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Indentation size effect of germanium single crystal with different crystal orientations

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Abstract: In order to study the indentation size effect (ISE) of germanium single crystals, nano-indentation experiments were carried out on the (100), (110) and (111) plane-orientated germanium single crystals. The true hardness of each crystal plane of germanium single crystals was calculated based on the Meyer equation, proportional sample resistance (PSR) model and Nix–Gao model, and the indentation size effect (ISE) factor of each crystal plane was calculated. Results show that, the germanium single crystals experience elastic deformation, plastic deformation and brittle fracture during the loading process, and the three crystal planes all show obvious ISE phenomenon. All three models can effectively describe the ISE of germanium single crystals, and the calculated value of Nix–Gao model is the most accurate. Compared with the other two crystal planes, Ge (110) has the highest size effect factor m and the highest hardness, which indicates that Ge (110) has the worst plasticity.

Key words: germanium single crystal; indentation size effect; Meyer equation; proportional sample resistance (PSR) model; Nix-Gao model

1 Introduction

Germanium single crystal is an important semiconductor and infrared optical material. Compared with the traditional semiconductor material, silicon, germanium has twice the electron mobility and is characterized with high infrared refractive index and low dispersion rate, which is beneficial to reducing image aberrations in the field of optical devices. Therefore, it is widely used in many important domains such as optical fiber communication, aerospace, and military equipment [1,2]. About 95% of germanium in the infrared optical field is used to make infrared lenses and viewing windows, such as infrared detectors on space shuttles and space stations, individual infrared night vision devices in military equipment, infrared night vision systems for tanks, and infrared

thermal imaging weapon defense systems. At the same time, the solar germanium-based substrate batteries that have emerged in recent years have high photoelectric conversion efficiency and have a longer service life than conventional silicon battery. However, germanium single crystal is a hard-brittle material and it is easy to produce shredded chips during the processing, which makes it difficult to obtain a smooth surface [3]. At the micro-nano scale, the germanium single crystal also exhibits a significant indentation size effect (ISE), and the hardness of the material will decrease with the increase of the applied load [4]. As the hardness of the material is closely related to other mechanical properties such as elastoplasticity, strength and wear resistance of the material, it is necessary to study the ISE of germanium single crystal under micro-nano conditions.

Most of the investigations on the ISE of metals

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and foil materials are based on nanoindentation experiment and crystal plasticity finite element method (CPFEM) model. Some scholars have carried out nanoindentation experiments on single crystal metals or semiconductor materials such as sapphire [5], silicon single crystal [6], aluminum single crystal [7] and iridium single crystal [8] to study their basic mechanical properties. LIU et al [9] studied the anisotropic accumulation of single crystal copper during indentation based on plastic slip theory and established crystal plasticity finite element method (CPFEM) model. The activation of different slip systems contributes to the anisotropy of pile-up patterns. Also, they studied the effects of grain/phase boundary (GB/PB) on mechanical properties and microtexture evolution of Cu bicrystals and Cu-Al bicrystals based on (CPFEM) model and Bassani and Wu hardening law [10]. LI et al [11] used two-dimensional crystal plasticity finite element simulation to study the grain scale plasticity of NiTi shape memory alloy (SMA) during uniaxial compression deformation at 400 °C.

Also, there are many studies about the size effect of metal foils or crystals. For example, OUYANG et al [12] studied the effect of grain size and material thickness on C5210 phosphor bronze thin sheets by tensile experiments. LIU et al [13] conducted a nanoindentation experiment on high-purity tungsten and studied the effect of indentation size and grain/sub-grain size on the hardness of the material based on the Nix-Gao model. LIU et al [14] studied the size effect of aluminum single crystal during indentation based on the CPFEM model, and indentation size effect was explained by the Mises stress, critical resolved shear stress and the lattice curvature. However, the ISE of germanium single crystal are rarely studied. Because of the anisotropy of germanium single crystal, the mechanical properties of different crystal plane orientations are different. Therefore, in this work, the load-depth and hardness-depth curves of different crystal plane-orientated germanium single crystals are studied bv nanoindentation experiment, and the ISE of germanium single crystal is analyzed based on Meyer equation, proportional sample resistance (PSR) model and Nix-Gao model.

2 Experimental

The samples used in the experiment were (100), (110) and (111) crystal plane-orientated germanium single crystals. The size of each crystal plane was 10 mm \times 10 mm \times 0.5 mm. The experimental materials were polished and the surface roughness is less than 0.5 nm. In order to ensure the accuracy of the experimental results, the surface of the material was firstly cleaned with acetone to eliminate stains on the surface and then the material was fixed on the metal block with glue (Fig. 1).



Fig. 1 Material fixed on metal block

The standard static method was employed in the nano-indentation experiment, the loading depths were set to be 30, 70, 100, 500, 1000 and 2000 nm to obtain mechanical properties such as hardness and elastic modulus of different crystal planes of germanium. The loading strain rate was 0.05 s^{-1} during the experiment, and the unloading process was not started until the load reached the maximum indentation depth for 10 s. In order to ensure that the adjacent test points did not interfere with each other, a separation distance of 100 µm was set between every two test points. After the relevant nano-indentation parameters were set, the experiment was performed, and the device the experimental connected to instrument automatically output the load-displacement curve.

3 Results and discussion

3.1 Mechanical parameters of material

Table 1 gives the mechanical parameters of the material obtained by the experiment, where h is the experimental design indentation depth; P_{max} is the

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Table 1 Mechanical parameters of materials obtained from experiment															
<i>h</i> /nm	$P_{\rm max}/{\rm mN}$			$h_{\rm c}/{ m nm}$			$h_{ m f}/ m nm$			E/GPa			H/GPa		
	(100)	(110)	(111)	(100)	(110)	(111)	(100)	(110)	(111)	(100)	(110)	(111)	(100)	(110)	(111)
30	0.14	0.19	0.14	25.3	27	19.7	15.8	14.3	8.14	231	244	275	13.4	16.9	25.0
70	0.81	1.31	0.91	65.6	66.8	61	30.8	29.6	29.1	157	257	190	13.1	17.4	14.6
100	1.97	1.98	1.77	96.5	94.1	89.1	38.4	41.3	39	160	186	170	13.8	21.7	13.3
500	42.5	43	40.5	498	490	488	251	233	243	151	159	160	9.82	12.9	9.88
1000	151	162	145	1002	987	991	502	520	556	132	142	125	9.12	9.88	8.25
2000	500	594	422	2022	1985	1997	1179	1135	1134	117	131	106	7.58	9.18	6.67

maximum loading load; h_c is the contact depth of the indenter and material; h_f is the residual indentation depth; *E* is the elastic modulus; *H* is the hardness of material.

3.2 Mechanical property curves and surface topography

Figure 2 shows the load-depth curves of germanium single crystal of different orientations. It is shown that at different indentation depths, the unloading curves of different crystal planes of germanium single crystal are not returned to the initial position, which indicates that the material produces plastic deformation during the unloading process.

Figure 2(a) shows that when the indentation is shallow, the load-depth curve of different crystal planes of germanium single crystal appears to fluctuate significantly, which is mainly caused by the work hardening of germanium single crystal [15]. Also, when the unloading depth is about 300 nm, a pop-out point is generated on the unloading curve (marked in Fig. 2(a)). During the unloading process, the deformed surface of the material gradually recovers. As is seen from the unloading curve, the load-depth curves of the material do not return to the initial point, which demonstrates that the material undergoes plastic during unloading deformation the process. Therefore, it can be considered that the pop-out point on the unloading curve is the critical point at material which the changes from elastic deformation to plastic deformation. As shown in Fig. 2(b), when the indentation depth reaches 600 nm, there are pop-in points on the load-depth curves of the three crystal planes, which demonstrates that the internal structure of the germanium single crystal material has changed. The reason is that there is a phase change inside the



Fig. 2 Load-depth curves of single crystal germanium at different indentation depths: (a) 500 nm; (b) 1000 nm; (c) 2000 nm

material or the generation of dislocation sources in the material. As the load increases, the crystal germanium structure of the single crvstal transforms from diamond cubic structure to the metallic β -tin phase, which results in the change of mechanical properties of the germanium single crystal [16]. Therefore, the loading curves become discontinuous, and there are pop-in points on the loading curve. At this time, the internal atomic lattice of the germanium crystal begins to break. Figure 3 shows a deformation diagram of Ge (110) under SEM at a depth of 500 nm. It can be seen from Fig. 3 that when the indentation depth reaches 500 nm, cracks begin to occur at the edge of the indentation.



Fig. 3 Surface topography of Ge (110) with indentation depth of 500 nm

Figure 2(c) shows that as the indentation depth is further increased, the pop-in points on the load-depth curves of the three crystal planes of the germanium single crystal begin to increase, which indicates that the brittle fracture of the material increases at this time. When the indentation depth is little, the germanium single crystal mainly undergoes elastic deformation, and the initial hardnesses of the three crystal planes are close, so that the load-depth curves are relatively close. Since germanium single crystal is an anisotropic material, the mechanical properties of each crystal plane are different. As the load increases, the atomic lattice fracture degree of each crystal plane is different, which results in a difference in the load-depth curve. When the indentation depth is 2000 nm, the differences in the load-depth curves of the three crystal planes become larger, and at the same indentation depth, the load of the Ge (110) is the largest, which demonstrates that the Ge (110) has a larger hardness than the other two crystal planes.

Figures 4 and 5 show the elastic recovery rate and the hardness/elastic modulus-depth curves of different crystal planes of germanium single crystal at different indentation depths, respectively.

It can be seen from Fig. 4 that the elastic recovery rate of different crystal planes of germanium single crystal shows a tendency to rise first and then decrease. When the indentation depth is little, the germanium single crystal has a high elastic recovery rate, which indicates that the elastic deformation mainly occurs at this time. As the indentation depth gradually increases, the elastic recovery rate begins to decrease, and the material mainly undergoes plastic deformation and is accompanied with elastic deformation. Combined with the load-depth curve analysis, it can be concluded that the germanium single crystal undergoes elastic deformation, plastic deformation and brittle fracture during the loading process.



Fig. 4 Elastic recovery rates of each crystal plane of germanium single crystal with different indentation depths

Figure 5 shows the variation of the hardness and elastic modulus of germanium single crystal under different indentation depths. The hardness and elastic modulus of different crystal planes of germanium single crystal both decrease with the increase of the indentation depth, showing an obvious ISE phenomenon. And when the indentation depth is 2000 nm, the hardnesses of the Ge (100), (110) and (111) are 7.58, 9.18 and 6.67 GPa, respectively, and the elastic moduli are 117, 131 and 106 GPa, respectively. The Ge (110) has the largest hardness and elastic modulus compared to the other two crystal planes, which is consistent with the law of the load-depth curve of germanium single crystal.



Fig. 5 Hardness-depth and elastic modulus-depth curves of different crystal planes of single crystal germanium: (a) Ge (100); (b) Ge (110); (c) Ge (111)

3.3 ISE of germanium single crystal

The reason for the ISE can be explained as follows. In the initial stage of indentation, the indenter only acts on the upper surface of the material, and the hardness and elastic modulus of the material mainly depend on the strain distribution of the surface layer of the material, which results in the significantly decrease of hardness and elastic modulus of the material in the low load region [17]. In addition, the dislocations, plastic deformation and residual stress are generated inside the material due to the subsurface damage during material processing. As the indentation depth increases, the work which is done by the indenter is converted into the plastic deformation energy of the material, and the dislocation density and residual stress of the material begin to decrease, which results in a corresponding decrease in hardness and elastic modulus of the material, thus exhibiting the ISE phenomenon [18,19].

Currently, there are three main methods for investigating the ISE of material: Meyer equation, strain gradient theory and proportional specimen resistance (PSR) model.

3.3.1 Meyer equation

The Meyer equation is the simplest and broadest method to describe the ISE [20,21]. According to the Meyer equation, the load and the indentation depth satisfy the following relationship:

$$P_{\max} = Ah_{\rm c}^n \tag{1}$$

where A and n are constants related to the material, and n is also known as the Meyer equation index; P_{max} is the maximum indentation load; h_c is the contact depth of the indenter with the material. The values of A and n can be obtained by taking a natural logarithm of the two sides of the equation and performing a linear fit. Figure 6 shows the fitting curve of $\ln P_{\text{max}}$ and $\ln h_c$ for each crystal plane of germanium single crystal, where r is the correlation coefficient of the fitted curve.

As is shown in Fig. 6, the Meyer equation index n of Ge (100), (110) and (111) are 1.8815, 1.8529 and 1.7700, respectively. When n=2, the material will not produce an indentation size effect; when n<2, the material will perform a significant size effect; when n>2, the material will exhibit an inverse indentation size effect.

Although the Meyer equation can well reflect the ISE of the germanium single crystal, the physical meaning of the parameters A and n in the equation is not clear and the true hardness of the material without indentation size effect can not be calculated so that it is usually used as an empirical formula [22]. Therefore, further research is needed to investigate the relationship between load and pressure depth.

3.3.2 Proportional specimen resistance (PSR) model The PSR model assumes that during the



Fig. 6 Fitting curves of $\ln P_{\text{max}}$ and $\ln h_{\text{c}}$ based on Meyer equation: (a) Ge (100); (b) Ge (110); (c) Ge (111)

loading process, there is the friction between the material and the indenter due to the elastic deformation of the material. The surface of the material will have a resistance against the indenter, and a part of the indentation load will offset this resistance [23]. The expression for this model is

$$P_{\max} - a_1 h_c = a_2 h_c^2 \tag{2}$$

where a_1 is the elastic resistance of the material and the frictional resistance between the material and the indenter; a_2 is the hardness of the material. For a given material, a_1 and a_2 are constants. Shifting Eq. (2) and then dividing it by h_c on both sides of the equation yield

$$\frac{P_{\max}}{h_c} = a_1 + a_2 h_c \tag{3}$$

The values of a_1 and a_2 can be obtained by fitting P_{max}/h_c-h_c to Eq. (3). For the Berkovich indenter, according to the Oliver–Pharr theory [24], the hardness of the material is calculated as

$$H = \frac{P_{\text{max}}}{24.5h_{\text{c}}^2} \tag{4}$$

namely,

$$H = \frac{a_2}{24.5} \tag{5}$$

Figure 7 shows the fitting curve between P_{max}/h_c and h_c . According to the fitting results, it can be seen that the PSR model can also effectively describe the ISE of each crystal plane of the germanium single crystal. The hardnesses calculated based on Eq. (5) of Ge (100), (110) and (111) are 4.991, 5.956 and 4.265 GPa, respectively. 3.3.3 Nix-Gao model

The Nix-Gao model was proposed based on the strain gradient theory [25,26]. The model considers that when the indenter is loaded into the material, the indentation is caused by a dislocation loop generated by geometrically-necessary dislocations in the material under the indenter. During the loading process, the strain gradient effect of the material results in the change of geometrically-necessary dislocations, which causes the ISE. Based on the strain gradient theory, the dislocation density can be expressed as [25]

$$\rho_{\rm G} = \frac{3\tan^2\theta}{2bh} \tag{6}$$

where $\rho_{\rm G}$ is a geometrically-necessary dislocation density; θ is the angle between the indenter and the surface of the material, and for the Berkovich indenter, θ =70.3°; *b* is the magnitude of Burger's vector; *h* is the indentation depth. According to the von Mises flow law and the Taylor relationship, the indentation hardness and the indentation depth satisfy the following relationship [27]:

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}} \tag{7}$$



Fig. 7 Fitting curves of P_{max}/h_c and h_c based on PSR model: (a) Ge (100); (b) Ge (110); (c) Ge (111)

where *H* is the hardness measured by the experiment; H_0 is the hardness of the material without considering the strain gradient effect; *h* is the indentation depth; *h** is the observable depth of the size effect, which is also known as the microscopic size characteristic length of the material.

The values of H_0 and h^* in Eq. (7) can be obtained by conducting the nonlinear fitting

function. According to the analysis of Fig. 5, three crystal planes of germanium single crystal do not show an obvious size effect until the indentation depth exceeds 100 nm due to the influence of work hardening. In order to ensure the accuracy of the analysis results, only the last four groups of data were taken for analysis. Figure 8 shows fitting curve of H-h based on the Nix–Gao model.

According to the fitting results, the microscopic characteristic lengths h^* of Ge (100),



Fig. 8 Nonlinear fitting curves of H-h based on Nix–Gao model: (a) Ge (100); (b) Ge (110); (c) Ge (111)

(110) and (111) are 203.456, 572.72 and 220.51 nm, respectively, and the corresponding hardnesses are 7.91, 8.21 and 7.28 GPa, respectively. When the indentation depth does not exceed h^* , the germanium single crystal will exhibit a significant size effect phenomenon; while the indentation depth is more than h^* , the size effect of the germanium single crystal will weaken. The Nix-Gao model considers the relationship between the hardness H and the indentation depth h, while the PRS model doesn't take the hardness H into consideration. Due to the existence of work hardening, the hardness value calculated based on the Nix-Gao model is higher than that of PRS model.

In order to quantify the strength of the size effect, MANIKA and MANIKS [28] investigated the power-law exponent relationship between the nanohardness and the indentation depth of the material, and found that the hardness and the loading depth satisfy the following relationship:

$$H=Ch^{-m} \tag{8}$$

where C is a constant, which is only related to the shape factor of the indenter; m is the indentation size effect index, whose value can reflect the strength of the indentation size effect. The value of the size effect index m can be obtained by conducting the linear fitting of $\ln H$ and $\ln 1/h$ from Eq. (8). Figure 9 shows the fitting curves between H and of 1/h.

From the fitting results, the size effect indexes of Ge (100), (110) and (111) are 0.191, 0.295 and 0.217, respectively. Usually, for metal and semiconductor materials, the value of m is between 0.12 and 0.32. And the better the plastic properties of the material are, the smaller the value of m is [28]. Compared with Ge (100) and Ge (111), Ge (110) has the largest size effect index m and the highest hardness according to the previous study. Therefore, it can be concluded that Ge (110) has the worst plasticity. MANIKA and MANIKS [28] also gave m values for several different materials. The values of m for Au, Ag and In are 0.15, and that for Sn is 0.16; while the value of m for Al_2O_3 is 0.32. It is known that as for metal materials such as Au and Ag, their atoms interact through metal bonds, while the Al₂O₃ atoms are connected by covalent bonds. Germanium is a semiconductor material but has metallic properties so that the value of *m* is between them. Therefore, it can be concluded that the value



Fig. 9 Relationship between hardness (*H*) and reciprocal indentation depth (1/h) of single crystal germanium: (a) Ge (100); (b) Ge (110); (c) Ge (111)

of m is related to the bonding mode between the material atoms.

Combined with the above analysis, all three models can effectively describe the ISE of germanium single crystal. However, there are inevitable defects in the crystal material. The Nix–Gao model considers the geometricallynecessary dislocations under the indenter, and the calculated result is close to the change trend of the hardness curve. Therefore, Nix–Gao model is more accurate in describing the ISE phenomenon of germanium single crystal.

4 Conclusions

(1) During the loading process, germanium single crystal undergoes elastic deformation, plastic deformation and brittle fracture, and there are pop-in points and pop-out points on the load-depth curve. Compared with the other two crystal planes, Ge (110) requires the largest load when reaching the same indentation depth.

(2) When the indentation depth exceeds 100 nm, the hardness and elastic modulus of each crystal plane of the germanium single crystal decrease with the increase of the loading depth, which shows a significant indentation size effect.

(3) Meyer equation, PSR model and Nix-Gao model can all effectively describe the ISE of germanium single crystal, but the Meyer equation cannot be used to calculate the hardness value of germanium single crystal. The hardnesses calculated based on the PRS model are 4.991, 5.956 and 4.265 GPa, respectively. The charactristic lengths h^* of Ge (100), (110) and (111) calculated based on the Nix-Gao model are 203.456, 572.72 and 220.51 nm, respectively, and the corresponding hardness values H_0 are 7.91, 8.21 and 7.28 GPa, respectively; the size effect index m are 0.191, 0.295 and 0.217, respectively. When the indentation depth does not exceed h^* , the germanium single crystal performs a significant ISE phenomenon; while the indentation depth is more than h^* , the ISE weakens.

(4) Compared with the Ge (100) and Ge (111), Ge (110) has the largest hardness and size effect index m. Namely, Ge (110) has the worst plasticity. Therefore, it is not suitable to select this crystal plane for processing in the actual production of germanium parts.

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不同晶体取向锗单晶的压痕尺寸效应

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摘 要:为了研究锗单晶的压痕尺寸效应,对(100)、(110)和(111)晶面取向的锗单晶进行纳米压痕实验。基于 Meyer 方程、比例试样阻力(PSR)模型和 Nix-Gao 模型计算锗单晶各晶面无压痕尺寸效应时的真实硬度值,并基于 Manika 幂律关系计算锗单晶各晶面的尺寸效应因子。结果表明:锗单晶在加载过程中发生弹性变形、塑性变形和 脆性断裂 3 个阶段,且 3 个晶面均表现出明显的尺寸效应现象。3 种模型均能较好地描述锗单晶的尺寸效应,其 中 Nix-Gao 模型的计算值最为准确。相比于其他两个晶面,Ge (110)的尺寸效应因子 m 值最高,且具有最高的硬 度值,表明该晶面的塑性性能最差。

关键词: 锗单晶; 压痕尺寸效应; Meyer 方程; 比例试样阻力(PSR)模型; Nix-Gao 模型

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