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LETTER

Nanomechanical properties of the stigma of dragonfly *Anax parthenope julius* Brauer

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Introduction

Natural biomaterials have many optimized structures and functions adapted to their living surroundings through the evolution over millions of years. Based on the structures of biomaterials, some biomimetic materials, including nanocomposite materials, have been developed [1]. There are more than one million species of insects in nature. Insects are important biological resource for developing biomimetic techniques [2]. Dragonfly can hover, flap its wings for flight and accelerate [3]. The mass of the wings of a dragonfly is only 1–2% of its whole body mass, but the wings can stabilize their body and have a high load-bearing ability during flight [4].

Living dragonflies, *Anax parthenope julius* Brauer (Odonata, Anisoptera, Aeschnidae, Anax) were collected in Chuangchun, China. Figure 1 shows the digital camera photograph of a female dragonfly *Anax parthenope julius* Brauer. The body length of the dragonflies used for tests is about 68 mm. The wing span of the forewings and the hindwings of the dragonfly are about 97 mm and 96 mm, respectively.

Two right wings of the dragonfly are shown in Fig. 2. A wing is composed of veins, membranes, nodus and stigma. The veins are mainly made up of chitin material, which is a kind of crystalline polymer with similar characteristics as cellulose or Teflon [4]. There is some resilin, a rubberlike protein, in the vein joints. The resilin material is used for controlling torsion [5]. The wings of the dragonfly have some typical structures, such as the nodus and the stigma. The nodus lies in the center of the leading edge of the wings and the stigma like a fuscous mark is situated near the wing tip. It was considered that the nodus and the stigma may not only improve the flexibility but also prevent fatigue fracture of the wings [6]. The stigma plays such roles as balance of the mass center, stabilization at high-speed flight and elimination of the airflow vibration. If cutting the stigmas off the wings, the dragonfly could still fly but the flight becomes unstable.

The nanomechanical properties of the stigma on the wings of the dragonfly *Anax parthenope julius* Brauer were investigated using a nanoindenter (TriboIndenter, Hysitron Inc., USA). The maximum and minimum indenting forces used for tests were 30 mN and 100 nN, respectively; the load resolution is less than 1 nN and the step size of lengthways displacement is 13 nm. A Berkovich tip was used for determining the nanomechanical parameters of the stigma. This kind of tip is often applied as a standard tip for nanoindentation tests [1].

The determination of the hardness of a material with a nanoindenter is based on the theory of nanoindentation, which was originally put forward by Boussinesq [7]. Oliver–Pharr [8] method for measuring nanoindentation modulus and hardness has been accepted extensively. The hardness (H) and the reduced modulus (E_r) can be computed based on the Oliver–Pharr method as follows:

$$H = \frac{P_{\max}}{A(h_C)} \quad (1)$$

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Fig. 1 Digital camera photograph of dragonfly *Anax parthenope julius brauer*

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A(h_C)}} \tag{2}$$

where $P_{\max}$ is the maximum indentation force; β is a constant related to tip geometry, such as, β equals 1 for a spherical tip, 1.034 for a Berkovich tip and 1.012 for a Vicker tip; $A(h_C)$ is the resultant projected contact area between the tip and specimen surface; S is the initial unloading contact stiffness.

In order to calculate H and E_r precisely, we must obtain the values of S and $A(h_C)$ firstly. According to the unloading segment of the force–displacement curve, the initial unloading contact stiffness (S) can be obtained, namely:

$$P = B(h - h_f)^m \tag{3}$$

$$S = \left|\frac{dP}{dh}\right|_{h=h_{\max}} = Bm(h_{\max} - h_f)^{m-1} \tag{4}$$

where B is a constant determined by an experiment; h_f is residual displacement; m is a constant related to the tip geometry and m equals 1 for a spherical tip, 1.5 for a Berkovich tip and 2 for a coniform tip; h is displacement and $h_{\max}$ is the maximum displacement.

The resultant projected contact area between the tip and specimen surface ($A(h_C)$) can be calculated as follows.

$$A(h_C) = C_0h_C^2 + C_1h_C + C_2h_C^{1/2} + C_3h_C^{1/4} + C_4h_C^{1/8} + C_5h_C^{1/16} \tag{5}$$

where C_0 is a constant which equals 24.5 for a Berkovich tip and 2.598 for a rectangular pyramid tip; $C_1 \sim C_5$ can be obtained from the experimental data; and h_C is the contact displacement.

The elastic modulus (E) can be derived from the following equation.

$$\frac{1}{E_r} = \left(\frac{1-\upsilon^2}{E}\right)_{specimen} + \left(\frac{1-\upsilon^2}{E}\right)_{indenter} \tag{6}$$

where υ is the Poisson's ratio. The first part on the right in (6) is dependent on the specimen material and the second one is dependent on the indenter material. E = 1114 GPa and υ = 0.07 for a diamond tip. The Poisson's ratio is about 1/3 for the rigid materials and a little smaller than 0.5 for the elastic materials and the toughness biomaterials [9]. Generally, υ is taken as 0.25 if the material of the specimen is unknown [10].

Figure 3 shows a stereoscopic photograph of the cross-section of a stigma of the dragonfly hindwings. The nanomechanical parameters of the stigma were derived based on the above theory of materials nanoindentation. The nanomechanical tests of the cross-section of the dragonfly stigma were performed using a trapezoidal loading function. A force–displacement curve of the stigma is shown in Fig. 4. The force–displacement curve

Fig. 2 The photo of a pair of right wings of the dragonfly

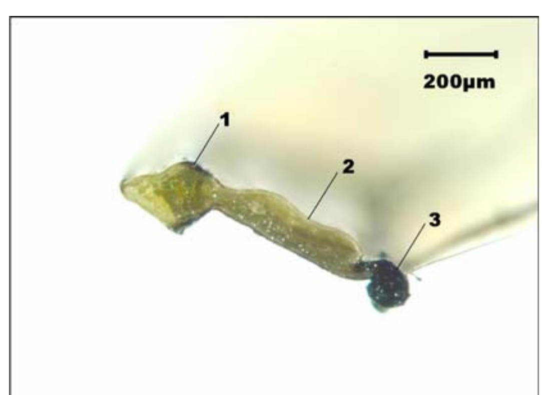

Fig. 3 The stereoscopic photograph of the cross-section of the stigma

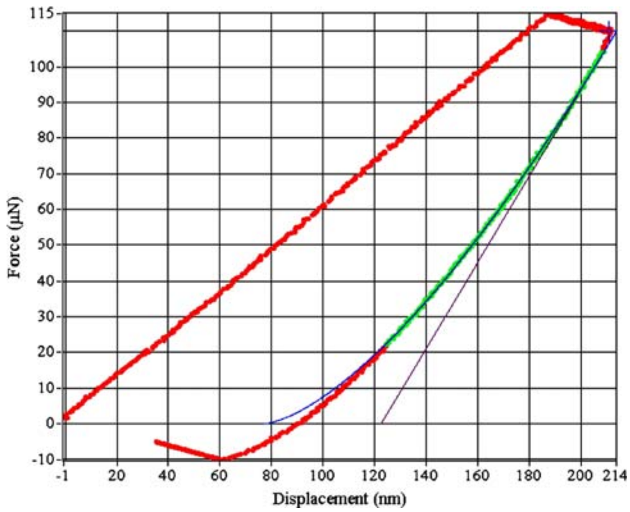

Fig. 4 The force–displacement curve of the stigma during the trapezoidal loading

illustrates a viscoelastic property of the stigma material, in particular, it can be observed during the holding time at maximum load under the trapezoidal loading. The force restarts to increase after the end of the unloading curve, indicating an adhesion phenomenon occurred between the indentation tip and the stigma material. This influence should be considered in tests of nanoindentation property of materials. In research of creep property of metal in nanoindentation experiment, two factors were considered. One was the holding period at maximum load (holding time) and the other was the loading rate [11, 12]. If the holding time was set as zero, the initial segment of unloading curve would appear evagination as a result of viscoelastic properties of biomaterials [1]. The effects of the holding time at the maximum load and the loading rate on elastic modulus and hardness are related to viscoelastic deformation for biomaterials [13, 14].

For effectively investigating the effects of holding time and loading rate on the experimental result, the experimental optimization design method was used. The method is a helpful tool for experiments. The experimental plan designed by the method will take the least experimental times and acquire accurate result [15].

First, the factors of holding time and loading rate were set as factor A and factor B, respectively. Then by the experimental experience, the two factors were considered to investigate in four level values which were 0 s, 20 s, 40 s, 60 s and 3 μN/s, 23 μN/s, 53 μN/s, 73 μN/s, respectively. By the experimental optimization design method, the test plan was made, as shown in Table 1, where the first row was set as test number, the second row was set as the factor A (holding time), the third row was set as the factor B (loading rate) and the fourth row was set as y (the experimental result of reduced modulus). The experimental scheme $L_8(4^2 \times 2^2)$ used for the nanomechanical tests was obtained from the reformation of the scheme $L_8(2^7)$ using the parataxis method [15]. According to the scheme, there only need to consider the level 1 and level 4 of the factor B when level 1 of factor A (equaling 0 s) was investigated. The other levels are similar as it. So, the whole tests were only eight times, instead of sixteen times without usage of the experimental optimization design method.

Then, the trapezoidal-type loading function will be according to the value of the factor A and B to arrange. Take example for the test 1, the holding time and the loading rate of a trapezoidal-type loading function used in test is 0 s and 3 μN/s, respectively. Ten repeating indentations were conducted to determine the average values of E_r and H. The test results were shown in y row in Table 1.

As for analysis of the results, as shown in Table 1, y_{j1}, y_{j2}, y_{j3} and y_{j4} were the sum of the y at the same level for the factor A and factor B, respectively. For the factor A, the y_{j1} is equal to the sum of the y of the test 1 and 2, the rest may be deduced by analogy. And the $\bar{y}_{j1}, \bar{y}_{j2}, \bar{y}_{j3}$ and $\bar{y}_{j4}$ is the average value of y_{j1}, y_{j2}, y_{j3} and y_{j4}, respectively. The R_j is the range of reduced modulus which is equal to the maximum value subtracting the minimum value of $\bar{y}_{j1}$, $\bar{y}_{j2}$, $\bar{y}_{j3}$ and $\bar{y}_{j4}$. Its value is the direct reflection of importance

Table 1 The measuring schemes and result analysis of nanomechanical parameters of the dragonfly stigma

The test numbers The factors	Holding time(s) A	Loading rate(μN/s) B	E_r(GPa) y
1	1(0)	1(3)	0.24
2	1(0)	4(73)	0.18
3	4(60)	1(3)	0.13
4	4(60)	4(73)	0.12
5	2(20)	2(23)	0.13
6	2(20)	3(53)	0.10
7	3(40)	2(23)	0.11
8	3(40)	3(53)	0.10
y_{j1}	0.42	0.37	
y_{j2}	0.23	0.22	
y_{j3}	0.22	0.21	
y_{j4}	0.25	0.31	
$\bar{y}_{j1}$	0.21	0.18	
$\bar{y}_{j2}$	0.11	0.11	
$\bar{y}_{j3}$	0.11	0.10	
$\bar{y}_{j4}$	0.13	0.15	
R_j	0.10	0.08	
Primary and secondary factors	A, B		
Combined optimization	A_2B_3, A_3B_3		

of factors. In the experimental optimization methods, a higher value of R_j means a greater influence to the test results [15]. So, the factor A (R_j = 0.10) has more effect than the factor B (R_j = 0.08) on the experimental result, which means the value of the holding time is more important to the stigma test.

The value of y_{j1} is larger than that of the others (y_{j2}, y_{j3} and y_{j4}) and almost 50% difference value. But the value of y_{j2}, y_{j3} and y_{j4} are close, suggesting that the results are unbelievable when the factor A is 0 s and the results are believable only when the factor A should be over 20 s. It was concluded that the accurate values of E_r can be obtained as long as the holding time was more than 20 s. It means that the effect of viscoelastic phenomenon on the test results of biomaterials' E_r and H can be eliminated when the holding time is beyond 20 s.

It was known by the experimental experience that the least value of $\bar{y}_j$ is the best. For the factor A, $\bar{y}_{j2}$ is 0.1145 and $\bar{y}_{j3}$ is 0.1093. The both values were very close each other especially when they were chosen in the 2 significant figures (the both were equal to 0.11 approximately). So, the most optimization combination were A_2B_3 (the holding time = 20 s, the loading rate = 53 μ N/s, and one test time is 31.32 s for the experimental conditions) and then A_3B_3 (the holding time=30 s, the loading rate=53 μN/s, one test time is 51.32 s for the experimental conditions). Because the investigated specimens were living biomaterial and the test time will affect their mechanical properties, the test time was the important factor on the experimental results. That is, the shorter the test time, the better the result is.

As above mentioned, the plan A_2B_3 will save 20 s during each test compared with the plan A_3B_3 . A trapezoidal-type loading function was utilized for indentation tests and the experimental parameters were determined to be a loading rate of 53 μN/s and a holding time = 20 s. Six repeating indentations were conducted to determine the average values of E_r and H. The experimental results were showed in the Table 1.

Generally, the peak depth (h_{max}) is required less than 10% of the thickness of the material specimen for a solid material or thin solid film for a nanoindentation test. The thickness of the stigma is about 200 µm, 102 µm and 107 µm at the position-1, the position-2 and the position-3, respectively. The peak depth (h_{max} = 303.8 nm) was much less than 10% of the thickness of the stigma. So, it is obvious that the nanoindenter can be applied well to determine the nanomechanical properties of the stigma of the dragonfly's wings.

The two specimens were investigated for position-1, -2, and -3 of the dragonfly stigma as shown in Fig. 3. Figure 5 illustrates the test results. It can be found that E_r and H at the position-1 are the maximum. The maximum values of E_r and H at the position-1 is because the position-1 is nearly the leading edge. E_r at the position-2 is the minimum and H at the position-3 is the minimum.

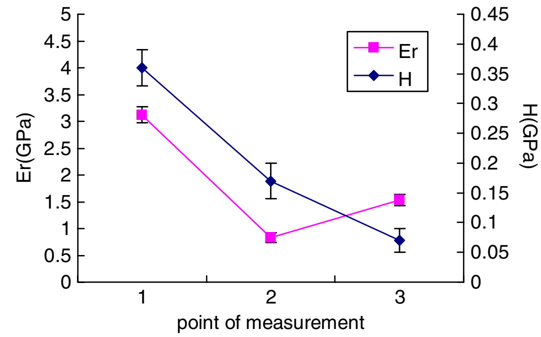

Fig. 5 The experimental results of the nanomechanical properties (reduced modulus (Er) and hardness (H)) of the dragonfly stigma at three positions

Because the influence of $A(h_C)$ or h_C is different to E_r and H, the error bars for them are different (shown as Fig. 5). By the formula (1), (2) and (5), the following can be obtained,

$$E_r \propto 1/h_C \tag{7}$$

and

$$H \propto 1/h_C^2 \tag{8}$$

While the difference of E_r and H as following,

$$\Delta E_r \propto \frac{1}{h_1} - \frac{1}{h_2} \tag{9}$$

$$\Delta H \propto \frac{1}{h_1^2} - \frac{1}{h_2^2} = \left(\frac{1}{h_1} - \frac{1}{h_2}\right)\left(\frac{1}{h_1} + \frac{1}{h_2}\right) \tag{10}$$

It can be found by formula (9) and (10) that the influence of h_C to the H is greater than that to the E_r. So, when the displacement changes, the difference range of H are greater than that of E_r suggesting that the error bars for H are much greater than those for E_r.

The nanoindenter provide a chance to resolve the investigation matter of stigma. The realization of mechanical properties of stigma of dragonfly is helpful to lucubrate its flying mechanism. And it will provide an inspiration for designing some new structures and materials of mechanical parts.

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