IOP Conf. Series: Materials Science and Engineering **394** (2018) 022066 doi:10.1088/1757-899X/394/2/022066

Tudy on the Scale Effect of Nan scale Indentation on Yield Strength and Modulus of Elasticity

Chao Wang[a], Xinhai Yu[b] and Jian Zhang[c]

School of ECSUT University, Shanghai 200000, China

[a]2269248712@qq.com, [b]yxhh@ecust.edu.cn, [c]364772239@qq.com

Abstract. In recent decades, many micromechanics experiments have proved that the mechanical behavior of metal materials at the scale of micron or submicron scale is quite different from that of materials under macroscopic scale (above millimeter order). When the characteristic length of the no uniform plastic deformation reaches the magnitude of micrometer or submicron, the material will show a very strong scale effect. In this paper, we mainly study the scale effect of the two parameters, the yield strength and the modulus of elasticity, which are very important in the mechanical constitutive equation. The scale effect of yield strength can be studied by using the H-P formula. With the further research, it is found that the modulus of elasticity have obvious scale effect.

1. Introduction

The experimental investigation found that when the material is at the micron scale, it has strong scale effect, for example, the twisting of thin copper wire and the bending of thin metal beam, the thinner metal wire and the thinner metal beam , the more serious the plastic hardening is [1]**.** During the analysis of plastic properties of metals, many materials can get their yield strength and other plastic properties through tensile tests, but this means macroscopical mechanical properties. At the micro level, it is not applicable, because the decreasing of scale and the enhancement of microcrystalline anisotropy will also increase the influence factors of mechanical constitutive factors, the scale effect is obvious [2]. The relationship between the stress and strain of each grain is different, and the macroscopic mechanical constitutive analysis is not very useful in the analysis of the micro scale. But now, with the maturity of nanotechnology, such as micro pillar compression test stress-strain curves have been studies so much by the analysis of plastic deformation by using this method we can synapse in micro scale of the load displacement curves processed by stress-strain curve, yield strength and elastic modulus can be calculated [3], scientists at home and abroad have made a lot of research, such as Dorner and Nix [4] and [5], Oliver Pharr, Cheng and Cheng [6].

It is well known that the main parameters in the constitutive relationship of the classical elastoplastic mechanics are the yield strength and the modulus of elasticity. The scale effect of yield strength has been studied in a large amount, and the relationship fitting of scale and yield strength can be completed by the famous Hall-Petch formula [7]. But for the elastic modulus, by virtue of our traditional understanding on the past, the elastic modulus of elasticity index is used to characterize the material, which is an intrinsic property of material, and chemical bond, there will be no scale effect similar to the yield strength, and many scientists have proved this conclusion [8]. By Jager [9] proposed that the surface cannot change the instrument testing elastic modulus stiffness modulus is an attribute of the index of material; while other scholars also believe that the elastic deformation and plastic deformation, the surface cannot have a significant impact on the elastic modulus of the test. However, I found through experiments that elastic modulus has obvious scale effect.

With the rapid development of nanotechnology, there are great breakthroughs in the research methods of mechanical properties of micro materials. At present, the most common way to study micromechanical properties is Nan indentation technology, so that we can complete mechanical analysis of yield strength and elastic modulus of micro pillars at different scales.

2. Experimental process

The material used in this paper is 304 stainless steel, which is common in daily life. Because of the high requirement for the clarity of grain boundaries, it has been treated by solid solution to obtain single austenite. After electrolytic etching, a clear grain boundary is obtained. Then the use of EBSD (electron backscatter diffraction) technique obtained grain orientation distribution of the sample surface, because the lattice parameters of the plane and the angle between the axis and the corresponding crystal type and crystal grain orientation, based on these data we can get different colors corresponding to the. 1, 2, 3, 4, four grains of the same grain orientation are selected to be marked for subsequent micro pillar processing, As in Figure 1.

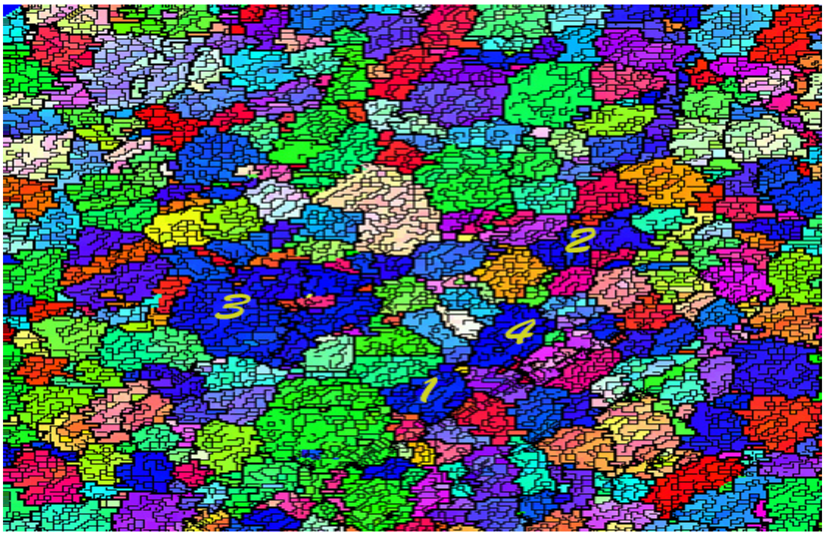

Figure 1. The distribution of crystal orientation

The method for processing the surface micro morphology are often used FIB (Focused Ion focused ion beam, beam), the principle is the liquid metal (most FIB use Ga) ion beam ion source produced by ion gun acceleration, focusing on the surface of the sample after irradiation to produce two electronic signal. The electronic image function and SEM (scanning electron microscope), or by means of the high current ion beam stripping of surface atoms, to complete the micro and nano scale surface processing. Therefore, the micro pillar can be produced by FIB (focused on cutting beam) in several different regions on the surface of the sample. Because the height and width of the micro pillar is also affected by the plastic deformation of the micro pillar, all the micro pillar are able to produce a uniform height to width ratio. At the same time, in order to study the plastic deformation under different scales, 4 sizes of micro pillar under different grain orientations were designed, which are shown in Table 1 below, and 2 is the schematic diagram.

Table 1. Micro pillar size

Scale d (um)	1	2	5	10
Diameter of the top a (um)	1	2	5	10
Diameter of the bottom b(um)	1.3	2.4	6	12
Height h (um)	3	6	15	30

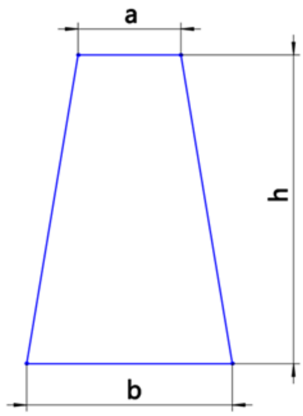

Figure 2. The sketch map of micro pillar size

Finally, the micro pillar compression experiment, because the experiment need to load and displacement with high resolution, and then reflect the deformation and plastic deformation degree of the curve of elastic material, so the method of measuring the mechanical properties of the materials used in the macro here is not applicable. So take a present the nan indentation technique for the micro determination of the mechanical properties which is very accurate, the technology is controlled by the computer load continuous change and online monitoring depth due to the applied pressure, displacement sensor monitoring and resolution is better than 111rn, so can obtain nanometer small (0.1 ~ l00nm the pressure) deep, which is suitable for thin layer materials of mechanical properties test and for natural micro pillar compression experiment. The micro pillar obtained after the experiment is shown in Figure 3 below.

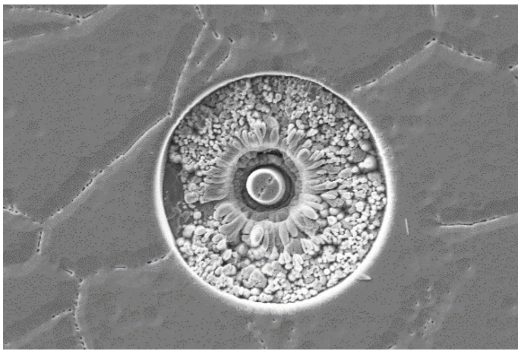

Figure 3. Micro pillar

3. correlation theory

The theory to describe the relationship between yield strength and scale are the main H-P formula, the formula by NJ-Petch of the University of Leeds in England [10] in 1946-1949 by measuring cleavage strength under different low temperature with different grain sizes and the precision improvement based on Hall formula, we will be able to study the relationship between the yield strength with the help of the formula and scale. The specific formulas are as follows:

$$\sigma_y = \sigma_0 + k d^{-1/2} \tag{1}$$

σ_y is the yield strength that represents the 0.2% strain of the material, σ_0 is the lattice friction resistance resulting from the movement of a single dislocation indicates a parameter related to the properties of the material and the size of the grain. D means the average diameter of the grain.

Based on our traditional recognition, elastic modulus is an index that represents the properties of materials, and will not change with the change of scale. Many scientists have proved this. But Sun changing who is the professor in Singapore Nanyang Technology University as the group leader in 2001 proposed structure between the surface layer and the inner layer is different between surface atoms, the bond length is short, resulting in larger bond energy, causing the surface strength of relatively large [11]. Moreover, Bao [12] equals to 2004 from the energy point of view. By introducing a parameter called Rs recovery resistance, we find the bridge between scale and modulus, and get the relation between the elastic modulus and the 4 square. Therefore, we can analyse the modulus of elasticity based on the experimental data.

4. Experimental results and discussions

4.1. The phenomenon of scale effect

The Nan indentation test is 20um cylindrical pressure head because of the smaller size of the micro pillar. Due to constant force loading, it is easy to cause errors in loading mode. So this experiment uses 10nm/s's constant displacement loading, and the loading depth is no more than 1/3 of the micro pillar height, so that it prevents excessive deformation of micro pillar fracture. Figure 4 below is the stress-strain curve of different sizes of grain under the same grain orientation.

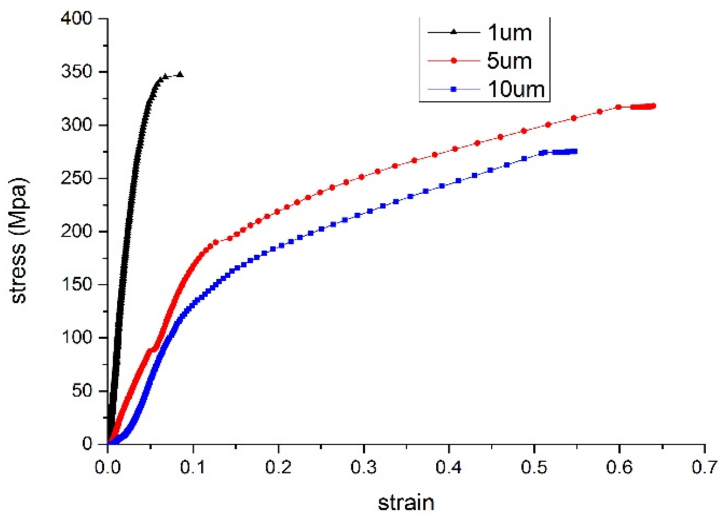

Figure 4. The strain curve in different dimension for the same grain

From the diagram, it is not difficult to find that with the continuous reduction of the scale, the greater the stress of the micro pillar, the larger the scale effect is, with the same strain. This is because the dislocation in the grain is slipping along with the compression process. The smaller the size of the micro pillar, the smaller the space where the dislocation can slip, which leads to the increase of the flow resistance, resulting in the phenomenon of "smaller and harder". And from the above figure we can clearly find that the material has a more obvious scale effect on the yield strength and modulus of elasticity at the micro scale.

4.2. The scal effect of yield strength

In order to compare the yield strength under different grain sizes, the stress value of plastic strain is 0.2%, and the yield stress of the grain scale is obtained according to the stress strain curve. The yield strength of each scale is as follows:

Table 2. The yield strength of different size

Scale (um)	1	5	10
Yield strength (mpa)	175.12	36.23	8.14

Then, according to the relationship between the yield strength and the two sides of the scale in the H-P formula, the yield strength under different scales is synthesized into a straight line.

$$\sigma_y = -73.72 + 247.23\, d^{-1/2} \quad (2)$$

From Figure 5 below, we can see that there is a clear linear relationship between the reciprocal of the two squares of the fitting yield strength and the scale.

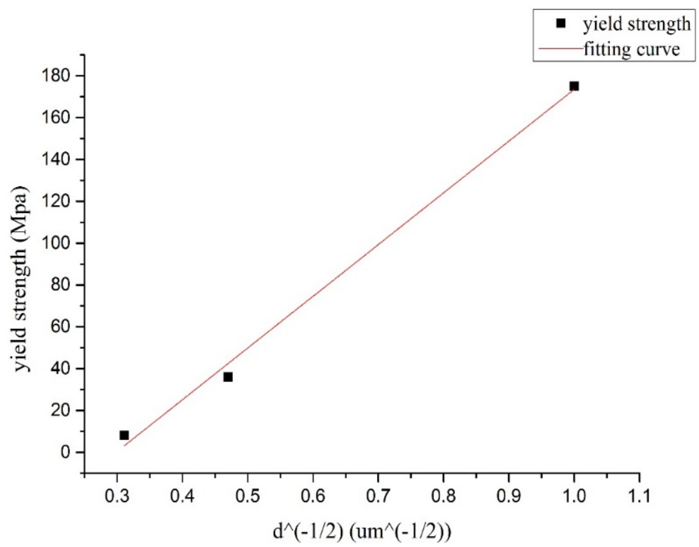

Figure 5. Fitting curve of yield strength

4.3. The scale effect of modulus of elasticity

Similarly, in order to study the scale effect of elastic modulus, we get the slope of straight line, namely the elastic modulus of each scale by fitting the elastic phase of stress-strain curve, and get the elastic modulus of each scale as shown in Table 1.

Table 3. The elasticity modulus of different size

Size (um)	1	5	10
Elastic modulus (Mpa)	9467	2157	622

According to the relationship between the modulus of elasticity and the scale of the four times, the relation formula is obtained:

$$E = -11182.01 + 20553.1\, d^{-1/4} \quad (3)$$

The fitting results are like Figure 6. From the figure we can see that there is a clear linear relationship between the modulus of elasticity and the reciprocal of the four square of the scale:

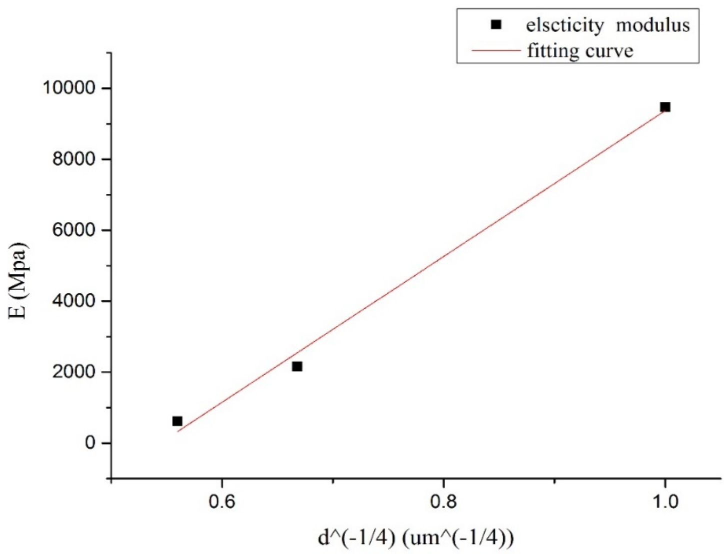

Figure 6. Fitting curve of elasticity modulus

4.4. Experimental demonstration

Although the micro pillar experimental curve fitting are good but still need to add a scale to prove, because the scale is too large the scale effect may be weakened, so adding test using the same aspect ratio, diameter of micro pillar 2um, also micro pillar compression experiment and its stress-strain curve. At the same time, the yield strength and the elastic modulus of the fitting formula of 2um micro pillar yield strength and elastic modulus were 101.10Mpa and 6101.01Mpa, and 2um micro pillar experiments yield strength and elastic modulus were 117.26Mpa and 5863.46 Mpa.it is very close to the theoretical that is further confirmed the correctness of the method.

5. The result of experiment

(1) With the help of FIB, EBSD, the scale effect of metal materials, advanced research means of Nan indentation experiments were carried out, and the experimental results of different scales, verify the elastic modulus and yield strength as the scale decreases, the growing phenomenon

(2) The relationship between yield strength and elastic modulus and scale is studied in this paper. Combining experimental and theoretical methods, we can deduce another scale's yield strength and elastic modulus according to the yield strength and modulus of elasticity of existing scale.

References

[1] Liu Yang, Chen Ding fang. Measurement of material mechanical properties using nanoindentation and finite element simulation [J].The Research Institute of Intelligent Manufacture and Control, Wuhan, 2003, 05: 690 - 693.

[2] Li Ming, Wen Shi Zhu. Theoretical methods on nanoindentation [J]. Chinese Journal of Mechanical Engineering, 2003, 03: 142 - 145.

[3] Wei Y. and Hutchinson J.W. Hardness trends in micron scale indentation. J. Mech.Phys.Solids, 2003, 51: 2037 ~ 205.

[4] M. F. Doerner, W. D. Nix. A method for interpreting the data from depth-sensing indentation instruments [J]. Journal of Materials Research, 1986, 1: 601 – 609.

[5] W. C. Oliver, G. M. Pharr. Improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments [J]. Journal of Materials

IOP Conf. Series: Materials Science and Engineering **394** (2018) 022066 doi:10.1088/1757-899X/394/2/022066

Research, 1992, 7: 1564 - 1583.

[6] Y. T. Cheng, C. M. Cheng. Relationships between hardness, elastic modulus, and the work of indentation [J]. Applied Physics Letters, 1998, 73: 614 - 616.

[7] Fleck, N. A., Muller, G. M., Ashby, M.F., et al. Strain gradient plasticity: theory and experiment. Acta Mater, 1994, 42:475 ~ 487.

[8] Zong Z, Soboyejo W. Indentation size effects in face centered cubic single crystal thin films. Mater Sci Eng, 2005, A404: 281 ~ 290.

[9] I. L. Jager. Surface free energy−a possible source of error in Nano hardness. [J]. Surface Science, 2004, 565: 173 - 179.

[10] Saada G. Mater Sci Eng, 2005; A400 - 401: 146.

[11] C. Q. Sun, B. K. Tay, S. P. Lau, X. W. Sun, X. T. Zeng, S. Li, H. L. Bai, H. Liu, Z. H. Liu, E. Y. Jiang. Bond contraction and lone pair interaction at nitride surfaces [J]. Journal of Applied Physics, 2001, 90: 2615 - 2617.

[12] Y. W. Bao, W. Wang, Y. C. Zhou. Investigation of the relationship between elastic modulus and hardness based on depth-sensing indentation measurements [J]. Acta Materialia, 2004, 52: 5397 - 5404.