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Materials

Determination of plastic equation of state by indentation method on 304 stainless steel

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Abstract: An indentation method for determining the plastic mechanical equation of state (PES) was studied. The constant loading rate and constant loading rate/load indentation tests were carried out. The method for determining the work-hardening coefficient and the strain rate sensitivity coefficient of PES were discussed in detail. 304 stainless steel hot-treated at 1100°C was used to verify the method. The work-hardening coefficient and strain rate sensitivity coefficient of 304 stainless steel were respectively determined as 0.30 and 0.015. These values are very close to those achieved by tensile tests. From the establishment of the PES of 304 stainless steel it is shown that the PES obtained by the indentation method is easier than that by the tensile test.

Key words: plastic equation of state; 304 stainless steel; work hardening coefficient; strain rate sensitivity coefficient

1 Determining the strain-hardening coefficient of 304 stainless steel by indentation experiment

1.1 Experiment

The experiment was carried out with a Nano Indenter II Mechanical Properties Microprobe (MPM) [1-3] with a pyramid-shaped tip (Berkovich tip) at 20±1°C. The constant rate of loading 7, 23, and 62 mN/s were respectively used to the maximum load of 700 mN then holding the load for 10 min. The 304 stainless steel specimen was treated at 1100°C in vacuum solid solution. The sample surface was electropolished. In order to eliminate the influences of environment, surface effect, power fluctuation, and the instrument itself, the experiment was repeated for 10 times for each loading rate and a total displacement over 1000 nm. Only reasonable results were used in the following discussion. **Figure 1** is the relationship between the load and time during the experiment.

1.2 Results and discussion

Figure 2 shows the relationship between the strain rate and the displacement at different loading rates. The strain rate ($\dot{\varepsilon}$)is obtained by strain *(ε)* differentiation with respect to time (t) during the loading and holding. During loading, the strain rate decreases continuously as the displacement increases. During holding, the strain rate decreases more quickly. The strain rate also decreases as the loading rate decreases.

Figure 3 shows the relationship between the hardness and the displacement at different loading rates during loading and holding. This hardness can be approximately considered as stress: the instant load divided by the instant contact area which can be calculated by an area function of the tip and contact depth.

$$H = \frac{P}{A} \tag{1}$$

where H is the hardness, P the load, and A the area function of the tip calculated from the following formula [4-5]:

$$A = ah_c^2 + bh_c + ch_c^{1/2} + dh_c^{1/4} + \cdots \tag{2}$$

where a, b, c, d ... are calibration constants, h_c is the contact depth which at any time during loading,

$$h_c = h_t - h_s \tag{3}$$

where h_t is the total displacement and h_s the displacement of the surface at the perimeter of the contact. h_t and h_s can be determined from the load-displacement data. The h_s is given by

$$h_s = \varepsilon_0 P_{max}/S \tag{4}$$

where ε_0 is the geometric constant, for the conical tip ε_0=0.72, S is the stiffness.

During the indentation, the hardness decreases considerably with the displacement increasing. The strain rate also decreases as the displacement increases. During loading, when the strain rate slowly changes

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the change of hardness is slow. But when the loading stops, the strain rate decreases rapidly and the hardness changes considerably.

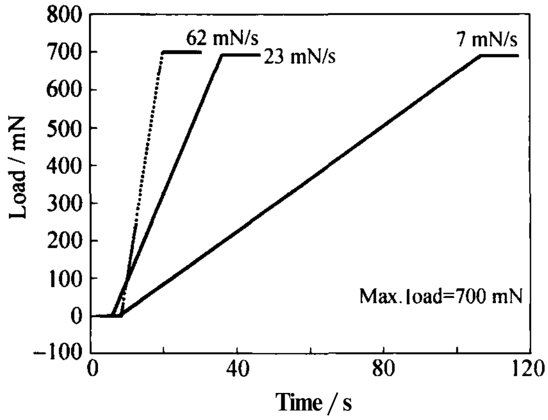

Figure 1 Load-time histories for the constant loading rate indentation experiment

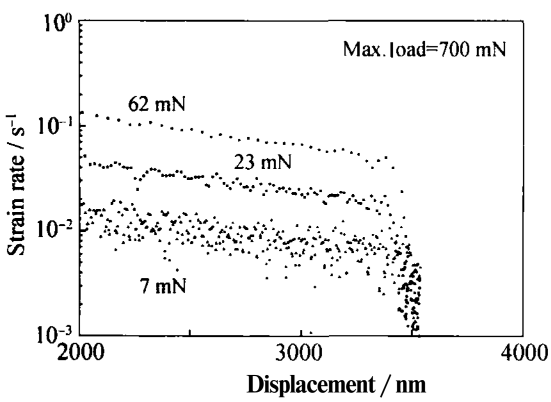

Figure 2 Indentation strain rates of 304 stainless steel achieved by using the load-time histories in figure 1.

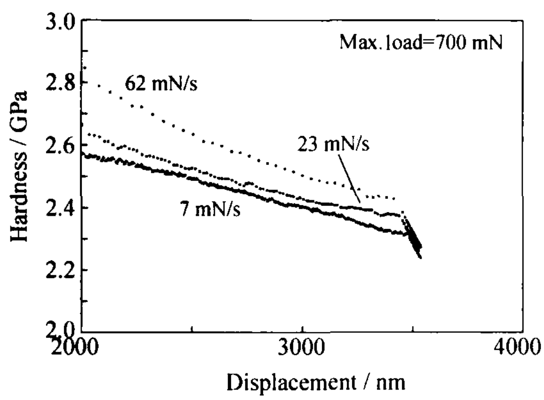

Figure 3 Hardness *versus* displacement of 304 stainless steel under the condition of constant loading rate.

Under the indentation condition (Berkovich tip), the load P, the compressive stress H and the displacement or indent depth h satisfy the following equation [2, 4]:

$$P = Ch^2H \tag{5}$$

where C is a constant related with the shape of indentation. We define the strain $d\varepsilon=dh/h=d(\lg h)$, and the strain rate $\dot{\varepsilon}=\mathrm{d}\varepsilon/\mathrm{d}t$. The hardening state of a material with a certain microstructure is calculated using the maximum indent depth h_{max} and maximum load P_{max}.

In the course of any indentation deformation at constant temperature, similar to the uniaxial test, the applied parameter load P, compressive stress H, indent depth h, indent strain ε and strain rate $\dot{\varepsilon}$ must be connected through equation (5) and the following differential relationship:

$$\mathrm{d}(\lg H)=\frac{\partial(\lg H)}{\partial\varepsilon}\Big|_{\dot{\varepsilon},T}\mathrm{d}\varepsilon+\frac{\partial(\lg H)}{\partial(\lg\dot{\varepsilon})}\Big|_{\varepsilon,T}\mathrm{d}(\lg\dot{\varepsilon})=\gamma\mathrm{d}(\lg h)+m\mathrm{d}(\lg\dot{\varepsilon}) \tag{6}$$

where m is a strain rate sensitivity coefficient:

$$m=\frac{\partial(\lg H)}{\partial(\lg\dot{\varepsilon})}\Big|_{\varepsilon,T} \tag{7}$$

γ is a nominal work-hardening coefficient, it is characterized by γ':

$$\gamma'=\frac{\partial(\lg H)}{\partial\varepsilon}\Big|_{\dot{\varepsilon},T}=\frac{\partial(\lg H)}{\partial(\lg h)}\Big|_{\dot{\varepsilon},T} \tag{8}$$

When the temperature is constant, m and γ' uniquely depend on the current values of stress and plastic strain rate [6].

$$\frac{\mathrm{d}(\lg P)}{\mathrm{d}\varepsilon}\Big|_{\dot{P},T}=\frac{\mathrm{d}(\lg P)}{\mathrm{d}(\lg h)}\Big|_{\dot{P},T}=\frac{\mathrm{d}[\lg(Ch^2H)]}{\mathrm{d}(\lg h)}=\frac{\mathrm{d}(\lg C+\lg H+2\lg h)}{\mathrm{d}(\lg h)}=2+\frac{\mathrm{d}(\lg H)}{\mathrm{d}(\lg h)}\Big|_{\dot{P},T} \tag{9}$$

where $\frac{\mathrm{d}(\lg P)}{\mathrm{d}\varepsilon}\Big|_{\dot{P},T}$ is the strain-hardening coefficient for a certain loading rate, $\frac{\partial(\lg H)}{\partial(\lg h)}\Big|_{\dot{\varepsilon},T}$ the strain hardening coefficient of a certain strain rate. $\frac{\mathrm{d}(\lg P)}{\mathrm{d}\varepsilon}\Big|_{\dot{P},T}$, $\frac{\mathrm{d}(\lg H)}{\mathrm{d}(\lg h)}\Big|_{\dot{P},T}$ and $\frac{\partial(\lg H)}{\partial(\lg h)}\Big|_{\dot{\varepsilon},T}$ can be obtained from the constant loading rate *(dP/dt)* and the constant strain rate ($\dot{\varepsilon}$) indentation experiment [6]. **Table 1** shows the values of each coefficient correspondent to the loading rate of 7, 23 and 62 mN/s respectively.

From table 1, it can be concluded that at a certain loading rate, the indentation strain rate decreases as the displacement increases. Because of the strain hardening effect, the displacement is smaller than that without strain-hardening. Furthermore, the deformation of material decreases gradually as the loading increases. Thus, the value of $\frac{\mathrm{d}(\lg H)}{\mathrm{d}(\lg h)}$ is negative. And the smaller the strain-hardening effect, the smaller the absolute value of this negative value. At a constant rate of loading *(dP/dt)*, although the strain rate ($\dot{\varepsilon}$) decreases as the displacement increases the absolute value of logH decreases at the same time. These two values change at the same order of magnitude. Therefore, it is reasonable that the value of $\frac{d(\lg H)}{d(\lg h)}|_{\dot{P},T}$ changes in the range of -0.16 to -0.22. For the same reason, from formula (6) it can be achieved that at room temperature the range of the loading rate *(dP/dt)* from 7 to 62 mN/s corresponds with the range of the strain rate of 0.0075 to 0.079 s^{-1}. For 304 stainless steel, the change of the loading rate is not sensitive in this range. Therefore, the change of the indentation strain-hardening rate is small. The average strain-hardening coefficient is:

$$\gamma' = \frac{d(\lg H)}{d(\lg h)}|_{\dot{\varepsilon},T} = 0.30 \pm 0.03 .$$

This indentation strain-hardening coefficient agrees with the strain-hardening coefficient from the tensile test in the range of 0.3-0.5 for austenite stainless steel [7].

Table 1 Indentation strain hardening coefficient

h/nm	*(dP/dt)* / $(mN \cdot s^{-1})$	ε / s^{-1}	$\frac{d(\lg P)}{d\varepsilon}|_{\dot{P},T}$	$\frac{d(\lg H)}{d(\lg h)}|_{\dot{P},T}$	$\frac{d(\lg H)}{d(\lg h)}|_{\dot{\varepsilon},T}$
1190-1796	62	0.07878	1.78459	-0.21541	0.33
	23	0.02931	1.79359	-0.20641	0.32
	7	0.00904	1.83241	-0.17759	0.27
2497-3114	62	0.06458	1.82131	-0.17869	0.29
	23	0.02433	1.81134	-0.19966	0.30
	7	0.00754	1.84218	-0.16782	0.26

2 Determining of the strain rate sensitivity coefficient of 304 stainless steel by indentation experiment

2.1 Experiment

The indentation experiment was performed under constant loading rate/load ($\frac{dP}{dt}\frac{1}{P}$). The maximum load is 700 mN, after reaching the maximum load it was held for 10 min. The loading rates/load used are 0.15, 0.05 and 0.005 s^{-1} respectively. Under these experiment conditions, the constant strain hardening rate ($\frac{d(\lg H)}{d(\lg h)}$) can be obtained by using the constant $\frac{dP}{dt}\frac{1}{P}$ for the material which has a unique strain-hardening state. **Figure 4** shows the relation between the load and the time for 304 stainless steel at different values of $\frac{dP}{dt}\frac{1}{P}$.

2.2 Results and discussion

Figure 5 shows the relation between the indentation strain rates and the displacement for the constant loading rate/load ($\frac{dP}{dt}\frac{1}{P}$) experiment. The indentation strain rates are achieved by displacement differentiation with respect to time then divided by the displacement ($\dot{\varepsilon} = \frac{dh}{dt}\frac{1}{h}$). From figure 5 it is known that during loading the indentation strain rate is almost constant. But at the end of loading the indentation strain rate decreases rapidly.

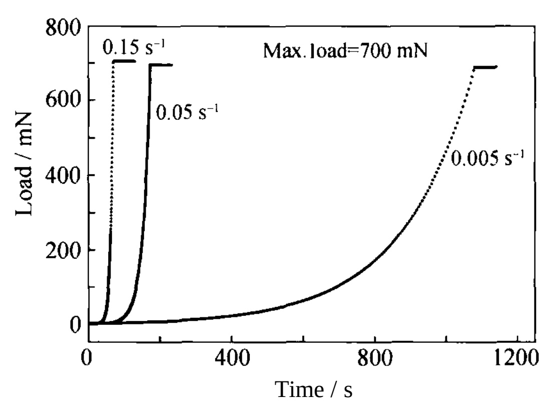

Figure 4 Load-time histories for the constant loading rate/load experiment.

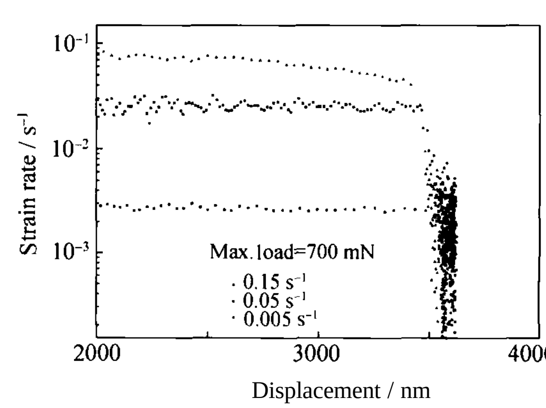

Figure 5 Constant indentation strain rates achieved by using the load-time histories in figure 4.

Figure 6 is a plot of hardness *versus* displacement showing the constant hardness as a function of depth in the constant $\frac{dP}{dt}\frac{1}{P}$ experiment. The hardness is achieved by the instant load divided by the instant contact area. During constant $\frac{dP}{dt}\frac{1}{P}$ loading, the hardness does not change. But during holding, the hardness decreases as the indentation depth increases. It is worth notice that the hardness decreases as $\frac{dP}{dt}\frac{1}{P}$ decreases. At different loading rates ($\frac{dP}{dt}\frac{1}{P}$ equals to 0.15, 0.05 and 0.005 s^{-1}), different hardness H and indentation strain rate $\dot{\varepsilon}$ can be obtained. By using this relationship and fitting the data, the strain sensitivity coefficient is achieved by the following equation (see figure 7):

$$m=\frac{d(\lg H)}{d(\lg \dot{\varepsilon})}=0.015\pm 0.009.$$

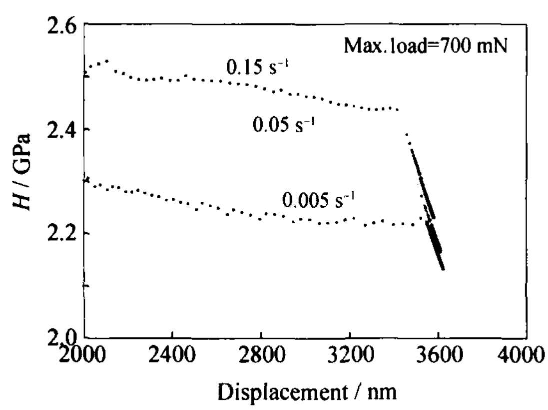

Figure 6 Plot of hardness *versus* displacement under constant *dP/dt*.

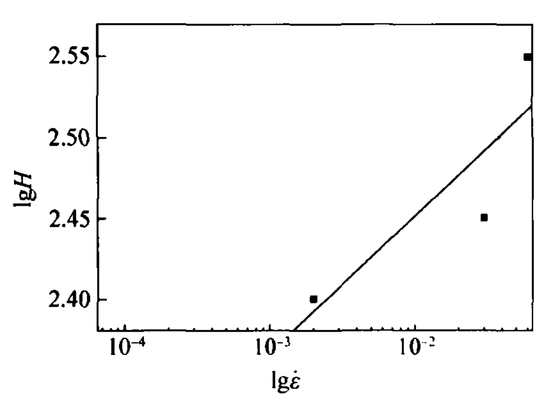

Figure 7 Fitted curve of average indentation strain rate *versus* average hardness from the data obtained during the constant d*P/dt* experiment

The reliability is 87%. The strain sensitivity coefficient m of indentation is close to the strain sensitivity coefficient achieved by the tensile test (0.014) [8].

Therefore, we can conclude that using Nano Indenter II Mechanical Properties Microprobe (MPM), the strain sensitivity coefficient can be obtained more easily and more quickly than by the tensile test.

The plastic equation of state could be measured by nano indentation approach. For 304 stainless steel the plastic equation of state is

$$D(\lg H)=\frac{\partial(\lg H)}{\partial \varepsilon}\Big|_{\dot{\varepsilon},T} d\varepsilon+\frac{\partial(\lg H)}{\partial(\lg \dot{\varepsilon})}\Big|_{\varepsilon,T} d\lg\dot{\varepsilon}=$$
$$\gamma d(\lg h)+m d(\lg \dot{\varepsilon})=0.30d(\lg h)+0.015d(\lg \dot{\varepsilon}).$$

3 Conclusions

Using Nano Indenter II Mechanical Properties Microprobe (MPM), the internal characteristic of plastic state of materials can be determined. An effective way of using the plastic equation of state to study an alloy and its organization was established. Furthermore, the deformation behavior of materials under a certain deformation trace can be deduced. The points described in this paper are summarized as follows:

(1) The strain-hardening coefficient can be determined by the constant loading rate indentation experiment.

(2) The strain rate sensitivity coefficient can be determined by the constant loading rate/load indentation experiment.

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