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Nanotribology of nacre: Anisotropic dissipation in a multiscale hybrid material

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ARTICLE INFO

Article history:

Received 23 October 2011

Received in revised form

17 September 2012

Accepted 21 September 2012

Available online 7 October 2012

Keywords:

Mother of pearl

Biocrystal

Strength

Organic matrix

ABSTRACT

Sheet nacre (so-called *mother-of-pearl*) is a hybrid biocomposite with a multiscale structure including nanograins of calcium carbonate (97 wt%–40 nm in size) and two organic matrices: (i) the “*intracrystalline*” (mainly composed by silk-fibroin-like proteins), and (ii) the “*interlaminar*” one (mainly composed of β -chitin and proteins). Micro/nanotribological behaviour was investigated on sheet nacre displaying various configurations (so-called *face-on* and *edge-on*), by varying the orientation of the matrices vs. the sliding direction. Different levels of frictional dissipated energy were observed as a function of both: (i) the type of matrix which is involved in the dissipation mechanism (*intracrystalline* or *interlaminar*), and (ii) the orientation of the matrices themselves vs. the sliding direction. These various dissipative ways can involve either, multiscale wear processes entailing the both matrices, or irreversible deformation only, without any wear process. They have been identified and explained by considering the double composite structure of sheet nacre.

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1. Introduction

Sheet nacre (the pearly internal layer of molluscan shells) is a hybrid biocomposite with a multiscale structure including nanograins of calcium carbonate (97 wt%) [1–3] and two organic matrices (3 wt%) [3–5]. At the nanoscale, biocrystals (ca 40 nm in size) are drowned in an “*intracrystalline*” organic matrix (4 nm thick) in order to form a microsized flat organomineral aragonite tablet (thickness about 500 nm) [6–9]. At a larger scale [10–14], these tablets are themselves surrounded by a porous “*interlaminar*” organic matrix (thickness: 40 nm, porosity in-between 35% and 59%) building up a material displaying highly interesting properties [15–21]: low density, good biocompatibility [22–25] and osteogenic properties for human bone regeneration [22,26–28]. Thus, this material is currently being studied as small prostheses (e.g. rachis and dorsal vertebra prostheses, which are both subjected to micro-slip and fretting wear), or for the creation of new organic/inorganic bio-inspired hybrid materials [29–35].

Recently, friction and wear behaviour of sheet nacre were studied in dry friction [36–39] and in liquid medium [36], respectively. Results have shown that:

- (i) the environment strongly influences the wear mechanisms of nacre by means of various physico-chemical interactions on the water-soluble “*intracrystalline*” organic phase [36];
- (ii) the energy absorption ability of the matrix is drastically reduced in presence of nanoshocks generated during friction by the dynamic solicitations. As a result, cracks migrate in the tablet involving the formation of wear nano-debris [37];
- (iii) at high mean contact pressure, additional thermal-induced wear damage involves the degradation of the organic matrices, that increases dramatically the wear rate of nacre [38–40];

Thus, for this kind of solicitations, nacre is clearly subjected to peculiar thermal [38,41] and mechanical [36,37] damage processes, which occur at two order of magnitude, involving its double composite structure—i.e. both organic matrices are always directly involved in the friction-induced energy dissipation mechanisms [42–48] and in the subsequent damage mechanisms, as well [42, 49–53]. So, the orientation of the tablets [14,54] – and especially the one of the matrices themselves—with respect to the sliding direction seems to be a key parameter for understanding the

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dissipation mechanisms of sheet nacre under dynamic solicitations, as met in tribological tests. Indeed, anisotropic specificities of sheet nacre under *quasi-static* solicitations— (i.e., tension [15,49,55,56], three- and four-point bending [44], shear [44,57], micro- [56–59] and nano-indentation [37,53,60–63]) have been actively studied or modelled [45,54] in the past. But, up to now, tribological results have been achieved with a *face-on* configuration sheet nacre only [36–39] – i.e. when the tablets are oriented more or less parallel to the friction direction—leaving the study of the real influence of the tablets orientation on the wear mechanisms not investigated yet.

This work aims to compare friction and wear mechanisms of sheet nacre cut with a *face-on* and a *edge-on* configurations, respectively, in order to understand how the orientations of the organic phases vs. the sliding direction can change the ways to dissipate the frictional energy. In order to avoid any additional thermal effects, and to better control the environment around the contact, tribological tests have been carried out under low contact pressures, by using a multi-asperity nanotribo-meter [64–66] working in an environmental glove box.

2. Experimental part

2.1. Samples

Samples are made of dry sheet nacre extracted from giant oyster *Pinctada maxima* [3,4,6–8,36,67] cut with a *face-on* and *edge-on*

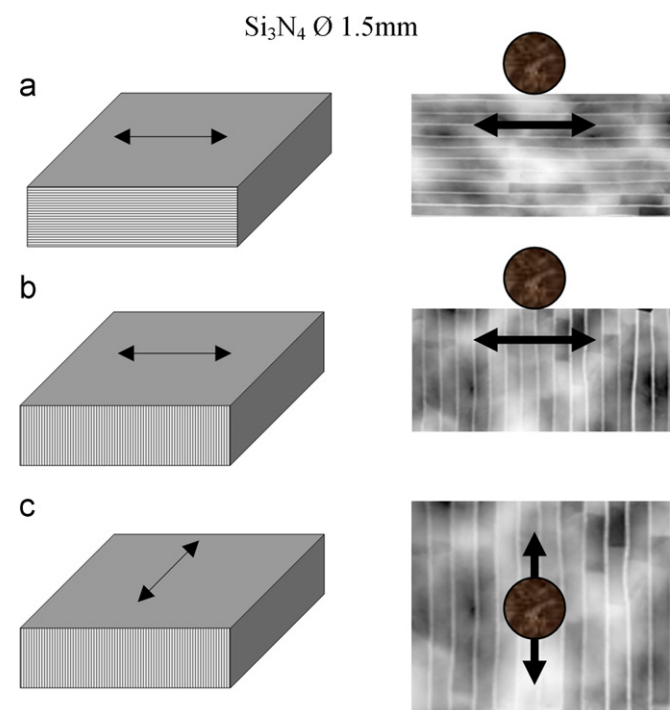


Fig. 1. Various configurations of the samples: (a) *face-on* orientation; (b) *edge-on* perpendicular to the sliding direction, and (c) *edge-on* parallel to the sliding direction.

configuration, respectively (Fig. 1). Surfaces are polished with a RMS roughness about 14.5 ± 0.6 nm. Three configurations have been tested with respect to the direction of sliding:

- a *face-on* configuration *parallel* to the direction of sliding (Fig. 1a);
- an *edge-on* configuration *perpendicular* to the direction of sliding (Fig. 1b);
- an *edge-on* configuration *parallel* to the direction of sliding (Fig. 1c);

As mentioned earlier, this type of sheet nacre has a multiscale structure [3,68,69]:

- On the one hand, aragonite tablets (500 to 700 nm thick) are surrounded by an “*interlaminar*” thin network (about 40 nm thick) of a biological porous organic adhesive (average porosity $49 \pm 12\%$ [63]) mainly composed of β -chitin and proteins [10–13].
- On the other hand, each aragonite tablet is constituted by nanosized biocrystals of CaCO_3 surrounded by a water-soluble “*intracrystalline*” organic phase (mainly silk-fibroin-like proteins) organised as a foam with very thin walls and closed porosity (4 nm) [3,4,6–8,36]. The average size of these initial biocrystals is about 38 ± 21 nm [62].

In addition, mechanical properties of each component—i.e. biocrystals and organic matrices—have previously been assessed by using SEM analysis, acoustic microscopy [63,70], spherical and sharp nanoindentation tests [37,60,62], combined with multiscale numerical simulations using FEM [37,39] and homogenisation approach [62]. Main mechanical features are reported in Table 1. The mechanical properties of the “*interlamellar*” matrix are very close to the ones determined by Xu et al. [71] using more local assessments.

2.2. Nanotribological setup

2.2.1. In situ friction assessment

The experimental device (Fig. 2a) is constituted by a *ball-on-disc* nanotribo-meter manufactured by CSM Instruments (Switzerland) [66,72]. A pin is mounted on a stiff lever, designed as a frictionless force transducer ($K_x = 265.1 \text{ N m}^{-1}$; $K_z = 152.2 \text{ Nm}^{-1}$). The friction force is determined during the test by measuring the deflection of the elastic arm (low load range down to $50 \mu\text{N}$). The ball (Si_3N_4 –1.5 mm) is loaded onto a flat nacre sample with a precisely known force using closed loop. The load and friction resolutions are about $1 \mu\text{N}$. Tribological tests are carried out in linear reciprocating mode at room temperature (22°C) under ambient air (RH 35%), in an environmental glove box. The normal load varies from 10 to 80 mN corresponding to a contact pressure varying from 0.15 to 0.65 GPa. The stroke frequency, the stroke length and the stroke length resolution are respectively 10 Hz, ± 0.5 mm, and 250 nm. The velocity and the sliding distance are respectively 1 mm s^{-1} and 0.2 m, corresponding to 100 cycles in order to reach the steady state in friction.

Table 1
Mechanical properties of elemental components of dry sheet nacre.

Elemental components of nacre	E (GPa)	ν	σ_y (GPa)
Aragonite biocrystals	96.75 (5.67) ^a	0.17 (0.05) ^b	13.4 (2.1) ^c
50% porous “ <i>interlaminar</i> ” organic matrix	13.4 (0.79) ^d	0.29 (1.7e–3)	0.118 (0.02)
“ <i>intracrystalline</i> ” organic matrix	3.81 (0.41) ^b	0.296 (1.4e–3) ^b	–

^a Assessed by sharp nanoindentation test [62].

^b Computed by a Mori-Tanaka model [62].

^c Computed by a Hertzian model [62].

^d Assessed by combining acoustic microscopy and spherical nanoindentation test [63].

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