Effect of Sample Tilt on Indentation and Scratch Behavior of Single Crystal Copper

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Abstract. Inclined factors bring a lot of errors to indentation and scratch experiments. The aim of this paper is to investigate the effect of sample tilt on indentation and scratch behavior of single crystal copper. Angled indentation and scratching experiments were conducted on the flat of 5°, 10°, 15° and 30°, and spherical-conical indenter was used to study the effect. In scratching simulation with SPH method, normal and lateral forces during scratching process increased when tilt. Contrary to simulation result, when contact depth was held as a constant, the forces declined as tilt angle increased. In geometry model, the actual indentation depth was less than the theoretical contact depth and the projected area of the tilt indenter was an enclosed area like an elipse with two circular arcs. Residual impression images of indentation and scratching indicated that not only the sphere section of the tip of indenter, but also the conical surface penetrated into sample and squeezed a slope. The results showed that the size of spherical-conical indenter has significant effects on the size and shape of wear debris and could not be neglected during indentation and scratching tests with sample tilt.

Keywords: Sample tilt, SPH, indentation, scratch, spherical-conical indenter.

1. Introduction
Indentation and scratch tests are well-developed method to obtain mechanical properties of material surface with almost no damage at micro/nano scale [1]. They contain abundant information of mechanical properties such as hardness and elastic modulus. In instrumented experiments, to obtain accurate determination of the real contact area, perfect aligned perpendicular between the tip and sample surface is a key factor. However, in actual operation, the tip is not strictly perpendicular for some inevitable reasons. It is due to numerous instrumentation and sample preparation factors such as roughness of the sample, the indenter probe shaft, mechanical assembling and so on [2]. The tilt condition will lead to a measurement error and should not to be neglected in mechanical experiments.

The verticality between the indenter probe and the specimen should be accurately guaranteed in order to gain a reliable results from experiments, which also make it more meaningful to investigate the effect of the tilt sample in mechanical experiments [3]. Xiaoming Liu [4] et al. investigated the effect of ploughing friction and nanohardness dependent on the tip tilt in nano-scratch test by using molecular dynamics simulations. The results showed that tilting forward (backward) to the scratch
direction has a larger effect on the friction coefficient than the case of tilting laterally to the scratch direction. Scratch hardness was also sensitive to the tip tilting forward (backward), while it was insensitive to the tip tilting laterally.

In conventional computer simulated scratch tests can be modeled by either mesh-based continuum mechanical methods, classical molecular dynamics and meshless continuum method such as SPH [5]. M.Varga and S.Leroch [6] studied the influence of temperature and load on scratch behaviour with smooth particle hydrodynamics (SPH) method, allowing for scratch investigations up to 1000 °C. Three materials for HT usage were chosen for investigation. The three materials showed very diverging wear behaviour at the investigated temperatures.

Indentation and scratch tests were conducted with Berkovich indenter in many papers, but excluding manufacturing irregularities, spherical-conical tips are really axisymmetric and widely used in scratching experiment [7]. However, no detailed studies on the effect of spherical-conical indentation and scratching, especially with a large cone angle and radius. Actually, the tip size of spherical-conical indenter has significant effects on the size and shape of wear debris and can not be neglected during indentation and scratching tests with sample tilt. The spherical-conical tip introduces a higher complexity, but also opens the opportunity to obtaining more information from the test. In this paper, a spherical-conical indenter with a large cone angle and radius was chosen to probe the effect of tilt sample in indentation and scratch experiments.

2. Depth and Area Function

The indenter used in indentation and scratch experiments is a spherical-conical diamond indenter as shown in Fig1(a). With the aid of a three-dimensional optical microscope (DXS-500,Olympus), the tip radius of 10 μm and cone angle 90° of the indenter’s tip were confirmed. Oliver and Pharr defined the contact depth as “the vertical distance along which contact is made” and Fischer-Cripps defined it as “the distance from the bottom of the contact to the contact circle” [8]. But that’s not uniformed with the actual experiment on the condition that samples or pyramidal indenter tilted. When \( h_c \ll R \), a new modified theory model applied to sample tilt indentation was shown in Fig.1(b).

![Figure 1](image)

**Figure 1.** Geometry of the indenter examined using OLYMPAS (a) and position of the indenter perpendicular to the tilted surface (b).

As shown in Fig.1(b), \( \beta=45^\circ \), a function relationship between actual contact depth \( h_a \) and theoretical contact depth \( h_c \) was shown as:

\[
h_a = h_c \cdot \cos \theta \tag{1}
\]

For sample tilt of \( \theta^\circ \), the projected area can be written as:

\[
A_{proj} = \pi \cdot ( h_c + \sqrt{2}R - R )^2 \tag{2}
\]

When sample tilt with the angle of \( \theta \), the projected area can be written as:
\[ A_{\text{proj}} = 2 \int_{x_1}^{x_2} \sqrt{(\tan^2 \theta + 1) x^2 - 2(r - h_c) \tan \theta \cdot x - 2r \cdot h_c + h_c^2} \, dx \\
+ 2 \int_{x_1}^{x_2} \sqrt{(\tan \theta \cdot x + \sqrt{2}r - r + h_c)^2 - x^2} \, dx \]  

(3)

The model indicated the actual contact depth was reduced compared with the set value. When the indentation and scratching test were held with a constant depth of 3μm \((h_c=3\mu m)\), the sample surface was in contact with sphere and cone shape. Projection area of contact area was axisymmetric and the enclosed area with two circular arcs like an ellipse as the images in Fig.2 showed. The projected area of different angles were shown in Fig.3. When the sample tilted 30°, the axis of projected area shifting much more than previous tilt angle.

3. Scratching Simulation with SPH Method

3.1. SPH Simulation Model
A SPH simulation model was founded as Fig.4. In terms of the model founded, oxygen-free high-conductivity copper (OFHC) was chosen as the sample material and the dimension was 120μm×60μm×40μm in x, y and z directions respectively. The scratching direction was defined as x axis and the speed was set with 100m/s. The SPH particles were set with 288000 in total and the diameter of each particle was 1μm. The indenter was spherical-conical with tip radius of 10μm and cone angle 90°.

3.2. Results and Discussion
The local top view of the scratched surface for distance of 75μm were illustrated as following. Fig.5. was the topographies for different tilt angles of θ. It was visible to see from the top view, when the tilt angle generated, the grooves of scratched surface were wider than normal state. When sample tilt angle \(\theta=10^\circ\), the width of groove increased 17%. With the angle increasing, the contact area increased.
Cross-section view of spherical-conical indenter during scratching process were shown in Fig.5. In the same contact depth, it was visible to see from the section view that the chips were piled up in front of the tip and increased with the declined angles generated. Fig.7 was the force and frictional coefficient during scratching process. When the angle of $\theta$ was within 10°, both of the normal force and lateral force were unchanged. When the angle increased to 15° and 30°, the normal force and lateral force increased evidently.

3.3. Experimental Analysis

All indentation and scratch experiments were performed on a single crystal of oxygen-free pure copper sample oriented in the <100> crystallographic plane with our laboratory-built tester in Fig.8(a). The experiments of indentation and scratching process were held with 3μm depth and control curve was shown in Fig.8(b).
3.4. Indentation Test

Fig. 9 was the Scanning Electron Microscope (SEM) image of angled samples after indentation tests. An indentation conducted perpendicular to the surface \( \theta = 0^\circ \) provided a regular circular indent as Fig. 9(a). Residual impression images of indentation of samples titled with 5\(^\circ\), 10\(^\circ\), 15\(^\circ\), 30\(^\circ\) were shown in Fig. 9(b), (c), (d) and (e). In changing the mounted sample \( \theta = 5^\circ \), the residual area was nearly remain unchanged. While, when the tilt angle changes from 10\(^\circ\) to 15\(^\circ\), circular area made a change evidently. The residual indentation area was enclosed with two circular arcs like an ellipse. In Fig. 9(f), when \( \theta = 30^\circ \), these two arcs were quite different, arc a-b-c was like a circular arcs and a-d-c was like a cone shape. The tip of indenter, the sphere section, pressed into sample and formed a residual indentation impression as arc a-b-c; however, the edge of the indenter, conical surface, squeezed the sample surface and formed a-d-c.

Another reason of the transformation of residual morphologies was the sliding friction between the indenter and sample. Deformation region due to sliding friction became larger with the tilt angle increased in Fig. 9(b), (c) and (d). Indentation area in Fig. 9(e) became invisibility at the same magnification as previous. This indicated that the depth of indentation decreased sharply and plastic deformation area increased evidently. More details were shown in Fig. 9(f) with an enlarged view. Residual area indicated that edge of the indenter penetrated less than the sphere section and squeezed a slope during indentation progress.

![Figure 9. SEM images of single crystal copper](image)

![Figure 10. Load-displacement curve](image)

Normal force measured of different angles were shown in Fig. 10. The dwelling process of \( \theta = 0^\circ \) was more evident than others. The change of the slope of uploading and maximum load were apparent in load-displacement curves because of the residual indentation topography at which the depth is held constant.

3.5. Scratch Test

The scratch experiment with the constant penetration depth leaves a groove on the surface accompanied by two pile-up peaks that can be observed in Fig. 11. The morphologies of tested sample were investigated using Olympus microscope. Burrs located around the scratch groove can be also observed on the surface of the sample because of side flowing. Along the scratch direction, the morphologies of the scratch profiles were approximately symmetrical, in which the groove of residual scratching surface were in the middle of the two pile-up peaks among Fig. 11(a), (b), (c) and (d). But in Fig. 11(e), obviously observed \( d_1 < d_2 \), the main reason that caused this phenomenon was due to the existences of profile of the tilted indenter. The wider section of the groove was formed by the extrusion of the profile of the indenter during scratching test.
4. Conclusions
In the deformation process, such as indentation and scratching tests, there are numerous contact mechanical factors may influence the mechanism of material deformation and removal. One of these experimental variables was the tilt angle of samples. According to spherical-conical indenter with a large cone angle and radius, it can also make a significant size effect despite small indentation depth during tests. The spherical geometry and cone profile of the indenter made effects in the indentation and scratching process simultaneously when tested samples were tilted. For more tilt angles, both sphere and cone have an influence on mechanical response.

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