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ALTERNATIVE METHODS FOR SURFACE TEXTURING¹

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Abstract

Surface texturing has a great potential to improve tribological performance. First, possible texturing methods were identified and classified according to their physical principles. In sequence, some alternative texturing methods are presented. Some of them are already currently used either in industry or in laboratory, and innovations or simplifications are described for them. Others are innovative techniques. Some were explored only tentatively, where basic ideas and simple experimental investigations were developed to check their validity. Others were explored in more detail, so that their practical applicability could be identified. The first texturing method was photochemical texturing using a simple and cheap apparatus. Masking with inkjet printing before chemical etching was also successful to texture metallic samples. A new method involving electrochemical texturing, without the need to previously mask the samples to be textured have been studied in terms of voltage, current, mechanical configuration of the apparatus and electrolyte flushing. Another method aims to generate randomly distributed circular pockets on steel surfaces and involves dispersion of small acid droplets in oil. The final method involves the selective formation of hard areas on a steel surface by locallised diffusion, which should then develop into a texture during wear.

Keywords: Surface texturing; PCT; Inkjet printing; MECT.

MÉTODOS ALTERNATIVOS DE TEXTURIZAÇÃO

Resumo

A texturização tem um grande potencial para melhorar o comportamento tribológico de superfícies. Inicialmente, métodos de texturização superficial foram identificados e classificados de acordo com seus princípios físicos. Em seguida, alguns métodos alternativos de texturização são apresentados. Alguns destes já são utilizados na indústria ou em laboratório, mas inovações ou simplificações são descritas para eles. Outros envolvem técnicas inovadoras. Alguns foram explorados somente de forma tentativa, onde idéias básicas e investigações experimentais simples somente verificaram sua validade. Outros foram explorados mais detalhadamente, de forma que sua aplicabilidade prática pudesse ser identificada. O primeiro foi texturização fotoquímica, utilizando um aparato simples e barato. Mascaramento por impressão a jato de tinta antes do ataque químico foi outro método que apresentou sucesso para texturizar superfícies metálicas. Um novo método envolvendo texturização eletroquímica sem a necessidade de mascaramento prévio das superfícies foi estudado em termos de tensão, corrente, configuração mecânica do aparato e fluxo de eletrólito. Um outro método objetiva gerar bolsos circulares distribuídos aleatoriamente em superfícies de aço e envolve a dispersão de gotículas de ácido em óleo. O método final envolve a formação seletiva de regiões duras em uma superfície de aço por difusão localizada, que deverão então se desenvolver em uma textura através de desgaste seletivo.

Palavras-chave: Microtexturização superficial; Ataque fotoquímico; Impressão a jato de tinta; Texturização eletroquímica.

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1 INTRODUCTION

The modification of surface topography, creating a uniform microrelief with regularly shaped asperities or depressions, has received great attention, especially in the last two decades. The process, known as surface texturing, has been successfully used in many applications with the aim of increasing the performance of surfaces in various respects.

Although many applications can be proposed for surface texturing, one of the most successful in engineering is the improvement of tribological performance. In opposition to smooth surfaces, which give large contact areas, and may eventually not have sufficient oil-holding capacity, textured surfaces, with reduced contact areas, can help to remove wear drebris and to improve lubrication.⁽¹⁾

Another reason for the recent interest in surface texturing is the vast technological advance in the area of microfabrication.⁽²⁾ However, many of these techniques are slow and expensive, which makes them inappropriate to produce a large number of cheap components, especially when texturing of large areas is required. For these applications, alternative texturing methods are needed, in order to make surface texturing cost-effective.

The main method used today to texture surfaces in engineering applications is laser texturing.⁽³⁻⁷⁾ However, the use of laser texturing presents limitations. First, the ablation mechanism leads to the formation of raised features around the pockets, which originate from the ejected molten material. The lateral rims are normally hard due to the heat involved in the process and can result in severe abrasive wear. Second, the process is relatively slow, since pockets are normally produced in a serial sequence. Many components that could have their performance increased by surface texturing, such as cylinder liners, are normally cheap. They require much cheaper texturing methods to make the increase in tribological performance achieved with texturing cost-effective.

In laboratory investigations, photochemical texturing has been largely used, because it allows complex patterns to be generated.⁽⁸⁻¹⁰⁾ However, it is complex, relatively slow and expensive to be used for mass production of cheap components.

Other texturing methods have also been reported in the literature. Major advances in microfabrication have driven the area of surface texturing. In principle, the majority of the methods used in the microelectronics industry could be adapted to surface texturing.

This paper aims to survey possible texturing methods, by identifying physical ways by which a surface texture could be produced. In sequence, it presents the development of some innovative texturing methods, with low cost, high texturing speed, and flexibility in terms of pattern geometry, which could greatly contribute to a more widespread use of surface texturing.

2 SURVEY OF POSSIBLE SURFACE TEXTURING METHODS

Alternative manufacturing technologies for surface texturing are needed to overcome the challenges of volume production. The technologies should be cheap and flexible. There should be flexibility both in terms of the shapes of the features and the shapes of the surfaces to be textured. This section identifies and reviews most of the existing texturing methods and also proposes some new possible methods. However, the review is non-exhaustive because surface texturing has been extensively studied in recent years. The methods presented here are reviewed either

because they have already been widely used in industrial applications or because they appear to present improved characteristics. The methods were categorised according to their physical principals into four main groups:

Adding material: the pattern features are created by addition of material to the desired surface, creating small areas of relief.

Removing material: the features are created by removal of material of the surface, creating small depressions.

Moving material: the change in the surface structure is attributable to plastic deformation and redistribution of material from some parts of the surface to others.

Self-forming: wear-resistant regions are formed on a surface, so that a texture develops through wear of the surface, with the wear-resistant regions being left standing above the surrounding material.

Tree structures for each family with their taxonomy are presented in Figure 1. All the processes marked by '*' are processes that require a masking step before the texturing step. In Figure 2, methods that could be used to mask the surfaces are organised in a tree structure.



Figure 1. Schematic representation of the tree structures for methods involving: (a) removing material; (b) adding material; (c); moving material; (d). self-forming by wear.

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Figure 2. Schematics of the tree structure for methods for mask generation.

3 INVESTIGATION OF INNOVATIVE SURFACE TEXTURING METHODS

3.1 Photochemical Texturing (PCT)

A normal laboratory bench was used, which greatly reduced the complexity and costs of the process in comparison to use of a clean room. However, care was taken to keep the environment reasonably clean. The room was isolated from external light and yellow illumination was used. A DC motor with maximum rotational speed of 7000 rpm was used to construct a sample spinner. The spinner ensures the formation of a thin and even resist layer on the metallic surface. A conventional hot plate was used to bake the samples. A conventional microscope light was used as a UV source to expose the resist. Various experimental conditions were tried, based on recommendations found in the literature and on the experience of the previous trials.

Before texturing, the samples were cleaned in an ultrasonic bath (USC) in acetone. The photoresist (AZ 5214, manufactured by AZ Electronic Materials) was spun on the surface. The coated sample was then pre-baked. The patterns were designed using Adobe Photoshop software and printed on A4 paper using a laser printer. They were then photographed to make the masks to be used during the exposure of the resist. This reduced the costs and time that would be involved in the production of conventional metallic masks. The exposure to UV light was made by contact printing for 300 s. The distance between light source and sample was 130 mm. After exposure, the samples were developed in AZ 351B developing solution (1:3.5 vol. in distilled water) for 30-60 s and then post-baked at 150 °C for 180 s to guarantee complete development of the resist. A 10 % nitric acid solution was used to etch the samples at room temperature. The depth of the features was varied by changing the etching time. After etching, the resist was stripped by USC in acetone at room temperature for 4 minutes.

Resists in photolithography may be negative or positive. In positive resists, photochemical reactions caused by UV exposure weaken the polymer by scission of the main and side polymer chains. Thus, the exposed areas will be more soluble in developing solutions. Negative resists are strengthened by UV exposure, which promotes random cross-linking of the main or side of the resist. The exposed areas of the resist will be then less soluble in developing solutions. In this work, the use of different UV light sources allowed the same resin to be used as both a negative photoresist (using a lower power light source) and a positive photoresist (using a

higher power light source). The conditions used for both conditions varied slightly, as summarized in Table 1.

Table 1. Experimental conditions for the photointhographic procedure									
	Sppining	Spinning	Pre-baking	Pre-	Exposure	Developing	Post-baking	Post-	
	rotation	time (s)	temperature	baking	time (s)	time (s)	temperature	baking	
	(rpm)		(°C)	time (s)			((°C)	time (s)	
Negative	3000	60	95	60	300	180	150	180	
resist									
Positive	4000	30	95	60	300	30-60	150	180	
resist									

Table 1. Experimental conditions for	r the photolithographic procedure
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In Figure 3, some examples of the textures generated for the positive resist show the generation of pockets and grooves, whereas in Figure 4 the textures generated for the negative resist are composed of pillars.



Figure 3. Examples of the surface topography of textured samples using a positive photoresist; left, perspective views; right, line profiles.



Figure 4. Example of the surface topography of a textured samples containing pillars using a negative photoresist; left, perspective view; right, line profile.



In principle, ink-jet printing should provide a method to mask printing for the texturing of engineering surfaces.^(11,12) This paper reports an investigation of the use of ink-jet printing as a masking technique for the surface texturing of steel surfaces, using a drop-on-demand printer and a solvent-based ink.

AISI 1010 steel coupons with dimensions of $60 \cdot 20 \cdot 1.5$ mm were used as substrates, and printing was performed with a Dimatix Materials Printer (DMP-2800) with a nominal 10 picoliter drop size. The standoff distance (nozzle-substrate) was 1 mm and the drop impact velocity was 7 m/s. The ink was a commercial lactate solvent-based black ink (dye-based, JetStream PCS 7561, Sun Chemical) with a viscosity of 12.1 mPa s at 25°C and a surface tension of 31.5 mN m-1. The printed samples were assessed by optical microscopy, and laser interferometry was also used to measure the thickness of the ink deposits. For those measurements, a thin film of evaporated gold was applied to improve the surface reflectivity of the ink deposits. After printing, the masked samples were etched with aqueous nitric acid at concentrations of 1%, 5% or 10% at a temperature of 25 °C for different periods of time. After etching, the ink deposits were stripped by immersion in acetone with ultrasonic agitation at 25 °C for 4 minutes. The samples were examined by optical microscopy before etching, after etching and after stripping. The surface topography of the textured samples was assessed by laser interferometry.

Figure 5(a) shows a pattern of circular dots printed on a polished steel sample, representing the smallest features that could be printed in these experiments, with each dot formed by a single droplet. The diameter of the dots was ~60 μ m, in comparison with the ejected ink drop diameter of ~27 μ m. Interference fringes, discussed in more detail below, show that the dried film thickness was uneven, but the ink film nevertheless provided good protection during etching. Optimal etching behavior was found for a nitric acid concentration of 5%. The ink protected the steel surface during etching and was easily stripped by ultrasonic cleaning in acetone. Figure 5(b) shows the final textured surface, in which a slight undercutting beneath the mask is detected. The diameters of the unetched islands remaining after etching were ~50 μ m, suggesting that the extent of the undercutting was ~5 μ m per edge.



(a) (b) **Figure 5**. Individual dots printed on polished steel; (a). after printing; (b). after etching and stripping.

Figure 6 shows that for an etching time of 5 minutes in 5% nitric acid the dimensions of the texture were regular, with the depths of the etched features being \sim 3 µm.

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Figure 6. Surface topography sample printed with dots after etching and stripping; (a). 3D map; (b) line profile.

The printing of more complicated patterns was more difficult to control, because of the complexity of the ink flow on the surface. While individual dots with diameters of 60 μ m could be printed, the minimum sizes of gaps of complex shapes distributed in a continuous background was larger. Parallel linear gaps were easier to achieve and gaps as thin as 20 μ m could be obtained, as shown in Figure 7(a). Attempts to reduce the size of the unprinted gaps further resulted in distorted patterns. For square gaps, the minimum size which could be achieved without gross distortion of the shape was around 40 μ m, as seen in Figure 7(b).



Figure 7. (a) 20 µm gap between parallel printed lines; (b) 40 µm square gap.

Although a black dye-based ink was used, these thin printed films on a highly reflective steel substrate showed a clear pattern of colored interference fringes. In order to interpret the interference colors, they were correlated with quantitative surface topography measurements at the same locations by laser interferometry. The ink film thickness was evaluated from line profiles at different locations, and consistent results were obtained. The ink deposit thickness deduced in this way varied between 0.1 and 0.4 μ m. The empirical correlation between film thickness and the interference colors is shown in Table 2. After calibration, this provided a simple and reliable method to evaluate dry film thickness.

Interference color	Approximate ink film thickness (µm)
Violet	0.4
Green/Blue	0.3
Orange	0.2
Yellow	0.1

 Table 2. Correlation between ink deposit thickness and color of interference fringes

3.3 Maskless Electrochemical Texturing (MECT)

This is a simple method for texturing metallic surfaces by electrochemical machining, termed 'maskless electrochemical texturing' (MECT). The method allows a single cathode tool, in which the texture is incorporated through a pattern of perforations, to be used for many texturing operations and avoids the need for masks

to be applied to individual workpieces. It therefore has significant advantages over conventional methods of texturing by electrochemical machining.

Initial trials were successful to texture carbon steel samples, where current efficiencies of more than 90% were achieved.⁽¹³⁾ During these trial tests, the process was characterised in terms of the effects of current pulse history and electrolyte flushing conditions on current efficiency, material removal rate and feature definition.⁽¹³⁾ The variables explored during such tests were the machining time, the pulse length, the pressure of the electrolyte and the separation between the polymer mask plate and the workpiece, obtained with the use of spacers with different thicknesses.

In this paper, the texturing method developed by Costa and Hutchings⁽¹³⁾ has been optimized. Another method (micro EDM) was used to to texture the tool covers, which enabled more complex features to be produced. AISI 420 stainless steel tool covers with 0.5 mm of thickness were machined by die sinking EDM. Patterns containing regularly spaced circular dots, arrays of chevrons and parallel arrays of dashed lines with dots could be obtained. To produce the regularly spaced dots, a tungsten wire with a diameter of 110 μ m was used. To produce the regular array of chevrons, copper sheets with thickness of 100 μ m were cut to create individual chevron-like features. The dashed lines with dots were produced using the copper sheets and the tungsten wires. Figure 8(a) shows the EDM tool with the array of dashed lines and dots and Figure 8(b) shows the corresponding tool covers produced by EDM. The tool cover produced by EDM with an array of chevrons is shown in Figure 8(c).In Figure 8(d) a tool cover with an array of circular dots produced by EDM is shown. After machining the tool covers, they were covered with na insluating paint, as shown in Figure 8(e).



Figure 8. Examples of the use of EDM to machine the tool covers: (a) EDM tool with array of dashed lines and dots which produced the tool cover in (b); (c) tool cover produced by EDM with array of chevrons; (d) tool cover with array of dots before painting; and (e) after painting.



Figure 9. Example of a steel sample textured by MECT; (a) interferometric map; (b) profile accros a line of circular pockets; (c) and (d) SEM images.

3.4 Use of Immiscible Liquids

The use of two immiscible liquids is another new texturing method proposed in this work. The idea was to design an alternative novel method with improved characteristics of cost and speed that could create random textures. The principle of the technique was to create small acid droplets dispersed in oil, using a similar principle to the production of grease. The procedure would consist of immersing the surface to be etched in oil and then dispersing small acid droplets close to the surface, so that the areas of the surface in contact with oil do not etch, but the areas in contact with the acid droplets do, creating a surface texture. Various approaches were tried to form these small droplets.

The simplest approach tried involved intense mixing of oil and acid, with the objective of creating a dispersion of small acid droplets in the oil, since the two liquids are immiscible. Stearic acid was dissolved in the oil to vary the surface tension, in order to prevent the droplets coalescing. A beaker containing a mixture of 50 ml olive oil and 5 g of stearic acid was ultrasonically mixed for 6 min to dissolve the stearic acid. The beaker was immersed in boiling water, in order to help dissolution of the stearic acid. After cooling, a manual mixing procedure was used to produce the



dispersion of small droplets of acid. The oil was mixed with 25 ml of a 10% nitric acid aqueous solution. It formed a very thick mixture of oil and small dispersed acid droplets, which could be spread, like a gel, over the surface to be etched. The etching time was 3 minutes. The surface was then washed in hot water and detergent, and dried. A result obtained with this procedure is presented in Figure 10. It shows that the principle of the method works. It is very simple, fast and cheap. However, it would need to be optimised to produce more uniform craters on the surface.



Figure 10. Textured steel surface obtained by mixing 50 ml of olive oil, 4 g of stearic acid and 25 ml of nitric acid solution (10%), etching time = 3 min.

3.5 Chemical Conversion + Thermochemical Treatments

The aim of this proposed method was to develop a cheap and fast 'self-forming' texturing method. Initially, a low-carbon steel sample (0.1% C) was ground and polished. Stripes of ink with width of 500 μ m were printed on the surface. The sample was then immersed in a saturated aqueous solution of copper sulphate. With immersion, a thin copper layer was deposited on the steel surface by chemical conversion. This is a displacement reaction, where Cu ions are reduced and Fe atoms are oxidised, due to their relative position in the electrochemical series. The printed stripes were then removed by USC in acetone. After cleaning and drying, the sample was placed in a box containing a mixture of charcoal with 10 wt% of barium carbonate at 950°C for 2 h to be pack carburised. It was then quenched in oil and tempered at 200°C for 1 h.

After pack carburising, a cross section of the textured sample was prepared. After polishing with 1 μ m diamond paste, the section was etched with a 5% Nital solution. The formation of stripes of hard martensite in a softer substrate was observed, as shown in Figure 11.



Figure 11. Optical micrograph of the cross-section of two hard martensitic stripes (white etching) produced in a low-carbon steel sample.

Vickers microhardness measurements were taken along the cross section, in two perpendicular directions. The microhardness profiles, presented in Figure 12, show a hardening depth of approximately 400 μ m and a hardening width of approximately 1 mm. The large widths of the hardened zones suggest the occurrence of lateral diffusion. This makes the method unsuitable if very thin features are desired. In order to further develop the method, the relationship between minimum feature width and depth must be investigated, since sideways diffusion will occur as well as depth diffusion, as shown in Figure 13. The carburised depth should be proportional to $t^{1/2}$, where *t* is the carburising time. More sophisticated printing methods should be used

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to mask the surface, to verify the smallest obtainable size of the individual features that composes the texture. Also, the formation of a texture on these surfaces in use, due to the difference in hardness between the carburised and non-carburised areas, should be investigated.



(a)

(b)

Figure 12. Vickers microhardness profiles for carbon-enriched stripes in a steel surface; measurements along: (a) the depth of the stripes; (b) the width of the stripes.



Figure 13. Hypothetical carburised region contours for increasingly longer carburising times ($t_1 < t_2 < t_3$).

4 CONCLUSIONS

Texturing methods were identified and classified in groups and subgroups, according to their physical principles. This included not only methods already existent either in industrial practice or in the literature, but also new possible methods that could be used to texture surfaces.

Several surface texturing methods were investigated in varying detail, in order to explore their viability, main characteristics, potential and limitations.

Although PCT is widely used in the electronics industry, the approach used in this work was much simpler and cheaper. It did not require the use of a clean room. The spinner was adapted from a variable-speed rotating motor. A conventional microscope light was used as the UV source for the exposure of the resist. Archive microfiche film was used to make masks.

MECT is a simple, cheap and fast texturing method. No masking of the surfaces to be textured is required. It involves the application of a pulsed voltage to the

electrochemical cell. The off-pulse time was shown to be essential to assist the flushing of the reaction products. Large gaps between tool and workpiece allowed good flushing, generating clean textured areas. In addition to textured patterns containing circular pockets, patterns with more complex features such as interrupted lines and chevrons could be produced.

A new technique involving texturing by small acid droplets dispersed in oil was proposed. In principle it is very simple and cheap, but can only form patterns containing a random arrangement of circular pockets.

Another new technique involved the localisation of carburising to certain areas of a steel surface, which became harder. A simple approach for the technique was implemented to generate hard stripes on a softer surrounding material. Lateral diffusion led to stripes significantly wider than the initial stripes painted on the surface.

Using a solvent-based ink and drop-on-demand ink-jet printing, patterns of circular spots and more complex masking patterns were successfully printed on to steel surfaces, which were then etched to produce textured surfaces. Spot sizes of 60 μ m were readily achieved on polished substrates. Printing of complex shapes was more difficult to control due to the flow of the ink on the polished sample. The thinnest parallel gap was ~20 μ m and the smallest square gap ~40 μ m across. Interference colors on the dried deposits of ink were correlated with thickness measurements by white-light interferometry. Film thicknesses were in the range from 0.1 to 0.4 μ m.

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