torque required to overcome the friction for the ring is:

$$dT = dF_f r = \frac{2\mu F_N r^2}{R^2} dr \quad (3)$$

Integrating over the entire contact surface yields the theoretical total torque T :

$$T = \int dT = \int_0^R \frac{2\mu F_N r^2}{R^2} dr = \frac{2\mu F_N R}{3} \quad (4)$$

The actual torque was obtained in real time through the torque sensor. Therefore, the real-time COF can be calculated as:

$$\mu = \frac{3T}{2F_N R} \quad (5)$$

It should be noted that the COF obtained based on torque single is an apparent nominal value. Although its accuracy can be affected by the actual contact uniformity, it is sufficient for a preliminary analysis of the frictional state. Fig. 2(b) shows how the COF of Ti-6Al-4V samples evolves with the number of rotational laps at 25 °C, 200 °C, and 350 °C. At 25 °C, due to the rapid morphological changes of the initial rough surface, the COF exhibits significant instability during the initial wear stage. After 8–10 running-in laps, it reaches a minimum value of approximately 0.45. Subsequently, it increases continuously and fluctuates within the range of 0.6–0.8 after about 30 laps. At 200 °C, the COF also shows pronounced variability at the initial stage but quickly stabilizes around 0.4. A slow increase is observed after 60 laps, eventually reaching about 0.55. When the temperature rises to 350 °C, the variation of COF can be divided into three stages. The average during the first 40 laps is approximately 0.27. It then rises and maintains at about 0.38 over laps 45–70, and finally decreases slightly to stabilize around 0.35 during laps 75–100. Overall, the COF tends to decrease with the increasing test temperature. Moreover, the temperature strongly influences the duration of each wear stage. At 25 °C, both the running-in stage and the steady-state wear stage are very short. The samples quickly enter the severe wear stage soon after the initiation of wear, accompanied by a sharp increase in the COF. At 200 °C, although the first stage is also transient, the steady-state wear stage is considerably prolonged, allowing the COF to remain at a relatively low level for an extended period. At 350 °C, no distinct running-in or severe wear stages are observed, and the COF remains relatively stable throughout the entire wear process.

Friction

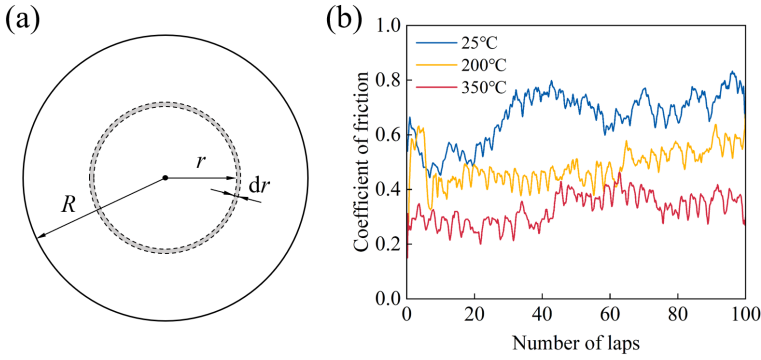


Fig. 2 Calculation method and results of the COF: (a) schematic diagram of the infinitesimal ring element on contact surface; (b) variation of the COF with number of laps at different temperatures.

Fig. 3 and Fig. 4 display the three-dimensional morphology and corresponding two-dimensional profile curves of the final worn contact surfaces obtained after 100 laps at different temperatures, in which (a1)–(c1) correspond to the upper samples, while (a2)–(c2) refer to the lower samples. Table 3 summarizes the roughness parameters of the worn surfaces. At 25 °C, significant material transfer occurs between the upper and lower samples. Wear debris predominantly accumulates on the upper sample, forming distinct local protrusions with a maximum height of about 30 μm. In contrast, the lower sample exhibits substantial material loss, characterized by concentric annular grooves reaching a depth of nearly 20 μm. Consequently, both surfaces exhibit relatively high roughness. At 200 °C, material transfer remains evident. The upper sample develops a conical feature with a raised central region and a depressed periphery. The peripheral zone is mainly characterized by material loss, producing annular grooves which are shallower than those observed at 25 °C. Meanwhile, debris accumulates near the center, forming pile-up approximately 30 μm in height. The lower sample also exhibits annular ridges and grooves, along with some randomly distributed pits. These features originate from electrical discharge machining of the samples. During the wear process, some deep pits are not completely filled or smoothed, thus remaining as residual depressions in the post-wear morphology. At 350 °C, significant material transfer is no longer observed, showing only shallow annular wear marks. Apart from some isolated deep pits, the overall surfaces are relatively flat, with a substantially reduced roughness.

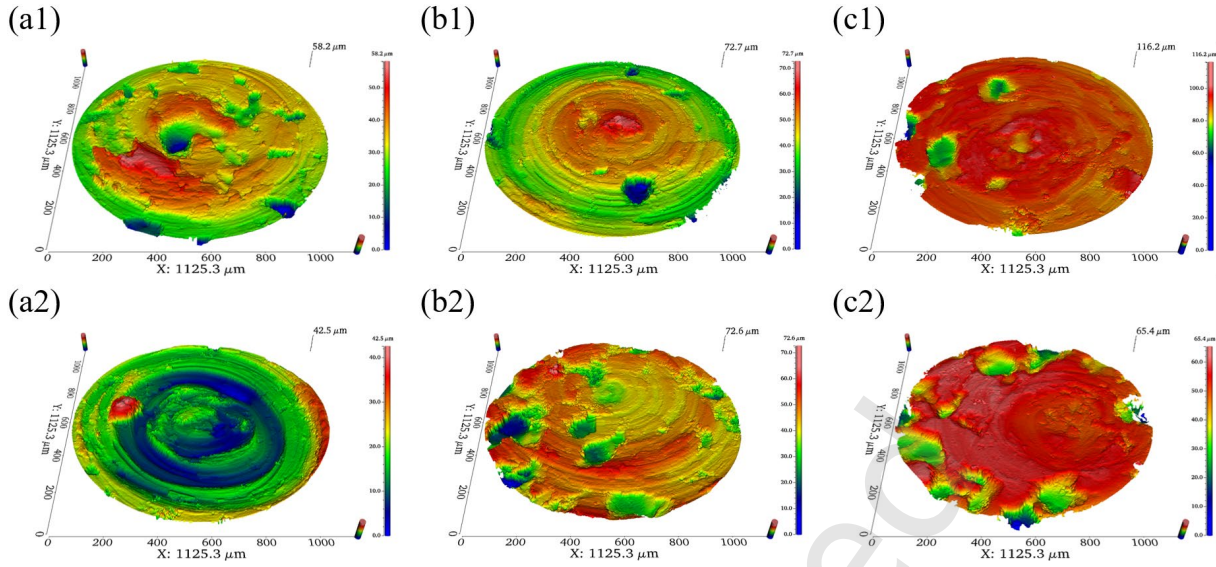


Fig. 3 Three-dimensional morphology of the worn surfaces after 100 laps at different temperatures: (a)25 °C; (b)200 °C; (c)350 °C.

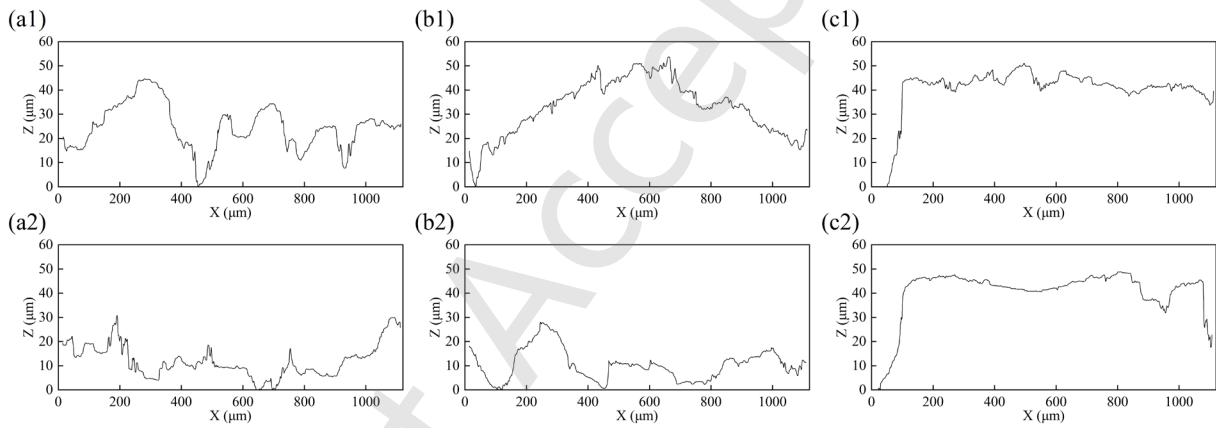


Fig. 4 Two-dimensional profile curves of the worn surfaces after 100 laps at different temperatures: (a)25 °C; (b)200 °C; (c)350 °C.

Table 3 Roughness parameters of the two-dimensional profile curves after wear (data at 350 °C exclude obvious pits)

Temperature	Sample	Ra (μm)	Rq (μm)	Rz (μm)
25 °C	upper	7.481	9.576	44.582
	lower	5.265	6.661	30.776
200 °C	upper	9.460	11.554	53.766
	lower	4.917	6.381	27.942
350 °C	upper	2.268	2.981	17.270

Combining the COF curves with the final worn surface morphologies, the frictional behavior of the samples can be preliminarily interpreted from the perspective of surface morphology evolution. At 25 °C, the final worn surface exhibits pronounced local protrusions and deep grooves with high roughness. This indicates severe material transfer and removal, as well as continuous surface degradation, which leads to a high and strongly fluctuating COF. At 200 °C, protrusions caused by material transfer are still observed, but the grooves are much shallower, the surface roughness is lower, and the morphological evolution is more moderate. This is consistent with the COF remaining stable in the early stage and then increasing slowly in the later stage. At 350 °C, only shallow annular wear marks can be seen, and the overall surface is relatively flat with substantially reduced roughness. This suggests that the contact surfaces remain well conformed throughout wear, providing a direct explanation for the low and stable COF. The temperature dependent of surface morphology and COF further indicate a fundamental shift in the underlying wear mechanisms. To elucidate the origin of these macroscopic differences, further investigation and detailed analysis are required in the following sections.

3.2 In-situ X-ray micro-CT analysis of contact interface evolution

Through in-situ X-ray micro-CT technology, valuable information inside materials during the wear process can be obtained, such as the generation of wear debris, changes in porosity, and evolution of the contact interface. This section provides a systematic analysis of the internal features based on the volumetric data acquired from micro-CT.

Fig. 5 presents the micro-CT slices in the XZ plane at three temperatures (25 °C, 200 °C and 350 °C) and four different numbers of laps (0, 15, 50, 100), illustrating the evolution of the vertical contact state between the upper and lower samples. In CT slices, dark regions correspond to air or internal pores with low X-ray attenuation, whereas bright regions represent solid material. Higher

brightness generally indicates greater local density. In the initial state (Fig. 5(a1)–(c1)), owing to the original surface roughness, actual contact between the upper and lower samples is established only through a limited number of discrete asperities. Consequently, the actual contact area is much smaller than the nominal contact area, which is consistent with classical contact mechanics theory. In the meantime, numerous irregularly shaped pores are present along the contact interface. The filling, closure, or regeneration of these pores during the wear process directly reflects the evolution of the contact state and serves as a key intrinsic indicator for tracking wear progression.

At 25 °C, the interfacial pores between the samples gradually diminish as wear progresses from the initial state to 50 laps (Fig. 5(a1)–(a3)). The opposing contact surfaces become increasingly conformal. However, after 100 laps, larger gaps reappear within the scanning area (Fig. 5(a4)), which is consistent with the obvious roughened surface morphology in Fig. 3(a). This indicates that the first 50 laps mainly correspond to the running-in stage, characterized primarily by plastic deformation of asperities under normal load and frictional shear, initial generation of wear debris, and the filling of pores. In the subsequent 50 laps, the transition of wear mode occurs and the severe wear stage begins. This stage is characterized by accelerated delamination of surface material and a sharp increase in the debris generation rate, ultimately resulting in the formation of new macroscopic pores along the contact interface. In contrast to the room-temperature behavior, at 200 °C the pores exhibit a continuous decreasing trend throughout the entire wear process (Fig. 5(b1)–(b4)). The contact surfaces become more conformal, and the actual contact area increases progressively. At 350 °C, this conformal process is further accelerated, and by only 50 laps the contact surfaces have already become highly conformal (Fig. 5(c1)–(c3)). After 100 laps, the interface appears nearly fully conformal at the present slice location and imaging resolution, with no discernible gaps remaining (Fig. 5(c4)). In addition, some low-grayscale shadows can be observed outside the nominal contact area, corresponding to loose debris that remains uncompacted and is expelled from the contact region during wear.

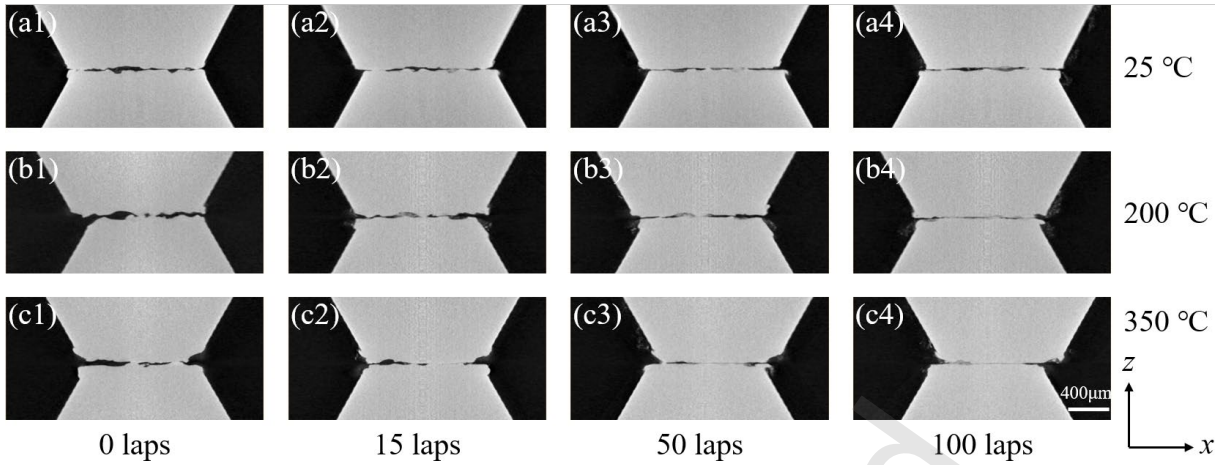


Fig. 5 Micro-CT slices in the XZ plane at different temperatures and laps, in which (a)–(c) correspond to temperatures of 25 °C, 200 °C, and 350 °C, respectively; labels 1–4 correspond to numbers of laps of 0, 15, 50, and 100, respectively. The numeric labels 1–4 in Figs. 6–9 represent the same meaning.

Figs. 6–9 show the slice sets in the XY plane acquired at 25 °C, 200 °C and 350 °C, respectively, with each set containing different slice indices and numbers of laps, which can be used to analyze the evolution of pore morphology and contact states on the horizontal plane. The selected slices are located near the central contact plane of the samples, and the interval between two adjacent slices is 5 voxels, corresponding to 11.6 μm. It should be noted that the exact position of the central plane varies among different tests due to sample dimensional tolerances, alignment conditions, and the cropping range during post-processing. In addition, the central plane may also shift slightly during wear owing to material removal or plastic flow. Therefore, it is not necessary for the slice indices to be identical across different temperatures; it is sufficient to ensure that the selected slices cover the central contact plane and its vertical offset range.

As shown in Fig. 6, in the initial contact state at 25 °C, the central plane is located around index 245. Within this plane, the actual contact regions between the samples are mainly distributed as discrete island-like spots, separated from each other by extensive pore areas (Fig. 6(b1)). In comparison, slices at indices 240 and 250 exhibit a higher area fraction of solid material with better connectivity, whereas the pores appear sparser and more dispersed (Fig. 6(a1) and (c1)). Both the contact spots and pore regions exhibit irregular geometries without apparent directionality, and the

boundaries between them are relatively distinct. After 15 laps, no fundamental change in the contact state is observed. The main developments are the gradual expansion and interconnection of contact spots, as well as the shrinkage and closure of pores (Fig. 6(a2)–(c2)).

After 50 laps, the morphology of the contact interface exhibits notable changes. In the slice at index 240 (Fig. 6(a3)), most pre-existing pores have been filled and compacted, leaving only some isolated larger cavities. A ring-shaped region with intermediate grayscale appears near the periphery, corresponding to the annular wear track. At index 245 (Fig. 6(b3)), the pores are further reduced, and the distribution of contact and pore regions transitions from a dispersed pattern toward a more concentrated morphology. Meanwhile, the boundaries become more diffuse, indicating that the interior of the pores and regions near the boundaries have been partially filled with loose debris particles. At index 250 (Fig. 6(c3)), the pores are almost completely filled and compacted, and even the few remaining pores exhibit obvious debris accumulation. When rotated to 100 laps, the contact state undergoes a drastic transition once again. At index 240 (Fig. 6(a4)), the annular wear track becomes more pronounced. At index 245 (Fig. 6(b4)), regions only near the center, at certain mid-radial positions and along peripheral arcs can provide effective solid contact, with large pore areas emerging between the opposing surfaces. At index 250 (Fig. 6(c4)), a large circular cavity appears in the center, with substantial debris accumulation observed within the central cavity.

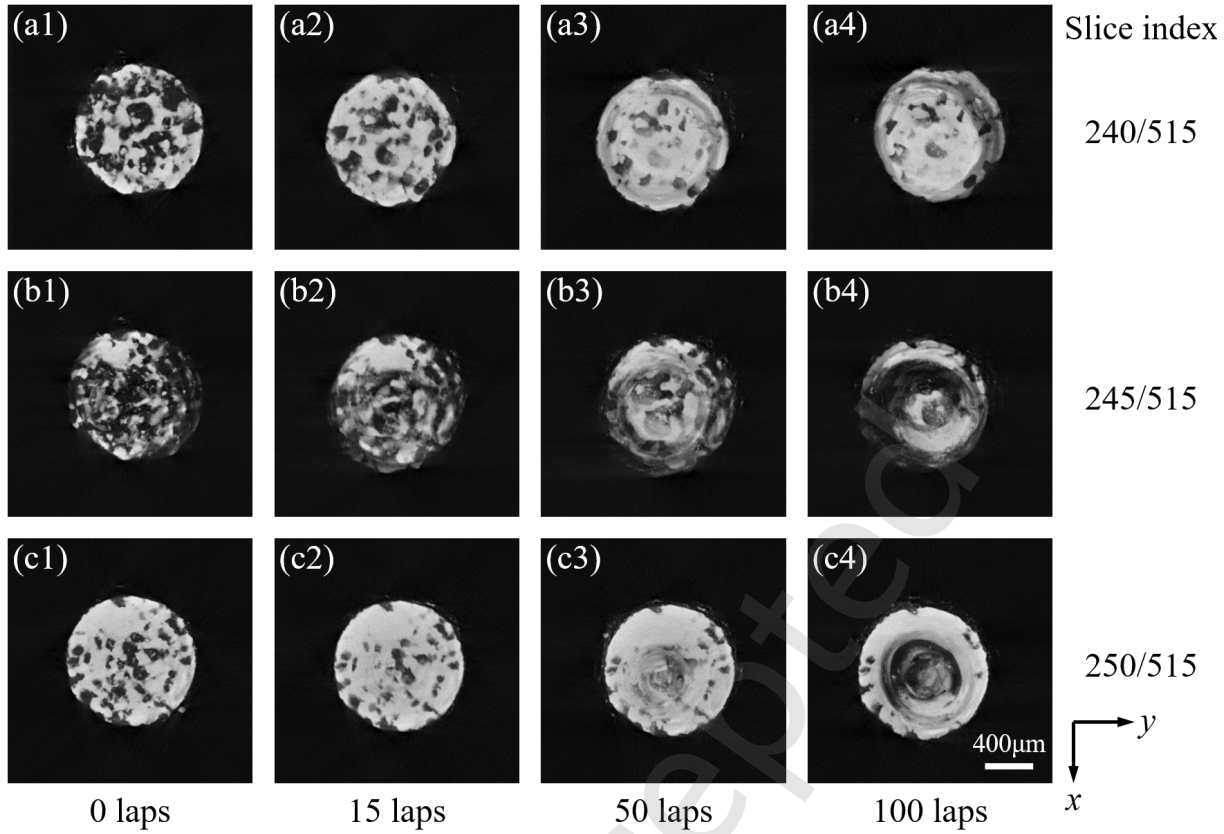


Fig. 6 Micro-CT slices in the XY plane at different slice indices and laps at 25 °C, in which (a)–(c) correspond to slice indices of 240, 245, and 250, respectively.

In the initial contact state at 200 °C, the contact region is also characterized by discrete island-like spots with limited interconnectivity (Fig. 7(a1)–(c1)). However, after only 15 laps, the contact state exhibits pronounced changes. In the slice at index 235 (Fig. 7(a2)), the contact region expands substantially in both radial and circumferential directions relative to the initial state, which contains a small number of residual cavities and an annular wear track. At index 240 (Fig. 7(b2)), the contact region becomes concentrated near the center, while the peripheral portion of the sample is occupied by large void areas. At index 245 (Fig. 7(c2)), the contact region considerably expands and becomes more interconnected based on its initial geometric shape and distribution. Only a few isolated contact spots remain near the periphery. During the wear process from 15 to 50 laps (Fig. 7(a3)–(c3)), the contact region continues to expand and connect, and the width of the annular wear track increases. After 100 laps, a comparison among the three slices reveals that the central contact plane has shifted upward, possibly reaching the vicinity of the slice at index 235. Within this plane (Fig. 7(a4)), the

contact area remains at a relatively high level, and the annular wear track fades to a large extent. In the slices at indices 240 and 245 (Fig. 7(b4)–(c4)), due to the considerable distance to the central plane, the imaged regions consist almost entirely of solid material. The pore areas are quite limited and it is difficult to maintain connectivity.

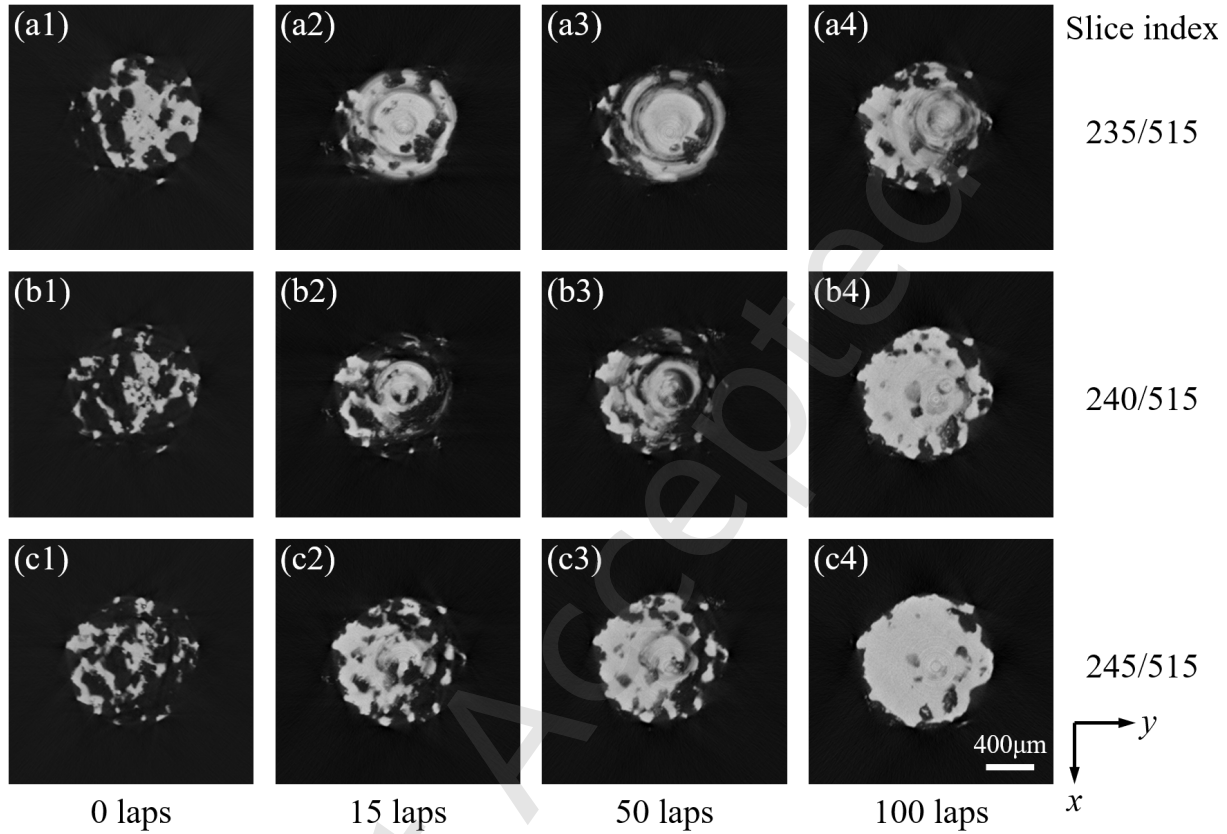


Fig. 7 Micro-CT slices in the XY plane at different slice indices and laps at 200 °C, in which (a)–(c) correspond to slice indices of 235, 240, and 245, respectively.

As shown in Fig. 8, the overall trend of contact state evolution at 350 °C is similar to that observed at 200 °C. As the number of rotational laps increases, the contact region progressively expands, the pore area decreases, and the central contact plane moves upward. After 100 laps, large-area effective contact is essentially achieved between the upper and lower samples, with very few residual pores remaining. A notable difference is that at 350 °C, the boundaries between the contact and pore regions are highly diffuse in some slices (such as Fig. 8(a3), (b3), (a4)–(c4)), exhibiting a grayscale gradient transition. This is attributed to the significant plastic flow and material transfer occurring at the contact interface at elevated temperature. The wear debris is thoroughly comminuted, compacted,

and intermixed with the substrate, forming a debris layer with a certain density. In CT images, the grayscale of this layer lies between that of dense solid material and pores, thereby eliminating the abrupt boundary between the two regions and resulting in the observed diffuse boundary characteristics.

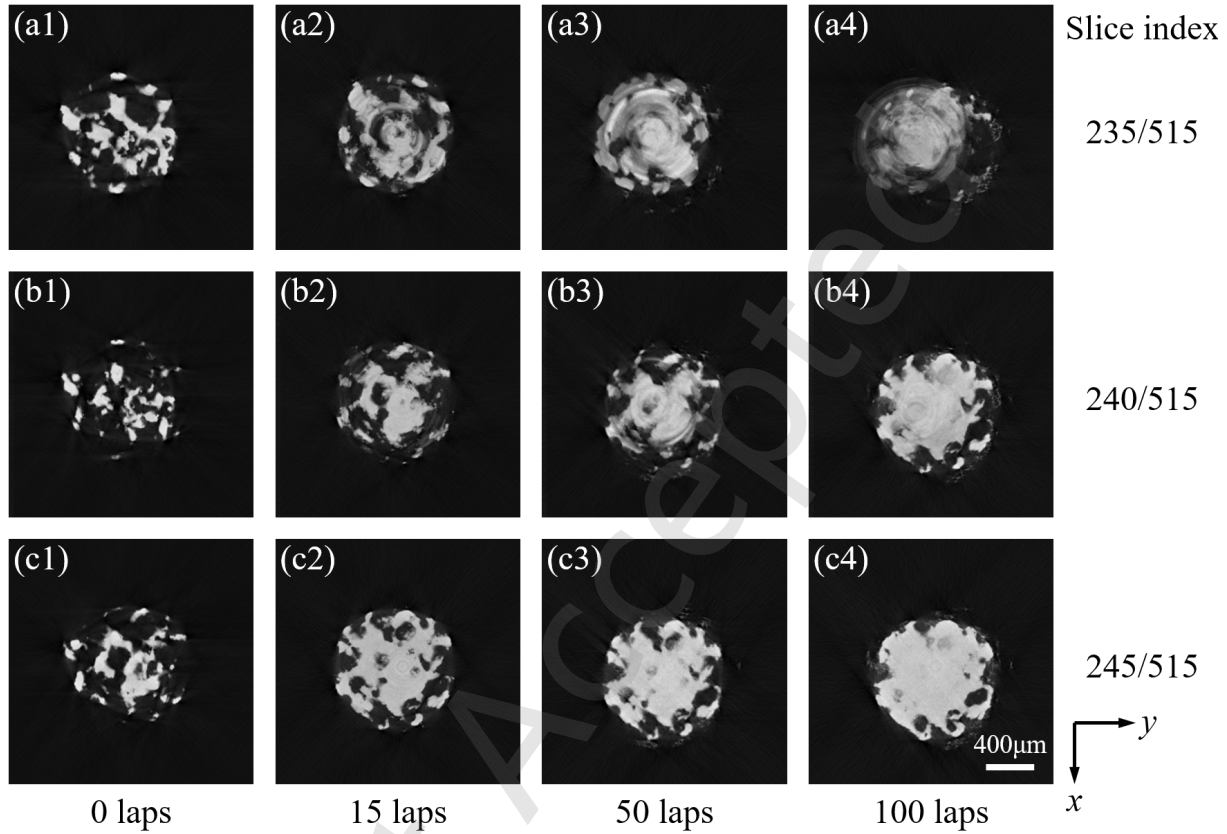


Fig. 8 Micro-CT slices in the XY plane at different slice indices and laps at 350 °C, in which (a)–(c) correspond to slice indices of 235, 240, and 245, respectively.

To more clearly and intuitively represent the three-dimensional spatial distribution and evolution of pores at the contact interface during the wear process, a cylindrical volume was selected and subjected to binary segmentation. The segmentation threshold was manually determined from the grayscale histogram of the initial unworn state for each temperature to ensure accurate identification of pore boundaries, since the presence of wear debris weakens the bimodal distribution of the histogram and makes automatic thresholding unreliable. Once determined, the same threshold was applied to all wear stages at that temperature. Voxels with grayscale values below the threshold were assigned a value of 1 and rendered in blue, representing pores or low-density regions, whereas voxels

above the threshold were assigned a value of 0, i.e., left blank, representing solid material. The three-dimensional models of the pores at different temperatures and numbers of laps were thus reconstructed, as shown in Fig. 9.

Consistent with the observations from two-dimensional slices, the three-dimensional pores are extensively and continuously distributed in the initial contact state (Fig. 9(a1)–(c1)). The actual contact spots are discretely and randomly embedded within the pore network. There are several distinct protrusions on the surface of the pore model, corresponding to the initial machined pits on the sample surfaces. After 15 laps (Fig. 9(a2)–(c2)), the pore volume decreases, and some contact regions begin to merge spatially. The number and height of the protrusions on the model are reduced, indicating that the initial surface pits are gradually smoothed by plastic deformation and debris filling. After 50 laps (Fig. 9(a3)–(c3)), the contact region further expands and tends to concentrate near the center to form a more continuous central contact area. The pores are mainly distributed near the periphery of the sampled volume. Additionally, the boundaries of the contact regions at room temperature are relatively fragmented, and there are numerous tiny pores near the boundaries. In contrast, the higher the testing temperature, the more complete and smoother the boundary between contact area and pores. After 100 laps, the spatial distribution of pores under room and elevated temperatures exhibits more significant differences. At 25 °C (Fig. 9(a4)), the samples undergo severe wear in this stage, and the pore volume increases sharply, occupying nearly the entire sampled volume. This is manifested spatially as a large sheet-like cavity. At 200 °C and 350 °C (Fig. 9(b4)–(c4)), extensive solid contact is maintained in the central region, and pore areas are relatively limited and primarily located at the periphery. The distribution exhibits distinct radial non-uniformity, which arises from the difference in linear velocity during rotational wear. The linear velocity is low near the center, resulting in relatively mild wear, whereas it is higher at the periphery, leading to more severe wear and greater material loss.

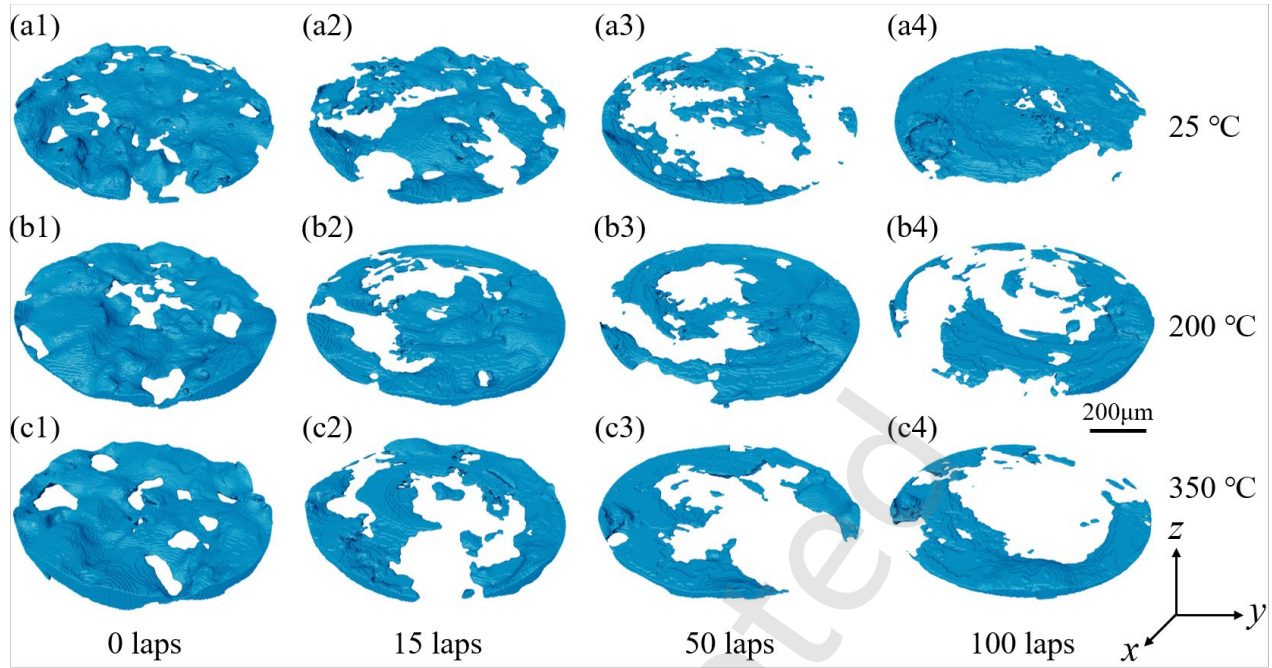


Fig. 9 Reconstructed three-dimensional model of pores at different temperatures and laps, in which (a)–(c) correspond to temperatures of 25 °C, 200 °C, and 350 °C, respectively.

To further quantify the evolution of pores, slice-by-slice image segmentation and statistical analysis were performed based on the volumetric data. Fig. 10(a)–(c) show the areal porosity per slice of the XY plane at different temperatures and number of laps. As wear progresses, the curves exhibit two systematic changes, except for the anomalous state at 25 °C after 100 laps. First, both the peak and overall height of the curves gradually decrease, indicating a continuous reduction in the maximum areal porosity and an overall downward trend of the porosity on each slice. Second, the distribution width of the curves gradually narrows, indicating that the number of slices with identifiable pores decreases, that is, the height of the pore layer gradually decreases. The volumetric proportion of pores within the sampled space was further calculated to obtain the bulk porosity, as shown in Fig. 10(d). Noted that the initial bulk porosity is closely related to the surface roughness and the specific morphology of the samples and thus exhibits considerable randomness. Therefore, the relative trends with increasing number of rotational laps are more meaningful than the comparison of absolute values. It can be seen that at room temperature, the bulk porosity decreases

continuously over the first three states but rebounds anomalously after 100 laps. At elevated temperatures, the bulk porosity decreases monotonically, with comparable reduction magnitudes in both cases.

In addition, the slice with the largest areal porosity in each set is defined as the central contact plane, and its vertical offset is noteworthy, as shown in Fig. 10(e). Similarly, because the initial position of the central plane is influenced by the alignment conditions and cropping range, only the relative variation is analyzed. The results show that at room temperature, the central contact plane gradually shifts downward as wear progresses, which can be attributed to the asymmetric wear behavior of the upper and lower samples. The lower primarily experiences material loss, while the upper receives material compensation through debris adhesion and accumulation to some extent. However, at elevated temperatures, material softening reduces the likelihood of asymmetric wear. The material loss rates of both upper and lower samples are comparable, and exceed the rate of debris accumulation. Since the upper samples are fixed along the vertical direction, and the lower samples are driven upward to compensate for the displacement and maintain contact pressure, this results in an upward migration of the central contact plane.

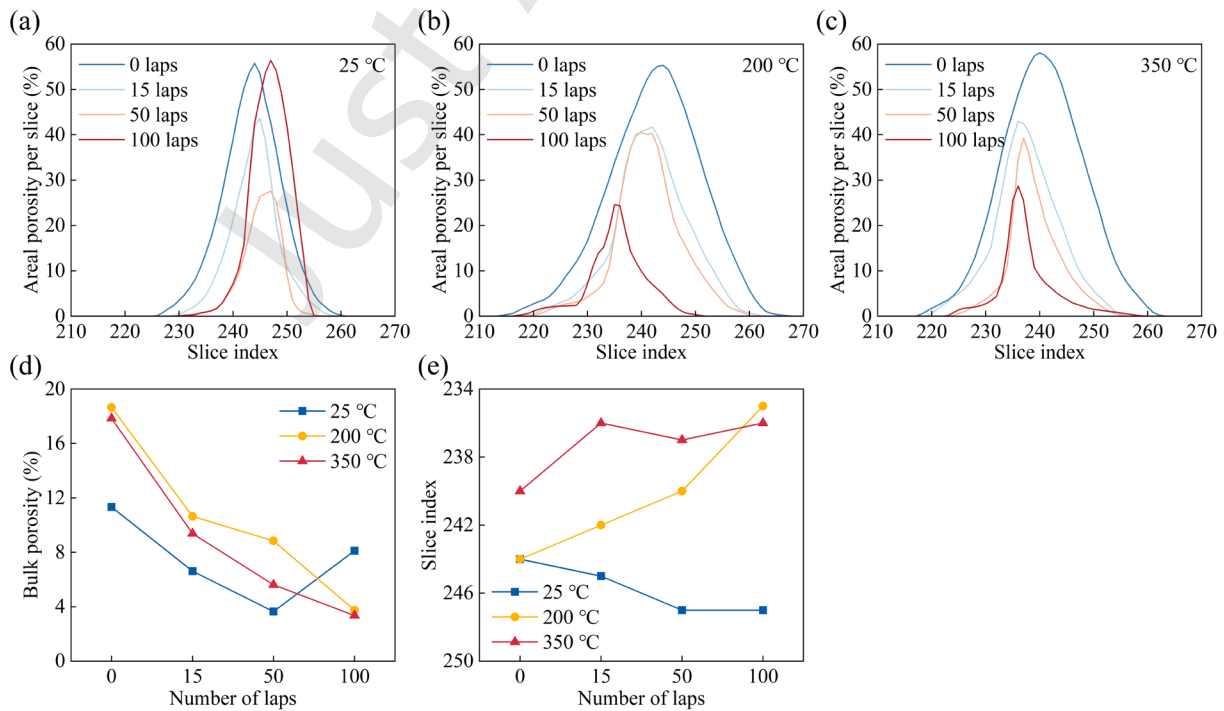


Fig. 10 Areal porosity per slice and the related quantities at different temperatures and laps: (a) 25 °C; (b) 200 °C; (c) 350 °C; (d) bulk porosity; (e) position of the central contact plane.

3.3 Microscopic analysis of wear mechanisms

The preceding CT analysis has elucidated the evolution of pores and contact state at the micron scale. However, limited by the scanning resolution, information on a more microscopic scale remains unresolved, such as crack initiation and propagation behavior, and the precise morphological features of the worn surfaces. In this section, the worn surfaces are further characterized using SEM and EDS to obtain higher-resolution morphological and compositional information, thereby improving the understanding of the underlying wear mechanisms.

Figs. 11–13 present the microscopic morphology of worn sample surfaces at 25 °C, 200 °C and 350 °C, respectively, in which (a)–(c) correspond to the upper sample and (d)–(f) to the lower. Fig. 11(a) shows obvious bulk debris, which originates from adhesive effects at the contact interface. Subsequently, the material experiences tearing, detachment and transfer, ultimately leaving debris within the surface depressions. Fig. 11(b) shows a bulk debris particle at a late stage of formation, characterized by a large and deep crack along its periphery. This type of cracks typically initiates from pre-existing material defects and propagate rapidly under stress concentration. Once the crack reaches the edge, the affected material separates from the substrate and forms free debris. Due to the irregular propagation of these cracks, the edges of debris generated at room temperature are generally sharp and also highly irregular in shape. In addition, although cracks possess considerable length (up to more than 10 μm), their width is far smaller than the dimension of a single voxel, making them undetectable in CT results. Surfaces in Fig. 11(d)–(f) are relatively flat and exhibit numerous flattened debris particles, a morphology commonly observed during three-body abrasive wear. The flattened debris particles exhibit a loose and porous structure, indicating that they have

experienced complex shear and compressive processes at the interface and may further evolve into a debris layer or a third-body layer with specific morphological characteristics.

Fig. 11(e) shows band-like wear tracks that are wide but shallow, consistent with the morphological characteristics of ploughing grooves. This occurs when hard abrasive particles or asperities slide over a relatively softer material surface without causing significant cutting. Instead, material is displaced to both sides through plastic plowing, creating alternating ridges and grooves. In Fig. 11(c), a group of approximately parallel cracks oriented perpendicular to the sliding direction can be observed in the raised regions between grooves. This is attributed to the uniform shear stress field in the subsurface generated by continuous rotational wear, which induces the simultaneous initiation and parallel propagation of multiple cracks. The group of cracks is exactly a typical feature of fatigue wear. A group of parallel cracks perpendicular to the sliding direction is also visible in Fig. 11(d), with finer sizes and larger spacing. They tend to be distributed in flatter regions where the stress field is more dispersed and the degree of fatigue damage is lighter than that within repeatedly plowed and compressed grooves. Fig. 11(g) presents the EDS result corresponding to the region in Fig. 11(d). The elemental map for oxygen appears rather dark and exhibits inhomogeneous distribution. The flat area on the left shows very low oxygen content, whereas the debris-containing region on the right displays slightly higher content. The distributions of the other three elements are relatively uniform.

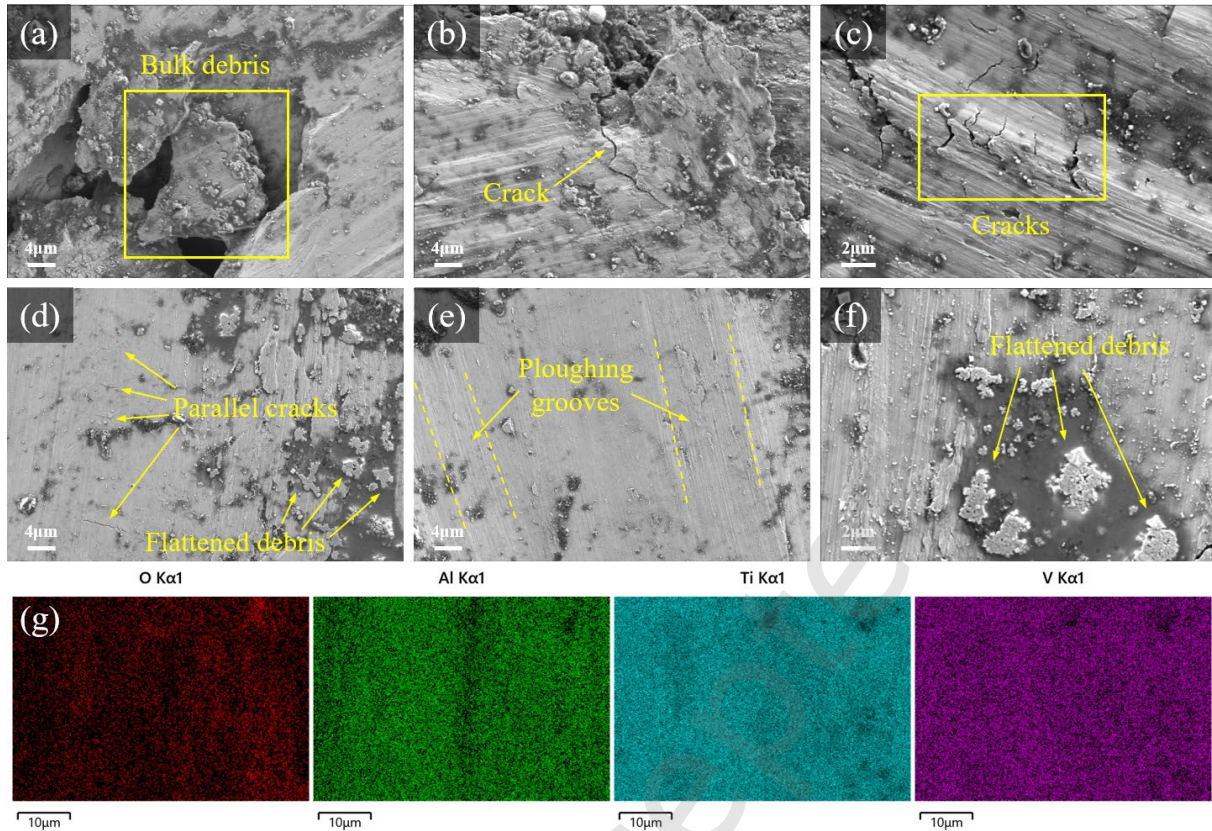


Fig. 11 Microstructural characterization of worn surfaces at 25 °C: (a)–(c) are located on the upper sample; (d)–(f) are located on the lower sample; (g) is the elemental map of the entire area shown in (d).

At room temperature, the wear mechanism of the Ti-6Al-4V alloy exhibits a mixed mode dominated by adhesive wear, with abrasive wear and fatigue wear as concurrent supplements. The predominance of adhesive wear is substantiated by the extensive evidence of material transfer (Fig. 3(a)) and the presence of pronounced bulk debris (Fig. 11(a)). Meanwhile, abrasive wear operates as a universal mechanism throughout the entire wear process, involving both two-body and three-body abrasion. Fatigue wear plays a supplementary role and is manifested by multiple groups of parallel cracks oriented perpendicular to the sliding direction. A notable synergistic and mutually transformative relationship exists among these wear mechanisms. On one hand, adhesive wear and fatigue wear can directly cause material loss; on the other hand, the wear debris they generated can enter the contact interface and freely roll or slide as third-body abrasives. These debris particles exert pronounced micro-cutting and plowing actions on the contact surfaces, further aggravating surface damage. In turn, the surface roughening and stress concentration induced by abrasive wear promote

additional adhesive actions and propagation of fatigue cracks. The mutual reinforcement among the wear mechanisms ultimately accounts for the macroscopic tribological behavior observed at room temperature, such as substantial material transfer and high frictional resistance.

The microscopic morphology at 200 °C exhibits characteristics significantly different from those under room temperature, as shown in Fig. 12. The rough surface in Fig. 12(d) presents deep grooves formed by intense plowing during wear, accompanied by pronounced ridge-like accumulations of displaced material along both sides of the grooves. As wear continues, asperities, bulk debris, and ridges are further flattened by repeated compression, as illustrated in Fig. 12(a) and (b). These flattened marks extend over a considerable distance along the sliding direction but are short in the vertical direction. This indicates that unlike the brittle fracture behavior at room temperature which tends to generate long cracks and large bulk debris, material softening and plasticity enhancement occur at 200 °C. Consequently, the material is more prone to undergo plastic flow under the action of tangential friction. Moreover, although the overall surface remains rough, the edges of the protrusions appear smoother and more rounded. Fatigue cracks remain visible in high-stress regions such as the interior of grooves (Fig. 12(c) and (f)), indicating that the continuous accumulation of subsurface damage under cyclic loading persists.

In Fig. 12(e), some fine debris different from the bulk debris at room temperature is observed. EDS analysis of this area (Fig. 12(g)) reveals that the fine debris exhibits a higher oxygen content than the surrounding substrate, suggesting that it consists of oxide particles generated at elevated temperatures. As a result, oxidative wear begins to participate in the surface evolution. The formation of such oxide particles originates from complex thermo-mechanical-chemical coupling. Firstly, high temperature accelerates the reaction between the metal surface and oxygen to produce an oxide layer that is harder and more brittle than the substrate. At this stage, however, the oxide layer is discontinuous, insufficiently thick, and inadequately bonded to the substrate. Moreover, the mismatch in thermal expansion coefficients between the oxide and substrate gives rise to substantial

internal stress. Consequently, under the combined action of tangential friction and cyclic contact stresses, this brittle oxide layer readily fractures and partially spalls, forming initial particulate or flake-like debris. Once these fragments enter the contact region, they are further crushed and comminuted under the immense contact pressure between the upper and lower samples, and the finer particles appear. During this process, the oxide particles also exert micro-cutting and plowing actions on the substrate, producing the narrow but deep grooves observed in Fig. 12(e). Finally, owing to their higher hardness compared with the substrate, these oxide particles are not easily flattened and incorporated into the contact region. Instead, they are progressively expelled from the contact region and accumulate along the sides of grooves or within surface depressions.

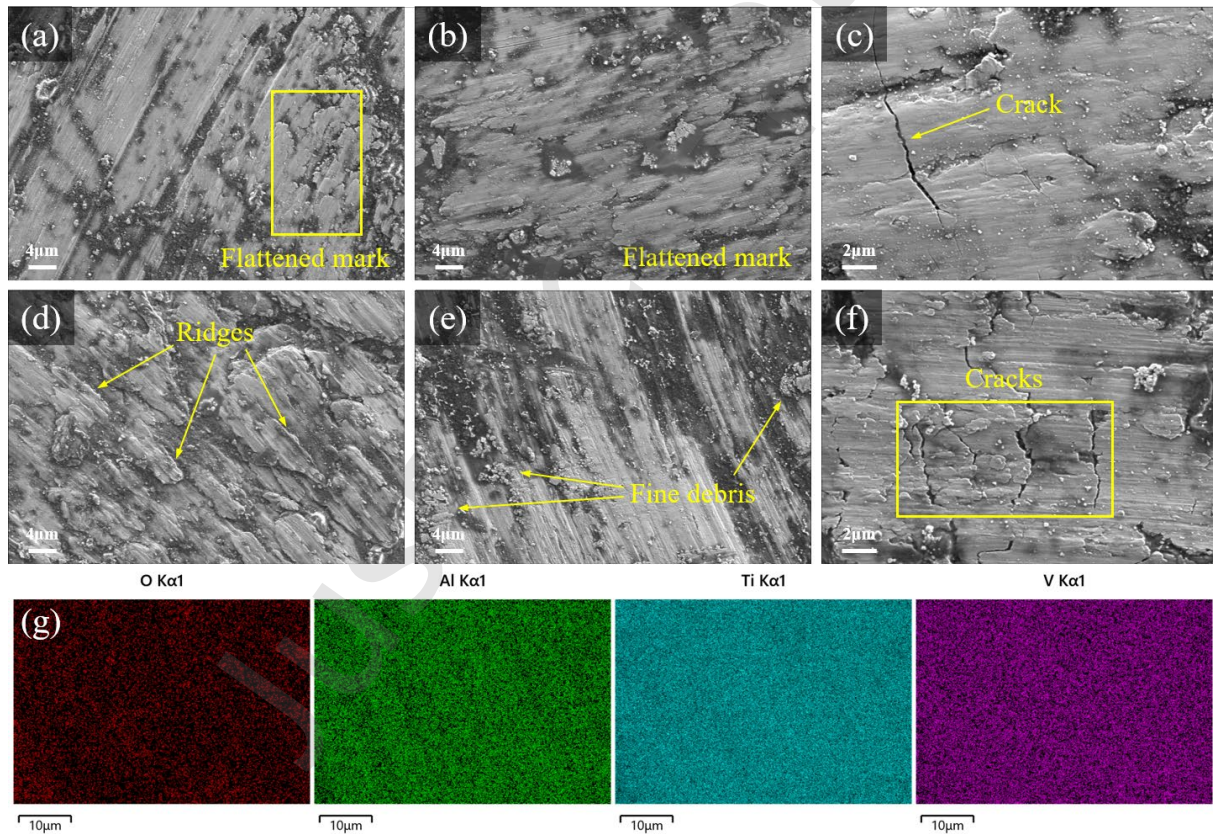


Fig. 12 Microstructural characterization of worn surfaces at 200 °C: (a)–(c) are located on the upper sample; (d)–(f) are located on the lower sample; (g) is the elemental map of the entire area shown in (e).

At this temperature, the wear mechanism of Ti-6Al-4V alloy undergoes a notable transition. While abrasive and fatigue wear continue to play significant roles, oxidative wear emerges as a non-negligible factor, and the extent of adhesive wear is substantially diminished. This is primarily

attributed to the crucial regulatory effects exerted by the elevated temperature and the generated oxide layer on the wear behavior of samples. On one hand, material softening occurs at elevated temperatures, which reduces the yield strength and enhances the plasticity. This increases the actual contact area and reduces the contact stress, even under the same nominal load. Therefore, the tendency for surface adhesion is reduced and surface flattening is promoted. On the other hand, elevated temperature accelerates the formation of oxide layer on the sample surface. This layer effectively transforms direct metal-to-metal contact into metal-to-oxide or oxide-to-oxide contact, thereby significantly mitigating adhesion and material transfer. Moreover, the oxide layer with low shear strength and frictional resistance provides a certain degree of solid lubrication. Consequently, the coefficient of friction at 200 °C is markedly lower than that at 25 °C and remains relatively stable over a considerable number of laps.

At 350 °C, the sample surfaces exhibit further evolution while retaining some features similar to those observed at 200 °C. For instance, in Fig. 13(a) and (b), abundant fine oxide particles can be seen expelled from the contact region and distributed along the sides of ploughing grooves and within deep surface grooves. Meanwhile, in Fig. 13(c), the enhanced plasticity of the material becomes more pronounced. Severe plastic deformation occurs in localized regions, even filling previous grooves to smooth the worn surface. The primary distinction lies in the intensified degree of oxidation. At 350 °C, the surface oxidation rate is high enough to promote the formation of a thicker and more continuous oxide layer. In Fig. 13(d), distinct spalling marks of the oxide layer are visible on a relatively flat surface, which is the prerequisite process for the generation of oxide particles. Besides, there are some microcracks on the flattened surface shown in Fig. 13(e). During the formation and cooling of the continuous and thick oxide layer, internal stress accumulates as a result of volume changes and constraint from the substrate. Given the inherent brittleness of the oxide layer, cracks initiate and propagate rapidly once the local stress exceeds its fracture strength. Unlike the fatigue cracks which are strictly perpendicular to the sliding direction, these elongated microcracks

exhibit no obvious directional preference. When the accumulated stress becomes sufficiently high and the energy release rate is large enough, cracks originate from surface defects or stress concentration sites and propagate radially, forming a more extensive crack network (Fig. 13(f)). EDS analysis of the region in Fig. 13(f) reveals a markedly elevated oxygen content on the surface, providing direct evidence for an oxidation-dominated process (Fig. 13(g)) [46-49].

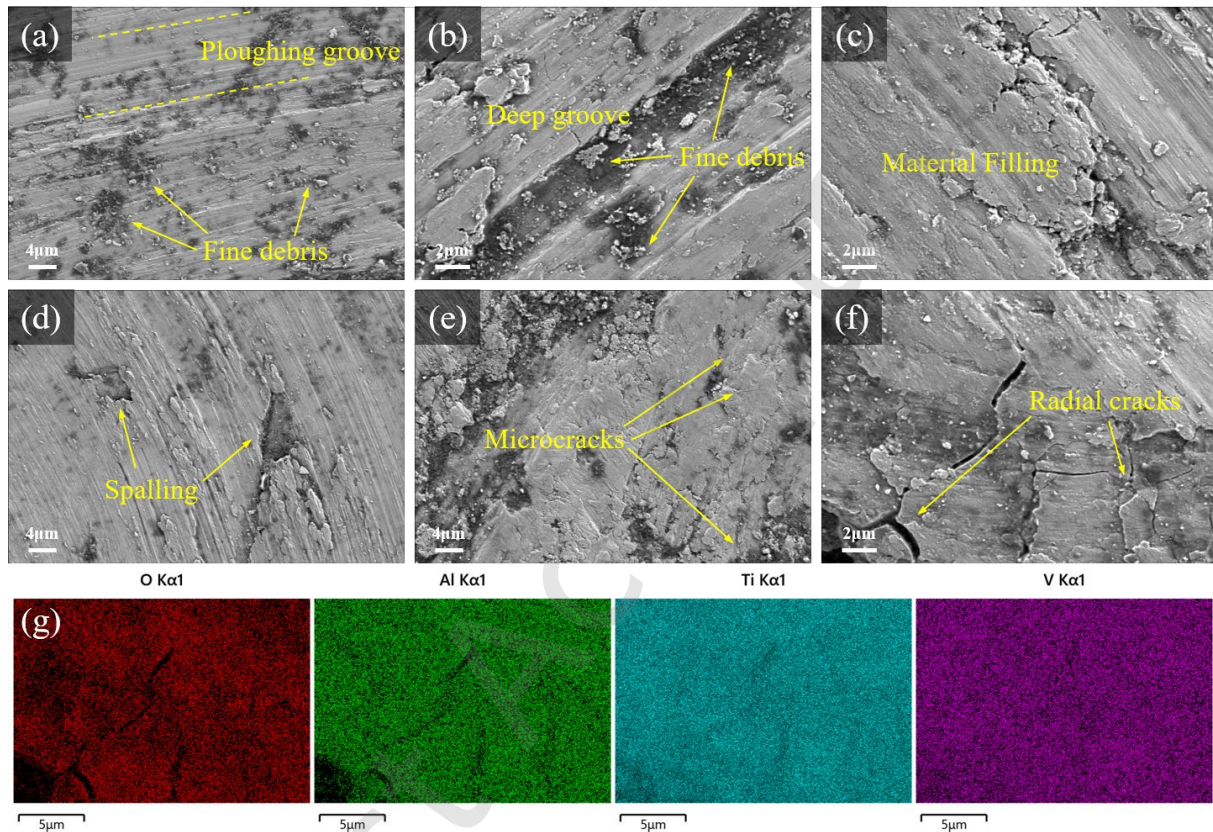


Fig. 13 Microstructural characterization of worn surfaces at 350 °C: (a)–(c) are located on the upper sample; (d)–(f) are located on the lower sample; (g) is the elemental map of the entire area shown in (f).

Based on the morphological features described above, it can be concluded that the wear mechanism at 350 °C undergoes a fundamental transition. Oxidative wear becomes the dominant mechanism, accompanied by pronounced plastic deformation, whereas the contribution of adhesive wear and fatigue wear is substantially diminished. At this temperature, the surface oxide layer evolves from the discontinuous state at 200 °C into a continuous and fully-covering layer. The origin of surface cracks and wear debris shifts from adhesion and fatigue to internal stress accumulation and brittle fracture within the oxide layer. Concurrently, the shear resistance at the interface

transforms from plastic adhesion between metallic asperities to shear or sliding of the oxide layer. Moreover, the enhanced plasticity of the material at this temperature enables the filling of surface grooves, thereby smoothing the worn surface to a certain extent and contributing to a further reduction in the COF. More importantly, when the surface oxide layer cracks or spalls, the freshly exposed substrate rapidly re-oxidizes, regenerating a lubricating oxide film. This immediate self-healing capability is supported by indirect evidence such as the final oxygen content and the CT interfacial evolution characteristics, and it is the key reason why the COF at 350 °C can be sustained at a low level over an extended period with a notably stable friction curve.

Quantitative analysis of the oxygen content at three temperatures was performed by randomly selecting three regions on each sample, as shown in Fig. 14. The results exhibit a systematic increase with rising temperature, clearly reflecting the temperature dependence of the degree of surface oxidation. At 25 °C, the oxygen content is generally below 20%, indicating mild oxidation mainly due to the slow adsorption of ambient oxygen and the limited promoting effect of frictional heat. At 200 °C, the oxygen content rises to the range of 16%–22%, corresponding to the discontinuous oxide layer and a mixed wear mechanism involving the participation of oxidative wear. When the temperature increases to 350 °C, the oxygen content further increases, with all measured values exceeding 20% and reaching a maximum of 25.8%. The presence of a well-developed oxide layer and the predominance of oxidative wear are thus confirmed. The results provide direct elemental evidence to identify the dominant wear mechanisms at different temperatures and offer quantitative support to assess the evolution of wear mechanisms.

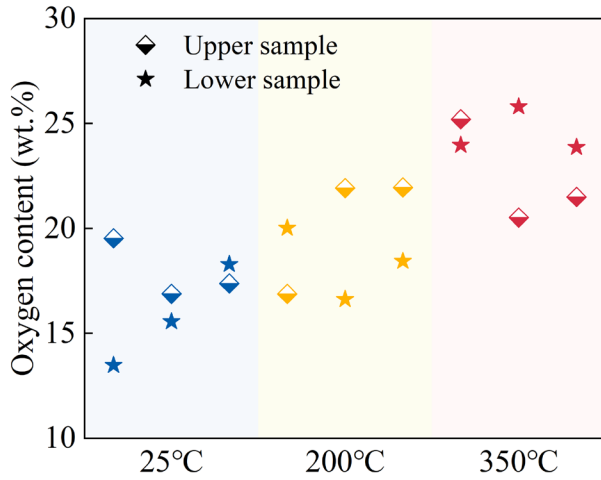


Fig. 14 Quantitative statistics of oxygen content on the worn surface at different temperatures.

4 Conclusions

In this research, the wear behavior of Ti-6Al-4V alloy was systematically investigated from room temperature to elevated temperatures using the developed in-situ micro-CT wear testing system. The contact interface was tracked at different wear stages, supplemented by COF monitoring, surface profilometry, and SEM/EDS characterization. The main conclusions are as follows:

- (1) The COF decreased markedly with temperature, from 0.6–0.8 at 25 °C to approximately 0.35 at 350 °C. At room temperature, a brief running-in stage was quickly followed by severe wear; at elevated temperatures, stable low friction was sustained over extended sliding. Moreover, there was significant material transfer and high roughness at 25 °C, whereas worn surfaces gradually became flatter and smoother as the temperature increased.
- (2) In-situ micro-CT demonstrated distinct temperature-dependent contact state evolution. At 25 °C, interfacial pores were initially filled but severe wear regenerated large gaps after 100 laps. In contrast, at 200 °C and 350 °C, progressive pore closure and conformal contact developed throughout the wear process.
- (3) At room temperature, a mixed mode dominated by adhesive wear, accompanied by abrasive and fatigue wear, caused severe material transfer and high friction. At 200 °C, oxidative wear began to

participate and adhesion was reduced. At 350 °C, oxidation became the primary mechanism, forming a continuous, self-healing oxide layer, which suppresses adhesion and maintains low friction [50-52].

This study represents an initial exploration, and certain limitations should be acknowledged. Only one series of tests was performed at each temperature due to the complexity and cost of in-situ CT wear experiments. In addition, owing to the limited spatial resolution of the current CT system, the reported porosity may not fully capture submicron-scale pores. Future work will aim to improve experimental efficiency, adopt higher-resolution CT for more accurate pore modelling and porosity quantification, extend the approach to other materials, vary loading conditions, and conduct replicate tests at key temperatures to further strengthen the statistical reliability and engineering relevance of the conclusions.

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Author contributions

Wentao Zhang: Writing – original draft, Data curation. **Hao Gong:** Writing – review & editing.

Jianhua Liu: Funding acquisition, Methodology. **Qinghua Wang:** Visualization, Software. **Yaping**

Ren: Validation.

Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Competing interests

The authors have no competing interests to declare that are relevant to the content of this article.

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