Ductile and brittle material removal mechanisms in natural nacre—A model for novel implant materials

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A B S T R A C T

Nacre is a composite material found in the inner layer of sea shells. It consists of soft organic and hard inorganic components arranged in a complex hierarchical structure. Due to this arrangement, nacre exhibits outstanding mechanical properties (elastic modulus: 64.70 ± 3.50 GPa, hardness: 4.41 ± 0.45 GPa, density: 2.6 g/cm³). Therefore, nacreous implant materials have a high potential in many fields. In medical science, these materials might be used for bone replacement. This article provides an insight into the material removal mechanisms occurring in the scratching of natural nacre. Scratch tests are a simplification of the grinding process and are used to investigate the influence of single input parameters on the material removal. Different scratch tool geometries and varying processing parameters are applied, so that the material removal efficiency can be evaluated by analyzing the process forces. Additionally, the scratch geometry is examined by using a scanning electron microscope (SEM) and optical profilometer images as well as photomicrographs. The results of these examinations provide knowledge on the machinability of nacre and also on the machinability of new nacreous materials.

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1. Introduction

Today, the materials commonly used for implants still have significant deficiencies regarding their life time. They often display unsatisfactory wear characteristics (Plitz, 2000). In particular, wear particles can cause inflammatory reactions and lead to a fracture of the prosthesis (Boehler et al., 2008). Among all materials used for the manufacturing of prostheses, ceramics generally produce fewer wear particles, but they are difficult to finish; roughness peaks can lead to a high concentration of stresses and actually to a fracture of the prosthesis (Boehler et al., 1994). Bronzino (2000) gives an extended overview of different biomaterials and their properties.

In nature, nacre is produced by mollusks, like seashells or oysters. A typical shell consists of two layers. The outer layer contains large calcite crystals, and the inner layer contains aragonite crystals combined with polymers—i.e. nacre. In scratch tests, the outer and inner layer showed very similar behavior. With an increasing scratch depth, the scratch forces increase linearly. However, in Vicker's indentation tests under dry and wet conditions, the scratch geometry is examined by using a scanning electron microscope (SEM) and optical profilometer images as well as photomicrographs. The results of these examinations provide knowledge on the machinability of nacre and also on the machinability of new nacreous materials.

The outstanding properties of nacre can be explained by the behavior of the material at the micro and nano level. The sliding of the aragonite platelets is essential for the high toughness of nacre. In tensile tests with nacre, the material did not break up to an extent of 2%. This mechanism is controlled by the nano-space between the aragonite platelets. The aragonite platelets have an average thickness of 0.2–1.5 μm. In the top view, the platelets have a polygonal shape with an average diameter of 5–20 μm.

The outstanding properties of nacre can be explained by the behavior of the material at the micro and nano level. The sliding of the aragonite platelets is essential for the high toughness of nacre. In tensile tests with nacre, the material did not break up to an extent of 2%. This mechanism is controlled by the nano-space between the platelets. The plateau of the plateau can slide up to 100–200 nm until the material fails due to the removal of the platelets. This leads to the following conclusions: the polymer layer must be weaker than the platelets, and it must be strongly bonded to them; there must be nanoasperities between the aragonite layers; the aspect ratio of
the platelets must be high enough, and the waviness of the platelets must be overcome before they can slide. After the destruction of the organic matrix, the nanoasperities and aragonite bridges prevent the sliding of the platelets, but they do not provide large resistance to the sliding beforehand. Therefore it is more probable that the waviness of the platelets prevents the sliding before the platelets are removed (Barthelat et al., 2007; Espinosa et al., 2009).

As mentioned above, the organic matrix contributes much to the outstanding strength of nacre. It ensures that the mechanical stresses are distributed evenly and thus prevents the generation of force peaks, which increases the strength and hardness of the material. Cracks are deflected along the organic layers, increasing their way and therefore the crack resistance of nacre. This mechanism as well as the bridges between the organic matrix and the platelets supports the pull-out of the platelets (Roessler et al., 2008; Wang et al., 2001). The special structure of nacre makes it anisotropic regarding its mechanical properties (Barthelat et al., 2007).

Nacre has also been proven to be biocompatible (Atlan et al., 1999; Barthelat et al., 2007). Several studies on the structure of nacre have been carried out so far (Wada, 1961; Wise, 1970; Grgoire, 1972; Mutvei, 1979; Rousseau et al., 2005). It has been demonstrated that nacre—like bone—is a composite material that is produced by biomineralization (Sailer and Weber, 2000; Gao et al., 2003; Roessler et al., 2008). These investigations prove that nacreous materials have a high potential for use in implant materials. An overview of the mechanical properties of nacre, bone and aluminum oxide is presented in Table 1.

In recent years, different methods for the production of biomimetic hybrid materials have been investigated. Gehrke et al. (2005) and Heinemann et al. (2006) have demonstrated the mechanisms by which these hybrid materials are produced in nature. They have also succeeded in producing artificial nacre by demineralization of the organic matrix. Initial approaches show that nacreous structures can be synthetically produced as thin layers, at least under laboratory conditions (Tang et al., 2003; Podsiadlo et al., 2005). For this purpose, a substrate with a thickness of 5 μm and with properties similar to those of nacre is produced by the layer-by-layer deposition of organic and inorganic materials. Another method to produce nacreous structures is directional solidification. This means that an aqueous polymer solution containing ceramic nano- or micro-platelets is frozen at a specific cooling rate while a local temperature gradient is being applied. Afterwards the ice is removed by freeze drying, leaving layered structures with open spaces where the ice-crystals used to be. These spaces can be filled with a polymer matrix (Kubera et al., 2004; Delville et al., 2006; Petersen et al., 2006; Wienecke et al., 2009). Until now, no one has succeeded in producing large amounts of nacreous hybrid materials which show mechanical properties similar to or even better than those of natural nacre.

No matter how exceptional the properties of nacre are, they are probably not sufficient for certain applications, for example for rolling and sliding applications in aqueous media. Therefore, researchers are trying to develop novel hybrid materials based on the natural composite-structure of nacre, but with improved properties. The properties of these hybrid materials can only be adjusted in a systematic manner by using an interdisciplinary approach (Menzel et al., 2007). The production of the composite is not the only problem to solve; furthermore, a low damage machining process for this composite has to be found and its general suitability for the production of implant materials has to be tested. Only few studies concerning the machining of ceramic materials with an organic component have been conducted so far (Denkena et al., 2009). This article focuses on the material removal mechanisms occurring in scratch tests on nacre with different input parameters.

Rotational scratch tests have been used in many investigations to model the grinding process of certain materials, e.g. of silicon carbide (Uhlmann, 1993) and aluminum oxide (Roth, 1994). The diamond tips, which are used as scratching tools, are either pyramidal or conical. In some cases, diamond abrasive grains are bonded into the tool material. In the respective tests, the diamond tips have a very strong negative rake angle, and different tip radii. The wear of the tool also influences the behavior of the specimen material. A key objective of these tests is to identify the critical single grain chip thickness \( t_g \), which indicates the transition from ductile to brittle material removal. In the present work, the results from scratch tests are used to model the grinding process of natural nacre and to gain knowledge on the machinability of nacreous materials.

### 2. Scratch tests on natural nacre

#### 2.1. Sample preparation

For the scratch tests, *Pinctada maxima* nacre has been used. It is also called "sheet nacre" due to the arrangement of its aragonite platelets, which are overlapping in a disorderly manner (Wang et al., 2001; Katti et al., 2003; Dashkovskiy et al., 2007). The examined nacre samples were rectangular (30 mm \( \times \) 20 mm) and had a thickness of approximately 1.3 mm. The samples have first been embedded in resin, then ground and polished. The grinding and polishing processes have been carried out using different SiC-grit sizes (1400–300) and using water as a cooling and flushing medium. In order to avoid an influence of soaked nacre samples on the results, drying periods of at least 24 h at room temperature were applied before the experiments. The hardness and the elastic modulus of two samples have been measured at the Institute of Microtechnology of the Leibniz University of Hanover, using a Hysitron Tribolindenter® with a Berkovich tip. Eight indentations were produced on each nacre sample. The average indentation depth was approximately 185 nm, the average elastic modulus was \( E_r = 64.70 \pm 3.50 \) GPa, and the average hardness was \( H_r = 4.41 \pm 0.45 \) GPa. These values are consistent with the results of previous examinations from nanoindentation tests (Menig et al., 2001) and three-point bending tests (Richter et al., 2010, work in review). It can therefore be assumed that the sample preparation processes did not affect the properties of the material surface.

#### 2.2. Experimental setup

Scratch tests are a simplified method for examining the influence of a single grain on a material removal process. The respective material is cut using varying input parameters to study the material removal mechanisms in a controlled environment. Due to the simplification of the tool geometry and the kinematics, the scratch test results cannot be directly transferred to processes like grind-

<table>
<thead>
<tr>
<th>Properties</th>
<th>Nacre</th>
<th>Bone</th>
<th>( \text{Al}_2\text{O}_3 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elastic modulus (GPa)</td>
<td>70 (wet–90) (dry)</td>
<td>3–30</td>
<td>290–330</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>800</td>
<td>600–3000</td>
<td>400–560</td>
</tr>
<tr>
<td>Compressive strength (MPa)</td>
<td>450</td>
<td>130–180</td>
<td>4000–5000</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>2.95</td>
<td>1.7–2.0</td>
<td>3.98</td>
</tr>
</tbody>
</table>

This table provides a comparative overview of the mechanical properties of nacre, bone, and aluminum oxide.
Table 2
Parameters used in the scratch tests.

<table>
<thead>
<tr>
<th>Input parameters</th>
<th>Process Parameters:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Workpiece: Pinctada maxima nacre</td>
<td>$d_p$ Infeed ($\mu$m)</td>
</tr>
<tr>
<td>Tool: Diamond tip mounted on an aluminum wheel</td>
<td>$h_{(\text{max})}$ (maximum) single grain chip thickness ($\mu$m) in axial scratch tests</td>
</tr>
<tr>
<td>$\gamma$ Tool tip angle (°)</td>
<td>$h_{(\text{max})}$ (maximum) single grain chip thickness ($\mu$m) in tangential scratch tests</td>
</tr>
<tr>
<td>$r$ Tool tip radius ($\mu$m)</td>
<td>$f_a$ Axial feed (mm)</td>
</tr>
<tr>
<td>$d_a$ Diameter of the aluminum wheel (mm)</td>
<td>$f_t$ Tangential feed (mm)</td>
</tr>
<tr>
<td>$\alpha$ Mounting platform inclination angle</td>
<td>$u_{f_a}, u_{f_t}$ Axial and tangential feed rate (m/min)</td>
</tr>
<tr>
<td></td>
<td>$v_c$ Cutting speed (m/s)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Process data</th>
<th>Output parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>$F_n$ Normal force (N)</td>
<td>$w_r, w_{th}$ Real and theoretical scratch width ($\mu$m)</td>
</tr>
<tr>
<td>$F_t$ Tangential force (N)</td>
<td>$d_r, d_{th}$ Real and theoretical scratch depth ($\mu$m)</td>
</tr>
<tr>
<td></td>
<td>$k_d, k_w$ Depth and width form factor (−)</td>
</tr>
<tr>
<td></td>
<td>$l_{h_{th}}$ (h$_{th}$) Length of the scratch ductile zone (mm)</td>
</tr>
<tr>
<td></td>
<td>$h_{cg}$ Critical single grain chip thickness ($\mu$m)</td>
</tr>
</tbody>
</table>

The experiments have been carried out on a CNC profile grinding machine of the type Blohm Profimat 307 (Fig. 1). This machine achieves cutting speeds of up to $v_c = 210$ m/s and feed rates of $v_f = 25$ m/min. The polycrystalline diamond tips used in the scratch tests have been ground with a tip angle of $\gamma = 120^\circ$ and a tip radius of $r = 50, 100$ and $150\ \mu$m, and have been soldered at the holder tool tip (Fig. 1, bottom right). An aluminum disk with a diameter of $d_s = 400$ mm has been used to hold the diamond tips. The embedded samples have been glued onto a steel plate, which has itself been attached to a Kistler force sensor of the type 9117A 1.5, and horizontally positioned by means of a mounting platform. A reference point on the surface of the sample has been determined by measuring the forces occurring during the contact between tool and sample. This point has later been used to compare the theoretical depth and width ($d_{th}$ and $w_{th}$) with the measured depth and width of the scratches ($d_r$ and $w_r$).

In the scratch tests with tangential translations, only the tangential feed rate $v_{f_t}$ has been varied in order to change the maximal single grain chip thickness $h_{(\text{max})}$, which has been calculated as follows (Zeppenfeld, 2005):

$$h_{(\text{max})} = r \left(1 - \left(1 - \frac{a_e}{r}\right) \cos \phi_2 \right)$$  

(1)

whereas $a_e$ is the depth of the scratch and $r$ is the diameter of the aluminum wheel. A detailed explanation for the calculation of the angle $\phi_2$ is given in (Zeppenfeld, 2005). For the scratch tests with axial translation, the maximum single grain chip thickness is $h_{(\text{max})} = a_e = a_p$. Fig. 1 (top right) shows that the grooves are generated by a superposition of rotational and translational movements. In the axial scratch tests, the axial feed rate $V_{f_a}$ is perpendicular to the cutting speed $V_c$. The variation of the input parameters has been carried out according to Table 3. To allow for statistical inferences, 10 scratches from two different samples have been analyzed (5 scratches per sample).

![Experimental setup](image1)

**Fig. 1.** Experimental setup for the scratch tests on natural nacre.
Table 3
Scratch tests on natural nacre using diamond tips.

<table>
<thead>
<tr>
<th>Series Nr.</th>
<th>Cutting speed $v_c$ (m/s)</th>
<th>Tool radius $r$ (μm)</th>
<th>Axial feed $f_a$ (mm)</th>
<th>Infeed $a_p$ (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td></td>
<td>100</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>10</td>
<td>20</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td></td>
<td>50</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>100</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>150</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td></td>
<td>100</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>50–50</td>
<td></td>
</tr>
</tbody>
</table>

Process: Tangential scratch test ($h_{umax}$ from Eq. (1))

<table>
<thead>
<tr>
<th>Series Nr.</th>
<th>Cutting speed $v_c$ (m/s)</th>
<th>Tool radius $r$ (μm)</th>
<th>Axial feed $f_a$ (mm)</th>
<th>Infeed $a_p$ (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>10</td>
<td></td>
<td>50</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>20</td>
</tr>
</tbody>
</table>

2.3. Determination of the critical single grain chip thickness $h_g$

In order to investigate the material removal mechanisms occurring in the machining of nacre, images of the scratches have been taken using an EVO 60 VP scanning electron microscope (Carl Zeiss AG) for the detection of secondary electrons. Additionally, the machined surfaces have been scanned by a μ-scan (Nanofocus AG, Oberhausen), a modular 3D profilometer system for non-contact optical surface measurement within the micrometer and nanometer range. In order to scan translucent materials like nacre, the sample surface must be coated with a 4 nm gold layer prior to the measurements. A resolution of 1 μm in the x- and z-directions and of 10 nm in the y-direction has been chosen; the directions of the coordinates are shown in Fig. 1. The depth, length, width and roughness $Ra$ of the scratches have been determined from these measurements. The macroscopic tool wear has been investigated by means of SEM images taken before and after the scratch tests.

2.4. Parameters for the evaluation of the scratch tests

To distinguish between brittle and ductile material removal, the width and depth form factors $k_w$ and $k_d$ are calculated as follows:

$$k_w = \frac{w_r}{w_{th}}$$

$$k_d = \frac{d_r}{d_{th}}$$

whereas $w$ and $d$ represents the width and depth of the scratches and the subindices $r$ and $th$ represent respectively the measured and theoretical values. When $k_d = k_w = 1$, the measured geometry of the scratch corresponds to the calculated one (Apmann, 2004) and the material removal is ductile. If the coefficients are below 1, the material recovers elastically after the scratch process. If they are above 1, the material removal mechanism is brittle.

The length of the ductile material removal zone $l_R(h_g)$ has been determined by measuring the roughness $Ra$ along the scratches. The deepest point of the ductile zone $h_g$ can be calculated using the values of the setup kinematics. For scratch depths with values above $h_g$, lateral and subsurface cracks along the scratches have been observed. Therefore, the determination of $h_g$ is an essential part of this work. To determine $h_g$, the roughness $Ra$ has been measured in the entry and exit scratch zones (Fig. 2, right) using the optical profilometer. Based on these data, the roughness $Ra$ in these two zones of the scratches has been measured. It was observed that in the ductile zone $l_R(h_g)$, $Ra$ was at least 50% below its value in the brittle zone. Apart from the roughness amplitude, the roughness peak frequency in the brittle zone also rises. Consequently, the resulting transition from the ductile to the brittle zone could easily be identified (Fig. 2, right).

The normal and tangential forces $F_n$ and $F_t$ have both been measured in the scratch tests. The forces are presented in Fig. 2 (left); they are overlapped with the profile of the scratch with $h_{umax} \approx 50 \mu m$. It can be observed that the force signal exhibits some peaks, which might be attributed to material breakouts indicated in the SEM image in Fig. 2 (top right). To avoid distortions of the
Fig. 3. Influence of the tool radius \( r \) on the material removal mode (middle zone of the scratches).

Fig. 4. Influence of the tool radius \( r \) on the crack distribution (scratch cross section).

As an addition to Figs. 3 and 4, the critical single grain chip thickness \( h_g \), the width and depth form factors \( k_w \) and \( k_d \) as well as the process forces are presented in Fig. 5. This figure shows that the critical single grain chip thickness \( h_g \) decreases with an increase in the tool radius \( r \). The form factor \( k_w \) also decreases; the form factor \( k_d \) remains almost constant. An exception occurs with \( r = 100 \mu m \), where \( k_d \approx 1 \), probably due to the cohesive chip formations (see also Figs. 3 and 4). The normal force \( F_n \) increases on a diminishing scale. This can be explained by the presence of polymers, which cause elastic material behavior. The tangential force \( F_t \) increases due to the increasing cross sections of undeformed chips.

3. Influence of the input parameters on the material removal mechanisms

3.1. Axial scratch tests

3.1.1. Influence of the tool radius \( r \) on scratch quality and process forces

Fig. 3 shows SEM images from the middle of 3 scratches machined with different tool tip radii. These images indicate that there is an increase in the breakout size with an increasing tool tip radius. With \( r = 50 \mu m \), there are many small breakouts at the base of the scratch. The scratch base also shows the orientation of the nacre layer. With \( r = 100 \mu m \), loose chips or “cohesive formations” appear instead of small breakouts, indicating a loosening of the polymers present in nacre. With \( r = 150 \mu m \), the forces are high enough to remove larger aragonite blocks, thus producing deeper cracks in the scratch base. The SEM images also show that the width of the scratches does not linearly increase with an increasing tool radius: with \( r = 150 \mu m \), the width of the scratch is almost the same as with \( r = 100 \mu m \).

Fig. 4 also illustrates that there is a higher amount of the radial cracks at the scratch base with larger tool tip radii. The larger tool tip radius causes higher process forces and an enlarged contact zone. As stated above, cohesive chip formations occur with \( r = 100 \mu m \). With \( r = 150 \mu m \), nacre is elevated at the sides of the scratch. This effect does not occur with \( r = 50 \mu m \) and \( r = 100 \mu m \) because the material is removed, producing lateral cracks in the scratch. The photomicrographs do not show increasing cross sections for larger tool radii, either. Instead, larger tool radii cause deeper and longer cracks. With \( r = 150 \mu m \), elastic material recovery takes place (see also Fig. 5, \( k_d, k_w \approx 1 \)).

Fig. 6 shows the middle zone of the scratches for different cutting speeds. At a cutting speed of \( v_c = 5 \) m/s, only small breakouts at the base of the scratch are observed, indicating that the aragonite layers are not parallel to the sample’s surface. By increasing the cutting speed up to \( v_c = 10 \) m/s, cohesive chip formation occurs in the scratch base. At higher cutting speeds (\( v_c = 40 \) m/s and \( v_c = 60 \) m/s), larger breakouts in the base and the side of the scratches are produced due to the detachment or “pull-out” of aragonite platelets. These breakouts are characterized by large flat surfaces.

Fig. 6 also illustrates that the breakouts extend into the bulk material. They expand deeper into the sample material at higher cutting speeds. Fig. 7 confirms this assumption. The depth and width of the scratch cross section increase at increasing cutting speeds. At cutting speeds of \( v_c = 5 \) m/s and \( v_c = 10 \) m/s, the scratch cross sections are smaller than those at higher cutting speeds. The cracks in the bulk material are not deep enough to produce more layer detachments in the scratch base. At lower cutting speeds, there are layer breakouts and probably platelet breakouts, but only a few layer detachments. At the right side of the micrograph signals by filters, raw data have been used for the evaluation of the results.
for $v_c = 5$ m/s (Fig. 7), there is a black section which might have been produced by the natural growth lines of the sample material (Barthelat et al., 2007). These growth lines (also called “mesolayers”), as well as the polymers which connect the aragonite platelets, serve as force or crack deflecting lines. In the case of $v_c = 5$ m/s, the connections fail producing a material elevation at the right side of the scratch. As indicated above, cohesive layer detachments occur at a cutting speed of $v_c = 10$ m/s.

The conclusions drawn from the SEM images and the photographs are supported by the results presented in Fig. 8. At higher cutting speeds, the critical single grain chip thickness $h_g$ decreases and the form factors $k_w$ and $k_d$ increase. The forces also increase slightly. The discontinuous increase and the high standard deviation are probably due to the polymer layers in nacre, which can also cause adhesions on the tool tip (Fig. 7, bottom right).

### 3.1.3 Influence of the infeed $a_p$ on the scratch quality and process forces

Fig. 9 shows SEM images of several scratches with different infeed $a_p$. Similar to the variation in the cutting speed $v_c$ and the tool radius $r$, an increase in the infeed $a_p$ leads to larger breakouts. For $a_p = 27 \ \mu$m and $a_p = 35 \ \mu$m, larger and deeper breakouts

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**Fig. 5.** Influence of the tool tip radius on the critical chip thickness $h_g$, the form factors $k_w$ and $k_d$ and the process forces $F_n$ and $F_t$.

**Fig. 6.** Influence of the cutting speed $v_c$ on the surface quality in the middle zone of the scratches.

**Fig. 7.** Influence of the cutting speed on the crack distribution (scratch cross section).
Fig. 8. Influence of the cutting speed $v_c$ on the critical single grain chip thickness $h_g$, the width and depth ratios $k_w$ and $k_d$, and the normal and tangential forces $F_n$ and $F_t$.

Fig. 9. Influence of the infeed on the surface quality in the middle zone of the scratches. are observed (Fig. 9), indicating the pull-out of platelets. The scratch widths also increase with an increasing infeed.

The photomicrographs in Fig. 10 for scratch tests with an infeed of $a_p \approx 18 \mu m$ already show less breakouts. With infeeds of $a_p \approx 27 \mu m$ and $a_p \approx 35 \mu m$, radial cracks are produced on the subsurface of the scratches. In addition, elastic material recovery occurs with $a_p \approx 18 \mu m$.

Fig. 11 (top left) shows the critical single grain thickness $h_g$ for different infeeds $a_p$. An increase in $a_p$ does not affect $h_g$. Moreover, an increase in the infeed leads to an almost linear increase in the process forces, whereas the normal force increases more rapidly than the tangential one. The tangential forces are affected by the shear strength, which is smaller parallel to the aragonite platelets because of the nacre polymers.

As already indicated by the SEM images and photomicrographs, the form factors $k_w$ and $k_d$ behave similarly; they rise with an increase in the infeed, indicating the presence of more breakouts. The diagram of $k_w$ and $k_d$ in Fig. 11 illustrates the transition from elastic to plastic behavior. For $a_p = 18 \mu m$, there is elastic material behavior, while for $a_p = 27 \mu m$ and $a_p = 35 \mu m$, plastic behavior

Fig. 10. Micrographs of the scratches on nacre showing the influence of the infeed on the geometry of the scratches in the scratch cross section.
occurs. However, the photomicrographs in Fig. 10 show that there is brittle material removal in all cases. This means that the results obtained from the photomicrographs and the ones from \( k_w \) and \( k_d \) are not consistent.

3.2. Tangential scratch tests

The SEM images for down and up scratching with different maximal single grain chip thicknesses \( h_{c,m} \) are presented in Fig. 12 (left and right respectively). By increasing \( h_{c,m} \) in tangential down and up scratching, larger breakouts are produced at the bottom and at the edges of the scratches. In down scratching, the cutting speed and the feed rate are in the same direction and the chip thickness at the entry is larger than that at the exit of the contact zone. On the contrary, in up scratching, the tool and the material move in different directions, so that the relative cutting speed is higher. Moreover, the chip thickness at the entry zone is smaller than that at the exit zone of the scratch. In general, the tendencies observed for axial rotatory scratching can be transferred to tangential rotatory scratching. The main difference is that the maximum single grain chip thickness \( h_{c,m} \) in Fig. 12 is much smaller than those in Figs. 9 and 6, where \( h_{c,m} = a_p \). For up scratching at \( h_{c,m} = 1.4 \mu m \), an almost ductile material removal can be observed.

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**Fig. 11.** Influence of the infeed \( a_p \) on the critical chip thickness \( h_g \), the form factors \( k_w \) and \( k_d \) and the process forces \( F_n \) and \( F_t \).

**Fig. 12.** Influence of the infeed on the surface quality in the middle zone of the scratches.

**Fig. 13.** Influence of the maximal single grain chip thickness \( h_{c,m} \) on the material removal mode in down scratching (scratch cross section).
The micrographs of the scratches for down and up scratching are presented in Fig. 13. The micrographs clearly illustrate that radial cracks in the damage zone mainly occur in down scratching. Similar tendencies can be derived from the SEM images in Fig. 12. This behavior might be due to different compressive and shear stress–strain behaviors in nacre (Barthelat et al., 2007).

The normal and tangential forces $F_n$ and $F_t$ are presented in Figs. 14 and 15. They rise progressively with higher single grain chip thicknesses. The forces in up scratching are smaller than those in down scratching. This indicates that the material can be removed more easily in the up scratching mode. The width form factor $k_w$ slightly decreases for higher values of $h_{\text{cumax}}$ and the depth form factor $k_d$ can be considered to be constant $k_d = 1$ in both up and down scratching.

4. Discussion

The results of this work are summarized by a schematic representation of the behavior of nacre in scratch tests under varying conditions (Fig. 16). Suitable parameters for the grinding of nacre can be derived from the experimental tests. The aim is to achieve ductile material removal, so that the base surface and the subsurface of the grooves remain undamaged. Ductile material removal is observed at the entry and exit zones of the scratches at a cutting speed of $v_c = 5–10 \text{ m/s}$ and a maximum infeed of $a_p = 20 \mu\text{m}$. Values of $h_{\text{cumax}}$ above 5 $\mu\text{m}$ cause brittle material removal. The highest critical single grain chip thicknesses are obtained with a tip radius of $r = 50 \mu\text{m}$.

Because of its special structural composition, nacre exhibits different mechanical properties in the parallel and perpendicular direction to the aragonite layers (Menig et al., 2001). The surfaces created in the sample preparation were almost parallel to these layers. As a result, typical eruption shapes at the bottom of the grooves occurred (Fig. 16, right).

To quantify the scratch results, different parameters have been evaluated. The form factors $k_w$ and $k_d$ do not provide relevant findings on the ductile behavior of nacre within the parameter ranges used in this work. In many cases, $k_d$ was below 1 although brittle material removal occurred. However, $h_g$ represented the limit of the ductile material removal mode more accurately under the influence of varying input parameters.

An increase in the tool tip radius $r$ results in a decrease in $k_w$, while $k_d$ remains almost constant. One explanation for this is the distribution of the cutting forces. With larger tip radii $r$, the cutting forces $F_n$ and $F_t$ spread over a larger area in the subsurface of the scratches, thus reaching more organic layers in the bulk material and causing elastic/plastic deformation. The tool compresses the material, but when the tool has passed on, the material recovers its initial shape, which is partially due to the presence of polymers. Consequently, scratches with smaller form factors are generated by tools with larger tool radii $r$. This also explains the diminishing increase in the normal force $F_n$.

The roughness $R_a$ in the entry and exit zones of the scratches produced in the rotatory scratch tests has been measured in order to obtain the critical single grain chip thickness. Ductile material removal has only been observed for small depths of cut. $h_g$ is smaller in the exit zone than in the entry zone. The material in the exit zone tends to be delaminated rather than to be chipped. Thus its critical single grain chip thickness is about zero. The obtained critical single grain chip thickness for nacre, with $h_g = 1–2.5 \mu\text{m}$, is much bigger than that for other brittle materials, such as silicon carbide ($h_g < 0.25 \mu\text{m}$) (Uhlmann, 1993). This indicates that the grinding of
Fig. 16. Schematic view and examples of material removal mechanisms in the nacre grooves as a function of the tool tip radius \( r \) \((a_p \approx 20 \, \mu m)\), the cutting speed \( v_c \) \((a_p \approx 20 \, \mu m)\) and the chip thickness \( h_{\text{chip}} \) \((t_c = 10 \, \mu m)\).

Nacre in the ductile mode can be carried out using a wide range of input parameters. However, it must be investigated to which extent the interaction with a coolant and the high temperatures can affect the material removal of nacre in the grinding process.

With an increase in the cutting speed, a transition towards brittle material removal has been observed. At a cutting speed of \( v_c = 5 \, m/s \), platelet breakouts have been noticed at the bottom of the scratches. The polymers at the interfaces of the platelets fail, so that whole blocks of nacre platelets are removed at once. At \( v_c = 10 \, m/s \), cohesive chip formations occurred. This is probably due to an increase in the temperature and the shear stresses which cause the pull-out effect.

Although it is evident that brittle material removal occurs at higher values of the infeed \( a_p \), the infeed has almost no influence on the critical single grain chip thickness, the form factors and the process forces. With an increasing infeed, the rake angle becomes only slightly more negative, and only for \( a_p > 30 \, \mu m \), a decrease in \( h_e \) and an increase in \( k_{\text{se}} \) and \( k_d \) is observed. It can be assumed that with higher infeed values, more defects of the material occur, which cause brittle material removal.

The SEM images and the micrographs show that the scratch base and subsurface obtained in the tangential scratch tests are smoother than those resulting from the axial scratch tests. This can be explained by the single grain chip thickness, which varies from \( h_{\text{cumax}} \approx 1.4 \, \mu m \) to \( h_{\text{cumax}} \approx 14 \, \mu m \) for tangential and from \( h_{\text{cumax}} \approx 18 \, \mu m \) to \( h_{\text{cumax}} \approx 35 \, \mu m \) for axial scratch tests. It can also be observed that the forces in axial scratching are always higher than those in tangential scratching, producing more subsurface damages and lateral cracks. The kinematics of the tangential scratching, which is similar to those in grinding processes, causes a chipping of the material rather than pulling-out of the platelets. Therefore it is expected to produce ductile material removal in the grinding of nacre.

Regarding the tool wear, adhesions on the tool tip have only been observed on the tool used at different cutting speeds. This might explain the increase in the process forces at rising cutting speeds. When the cutting speed increases, the temperatures in the shear zone rise. As a result, the nacre polymers can melt and adhere to the tool tip, expanding the contact zone and increasing the process forces.

The influence of the anisotropy on the material removal has not been investigated within the frame of this work. But it is important to notice that despite the different orientation of the nacre layers within the samples as well as different properties of the material in different orientations and many natural defects which may occur in any natural material, the results can be reproduced statistically.

A major advantage of nacre compared to conventional implant materials like titanium, stainless steel, ceramics or polyethylene is its ability to induce bone stimulation and bone cell recruitment (Atlan et al., 1999). Although nacre exhibits outstanding fracture toughness and other mechanical properties which make it favorable for implant materials, it does not meet the requirements of certain applications, e.g. knee implants, where high peak forces can occur and can cause plastic deformation of the implant. In order to fulfill these highly challenging requirements, it is necessary not only to copy the structure of natural nacre but also to improve the properties of this natural composite material. Especially the design of the layers between the inorganic materials should be enhanced for the purpose of making the material more crack resistant and for avoiding major displacements between the layers under dynamical load.

5. Conclusions and outlook

Nacre is a composite material with outstanding mechanical properties and is therefore used as a model for new composites. The purpose of this work has been to study the material removal mechanisms occurring in the scratching of nacre in order to determine the input parameters which cause ductile material removal. The material removal mode can be measured by means of the critical single grain chip thickness, which is obtained from the roughness of the scratches in the entry zone. Ductile material removal has been achieved by lower cutting speeds and lower depths of cut in the scratch tests. By exceeding the critical single grain chip thickness
The results presented in this paper will first be used to design a grinding process for nacre, and then to design a grinding process for newly developed nacreous implant materials. This can, e.g., be done by comparing the obtained critical single grain chip thickness $h_g$ from the scratch tests with the single grain chip thickness in grinding $h_g$. The temperatures in grinding processes are higher than those in scratch tests. Therefore, additional experiments regarding the influence of the temperature on the material removal mode must be carried out. Furthermore, the influence of the material anisotropy on the material removal mode must be investigated in future work.

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