Micro-indentation hardness model of AISI1045 steel during micro-plasticity indentation experiment considering size effect

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Abstract: The analytical method is used to study micro-plasticity indentation and the increment equation is worked out by analyzing the contacted nodes between rigid indenter with material. Then the experiment under various maximum loads (50mN, 100mN, 200mN, 300mN, 400mN, 500mN and 650mN) at the constant loading speed 9.6841mN/s is carried out on MCT-W501 micro-indentation tester and the constitutive equation of AISI1045 steel is obtained by Taylor equation. The experimental result shows great indentation size effects, so the intrinsic material length is introduced to accurately simulate the micro-indentation plasticity changes. Based on ABAQUS analyzing software, the equation obtained by experiment is input to simulate the loading curve between indentation load and indentation depth considering the effect of strain gradient. The result shows that the square of hardness relative value has a linear decrease with the increase of indentation diagonal. Based on the result of simulation and calculation, the final constitutive equation is proposed and the data of modified equation is quite coordinate with experimental and calculated data with relative error less than 4.92% thus the constitutive equation is verified.

1. Introduction
Micro-indentation technology is the expansion of conventional hardness test at micro- scale (no more than sub-millimeter) [1], where the principle is to get parameters of micro-indentation hardness and elastic modulus according to loading/unloading curves of load and displacement. It becomes an important means to measure material properties and is widely used in the fields of medical devices, bio-engineering, aircraft and film material research. Material physical parameters can be calculated through various simulation methods to provide support for performance calculation [2-5]. Until now, there are many reports on micro-indentation [6-8], however, these researches are almost about the experimental analysis and its numerical calculation has not studies yet. Commonly, size effect in classical plasticity theory is not considered [9-10] and practical micro-indentation may led to significant size effect with linear decrease of micro-scale deformation [11-12], the intrinsic material length has to be considered to obtain the accurate micro-indentation hardness considering size effects. Gao and Huang [13,14] have proposed the mechanism-based strain gradient plasticity theory based on dislocation mechanism under the enlightenment of Nix et al.. Models commonly used during micro-indentation process mainly include elastic recovery model, Kick model, proportioned sample.
model, Hays-Kendall model, and Taylor dislocation model [15]. Taylor dislocation model considering the influences of statistical stored dislocation density and geometrically necessary dislocation density [16] has been inherited. Meanwhile, intrinsic material length is introduced to study the effect of strain gradient on micro-plasticity properties considering size effect. In this paper, analytical method is used to solve the micro-indentation hardness calculation with indentation depth during micro-indentation process to establish the constitutive model of AISI1045 steel. Besides, micro-indentation was simulated by finite element method with constitutive equations confirmed by related experiment. The intrinsic material length was then determined. With contrastive analysis of simulated and experimental data, the square of relative hardness value decrease linearly with the increase of indentation diagonal was finally obtained. The influencing law of conical indenter radius (half-cone angle is 70.3°) on the micro-indentation hardness during micro-indentation process, incremental equation of micro-indentation hardness was given out. Constitutive model of AISI1045 steel during micro-indentation process was revised combined with experimental and simulated data with small absolute error, which provides a strong theoretical basis to the calculation of micro-plasticity constitutive equation of power hardening materials and the prediction of micro-indentation hardness during indentation process.

2. Experimental scheme and results discussion

2.1 Experimental scheme

Material tested is AISI1045 steel, and its chemical composition (wt.%) is as: C: 0.42~0.50%, 0.17~0.37%, Mn: 0.50~0.80%, P: 0.02%, Cr: ≤0.25%, Ni: ≤0.30%, S: 0.02%, others Fe. Experiments were carried out on MCT-W501 micro-indentation tester with Berkovich indenter. Samples with dimension Φ8mm×4mm underwent mechanical and electrolytic polishing, then lapped to a mirror. The experimental maximum loads of 50mN, 100mN, 200mN, 300mN, 400mN, 500mN and 650mN were separately selected by loading/unloading with the speed of 9.6841mN/s, the load-depth curves were recorded. Besides, indentation diagonal lengths were measured by data collection software. Parameters of AISI1045 steel in uniaxial tensile and indentation tests is in Table 1.

| $E$ (GPa) | $\nu$ | $\sigma_y$ (GPa) | $n$ | $\delta$ | $F_h$ (kN) | $P_{max}$ (mN) | $h_{max}$ (nm) | $H$ (GPa) | $P_s$ (mN) | $H_s$ (GPa) |
|-----------|-------|-----------------|-----|--------|-----------|---------------|---------------|--------|---------|----------|
| 226.298   | 0.2   | 897.63          | 0.2 | 45     | 26.492    | 641.698       | 2.616         | 4.371  | 18.275  | 4.033    |

In the Table 1, $E$ represents elastic modulus, $\nu$ is Poisson ratio, $\sigma_y$ stands for yield limit, $n$ means hardening exponent, $\delta$ is elongation, $F_h$ is the maximum of tension stress, $P_{max}$ is the maximum of loading stress, $h_{max}$ is the maximum of indentation depth, $H$(GPa) is indentation hardness, $P_s$ (mN) is the stable loading stress, $H_s$ (GPa) is the stable indentation hardness. Hardening exponent value $n$ can be calculated by the uniaxial stress-strain curve.

2.2 Analysis of experimental results

Figure 1 shows the area of micro-indentation measured, loading/unloading curves under various conditions.
As in Figure 1 (a)-(b), the experimental data are repeated five times and averaged under a loading condition. The area enclosed by loading curve, unloading curve and horizontal axis represents the plastic deformation work, while the area enclosed by unloading curve, horizontal axis and the vertical ordinate of maximum load represents elastic deformation work. It is found in experiment that scale effect exists below a stable loading stress. We can see that plastic deformation work occupied a dominated proportion and elastic deformation work is small. Besides, loading curves are roughly with the same tendency thus could be simplified by Kick model.

3. Finite element analytical method of micro-indentation

Assuming that conical indenter is fully rigid and there is no friction between indenter and material [17], the load of node can be adopted approximately at normal direction. Assuming that conical indenter is rigid and the contact surface with indenter is under friction-free state, the schematic diagrams of load state and displacement state brought by conical indenter pressed are in Figure 2.

As in Figure 2, the displacement boundary on contact sphere and the contact boundary of interfacial nodes at the top of indenter must keep the sliding condition at spherical part. Moreover, the displacement boundary on conical surface and the contact boundary of interfacial nodes at conical part of indenter must keep the sliding condition too. The displacement boundary of node $i$ at spherical part and the incremental displacement of node $j$ at conical part under the incremental calculation step $n$ during micro-indentation process can be described as:

$$\frac{r_i}{\sqrt{a^2 - r_i^2}} \cdot \Delta u_i + \Delta v_i = -\delta_n$$

$$\Delta u_j \cdot \tan \theta + \Delta v_j = -\delta_n$$

Where $r$ and $z$ are cylindrical coordinates, $(r_i, z_i)$ stands for coordinate of node $i$; $\Delta u_i$ and $\Delta v_i$ are incremental node displacements in $r$ and $z$ directions, $a$ is the hemispherical radius of indenter, $\delta_n$ is incremental displacement caused by indenter with material being pressed, $\theta$ is $70.3^\circ$ for Berkovich
indentor. The nodal force at node \( N \) can be divided into \( F_{Nr} \) and \( F_{Nz} \) at directions of \( r \) and \( z \), so the incremental loads at directions of \( r \) and \( z \) can be separately expressed as \( \Delta F_{Nr} \) and \( \Delta F_{Nz} \). The incremental loads of spherical contact node \( i \) and conical contact node \( j \) are:
\[
\Delta F_r \cdot \cos \alpha + \Delta F_z \cdot \cos \alpha = 0 \tag{3}
\]
\[
\Delta F_r + \Delta F_z \cdot \tan \theta = 0 \tag{4}
\]
The modified incremental load \( \Delta F_e \) must be added in the direction of radius to keep the deformation load along the direction of normal at contact surface required, the expression of \( \Delta F_e \) can be described as follows:
\[
\Delta F_e = -F_{r} - F_{z} \cdot \tan \alpha = 0 \tag{5}
\]
Where exists the equation: \( \alpha = \arctan (r_i / \sqrt{a^2 - r_i^2}) \).

For the regular resolution of contacted fine grid section, the incremental finite element equation is as:
\[
[K] \{ \Delta d \} = \{ \Delta F \} \tag{6}
\]
Where \( [K] \) is total stiffness matrix, \( \Delta d \) and \( \Delta F \) respectively represent incremental displacement and load. Insert load boundary conditions and displacement boundary conditions of corresponding contact surface nodes into equation (6), equations of (1), (3) and (5) is inserted for the ball contact surface node \( i \) while the proportionally conical surface contact node \( j \) with equations of (2) and (4) inserted, so the Micro-indentation hardness obtained ultimately can be resolved by the simultaneous equations described as follows:
\[
\begin{bmatrix}
k_{21,1} + k_{21,1} \cdot \tan \alpha & \cdots & k_{21,1,1} + k_{21,1} \cdot \tan \alpha & k_{21,2,2} + k_{21,2} \cdot \tan \alpha \\
0 & \cdots & -r_i / \sqrt{a^2 - r_i^2} & 1 \\
k_{22,1} + k_{22,1} \cdot \tan \theta & \cdots & k_{22,1,1} + k_{22,1} \cdot \tan \theta & k_{22,2,2} + k_{22,2} \cdot \tan \theta \\
0 & \cdots & -1 / \tan \theta & 1
\end{bmatrix}
\begin{bmatrix}
\Delta u_i \\
\Delta v_i \\
\Delta u_j \\
\Delta v_j
\end{bmatrix}
= \begin{bmatrix}
-F_{r} - F_{z} \cdot \tan \alpha \\
-\delta_n \\
0 \\
-\delta_n
\end{bmatrix} \tag{7}
\]
Solving equations of the contact nodes in equation (7) as well as non-contact nodes treated by conventional computing method, displacement distribution during unloading from \( P_{\text{max}} \) can be finally obtained.

Micro-plastic constitutive equations considering size effect has been studied in references [18-20]. Assuming that pile-up is negligible, the amount of sink-in, indentation depth \( \delta \) at any time during loading is as:
\[
\delta = h_c + h_s = h_c + \varepsilon \cdot \frac{P_{\text{max}}}{S} \tag{8}
\]
Where \( h_c \) represents the indentation depth (nm), \( \varepsilon \) is a constant depended on indenter shape with its value of 0.75 for Berkovich indenter, \( P_{\text{max}} \) is the maximum load rate, \( S \) is the slope of unloading curve, \( h_s \) is the displacement (nm) over the initial surface nearly the section between indenter and tested sample.

4 Numerical simulation of micro-plastic constitutive model

4.1 Meshing and Modeling
Parameters of \( E, \nu, n \) are set in Table 1, strengthening coefficient is 0.468. Meshing diagram is in Figure 3.
The six-node linear elements and axisymmetric model are adopted. Berkovich indenter is assumed to be rigid and the deformation is subjected to the Mises yield criterion and isotropic hardening criterion. As the material belongs to power-hardening type, one sixth of model is adopted in simulation with fraction coefficients of 0 and 0.3 between indenter and material. To save computing resources, transitional grid division method is adopted, with fine grid used nearby contact deformation zone of indenter and sparse grid away from deformation zone, with 7600 fine grid in contact zone of 0.3μm×0.3μm. Besides, the load is directly pressed on material without using contact mode during loading. Five degrees of freedom are limited except z-axis, the bottom plane is fixed, and then material model in the subroutine UMAT of ABAQUS is adopted.

4.2 Simulation results and analysis
Micro-indentation constitutive model is used in the simulation by FEA method. Indentation morphologies and stress distribution diagrams under various friction coefficients with depth of 30nm are in Figure 4.

![Figure 4 Stress distribution diagrams under various friction coefficients](image)

As is shown in Figure 4, the maximum residual stress after unloading on the ideal contact condition distributes just below the indenter, while the maximum residual stress after unloading on the friction condition distributes around the top of indenter and the residual stress below the indenter is not too large.

5. Data contrastive analysis of simulation, calculation and experiment
The simulation accuracy can be quantitatively described by averaged absolute relative error (AARE) [21]:

\[
AARE(\%) = \frac{1}{N} \sum_{i=1}^{N} \left| \frac{E_i - P_i}{E_i} \right| \times 100
\]  

(9)

Where, \( E_i \) represents experimental values, \( P_i \) is simulation values, \( N \) is the selected points number. Comparison of simulated, calculated and experimental data and hardness model is shown in Figure 5.
Figure 5 Comparison of simulated, calculated and experimental data and hardness model

As in Figure 5 (a), loading curve measured by micro-indentation experiment based on Kick model is fitted. The analysis revealed that the model is reasonable for the loading curve of indentation test. The intrinsic material length of AISI1045 steel was ultimately determined as 5.06μm. The simulated Kick model after optimization is expressed as $P=25.186h^{1.692}$. So the constitutive model established during micro-plasticity deformation of AISI1045 steel is precise, and its averaged AARE is about 4.92%. Relationship between $(H/H_0)^2$ and $d$ is shown in Figure 5(b). From the data analysis, the relation between reciprocal of indentation diagonal length $d$ and square of relative micro-indentation hardness value $(H/H_0)^2$ is:

$$
\left( \frac{H}{H_0} \right)^2 = 0.9852 + \frac{2.2857}{d}
$$

Where $H$ is indentation hardness (GPa), $H_0$ is indentation hardness without considering strain gradient effect, $d$ is the reciprocal of indentation diagonal length.

6. Conclusions

Through contrastive analysis of simulated curve and experimental curve, a function of square error sum during loading process was proposed and optimized based on simulated and experimental data. Micro-indentation was theoretical analyzed and micro-indentation hardness was obtained. Results are as:

1. Micro-indentation has been simulated on ABAQUS and indentation topography maps under various friction coefficients have been finally obtained, which provides basis of theoretical guidance practical analysis.

2. Kick model was adopted to fit the loading curve of AISI1045 steel obtained by micro-indentation experiment, and the accuracy of Kick model was analyzed and verified by sample variance. So the model is thought to have a better description of mechanical behavior during micro-indentation process.

3. By analysis of analytical solution and experimental data, micro-indentation hardness equation of AISI1045 steel during micro-indentation process has been given out.

Moreover, the square of relative hardness value linearly decreases with the increase of indentation diagonal, and the hardness equation was finally verified.

Acknowledgements

This work is supported by Shenzhen Basic Research Project (No.JCYJ20160518112621719). Thanks to the support of Xiangjiale technology (Shenzhen) co., LTD and southern university of science and technology.
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