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Precise fabrication of microtextured stainless steel surfaces using metal injection moulding

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ABSTRACT

In this study, we show a new, cost effective and straightforward method to fabricate stainless steel (SS) microtextures with various geometries. We were able to design and texture a durable Ni mould insert by a microworking robot technique. Furthermore, the obtained microtextures were replicated onto stainless steel surfaces by metal injection moulding. A computer-controlled program enabled precise control of the spacing between the textures giving a broad choice for surface design. Surface geometries, such as micropillars and micropits with different dimensions, were produced on planar and curved surfaces. These results introduce a new platform for mass production of microtextured stainless steel surfaces with high surface control. The obtained surfaces have potential applications by meeting the demands of reducing surface contact and providing surface protection from mechanical damage.

1. Introduction

Stainless steel materials have excellent mechanical properties; for instance, precipitation hardened 17-4PH stainless steel exhibits an excellent combination of high strength, hardness, and corrosion resistance, making it a reasonable material to use in aerospace, the automotive industry, and nuclear reactor components [1–3]. Austenitic 316L stainless steel is less durable; however, it is still, corrosion resistant, ductile, tough, and meets the needs of a wide range of applications, for example, in marine components and consumer products [4]. Both 17-4PH and 316L stainless steels possess a great biocompatibility, which make them a significant material resource for fabrication of medical devices, for instance, in orthodontics and joint implants [5,6]. Moreover, 316L and 17-4PH stainless steels are well known for their ease in processing, which makes them ideal candidates for research, such as in surface engineering, particularly, in the fabrication of micrometer-scaled structures.

Surface engineering and miniaturization of stainless steel materials have become an important area in material research due to the ability to control their surface properties in certain application purposes, for example, in medicine [7], anti-biofouling [8], self-cleaning surfaces [9], tribology [10], etc. For example, S. Zouaghi et al. fabricated micro-structured SS surfaces by using femtosecond laser ablation and obtained an antifouling effect under industrial diary pasteurization conditions [11]. Microtextured microgrooves on stainless steel surfaces can affect the tribological properties of SS [12–14] and can also be used in microfluidic devices [15]. The methods used to fabricate the functional miniaturized stainless steel parts in a small production batch are represented by mechanical machining methods, such as laser texturing, micromilling, and electrically discharged machining (EDM). Laser surface microtexturing of SS has emerged as the most recently developed method to create microtextured surfaces [16–18] due to its high precision and simplicity of use. However, laser textured surfaces have relatively high surface roughness, and moreover, the method has limitations in regard to geometric flexibility and it is time-consuming. Micromilling is a relatively faster method than laser texturing; however, the surface tends to be lacking in quality [19]. EDM is an alternative method to successfully fabricate microtextures with complex shapes and very high aspect ratios. However, the method is slow, and the resulting surface roughness is high. Moreover, the method is limited to only electrically conductive materials and can be a potential fire hazard because of the use of oil-based dielectrics [20].

Recently, metal injection moulding (MIM) has reached a remarkable technological level and has been introduced into the market for producing precision moulded microparts due to its simplicity and ability to be used in large batch production. Among the various net shape
manufacturing technologies in powder metallurgy, MIM techniques have a broad range of material choices, low cost, and the possibility to create various textures and complex geometries by simple replication. Moreover, the MIM technique has minimal material loss and a high sintering density of the parts [21]. Nevertheless, in order to succeed in the replication processes, preparing the mould insert requires sufficient accuracy and precision. The abovementioned mechanical methods of surface texturing can be used for preparation of the mould insert and further utilized in the injection moulding process; however, the quality of the mould inserts tends to be low. In this sense, the so-called lithography-based LIGA (lithographie graphik abformung) [22] process allows for fabrication of complex and reliable moulds. Thus, Nishiyabu et al. produced micropillar-structured 316L parts with a high precision by combining the LIGA method with MIM [23]. Nevertheless, a microfabrication method with combined lithography and metal injection moulding techniques requires additional preparation steps of a sacrificial plastic resist, which is complex and time-consuming. Therefore, a method for preparation of the mould insert in only one fabrication process is in a high demand to ensure the simplicity of the manufacturing process.

In the past few decades, surface modifications of the mould insert with a robotically controlled technique was developed in our research group [24,25]. The robotic technique was effectively used for fabrication of Al and Ni mould inserts with various surface geometry designs starting from simple one-levelled microtextures and developing into hierarchical textures. These textured mould inserts have been used in plastic injection moulding for replicating the topographies on plastic surfaces. The prepared plastic surfaces have precise controlled topographies and have been proven to be effective in controlling surface wettability and friction and their functionalization [26–28]. The simplicity and low cost of the method inspired our research group to further expand the fabrication of microtextures not only on plastic surfaces but also on stainless steel material.

In this study, we show the possibility of fabricating microtextures on 17-4PH and 316L stainless steel surfaces in a straightforward and inexpensive way, allowing for mass production either on planar or curved surfaces. The aim is to assure that with a computer-controlled microworking robot with high precise control we are able to create microstructures on the Ni foil, which can be used as a mould insert for further injection moulding processes. We probed different positions of the microtextures to show a variety of geometries that can be produced, and we calculated the surface coverage of the micropillared surface compared to the smooth surface. Another aim is to demonstrate that combining microtexturing techniques with metal injection moulding provides a new, simple mass production approach to create stainless steel microtextures.

2. Experimental

2.1. Fabrication of microtextured stainless steel surfaces

Fig. 1 shows a schematic representation of the fabrication process of SS microtextures, using, as an example, a rectangular shaped mould insert giving two variations of microtextures, micropillars, and micropits.

2.1.1. Ni mould insert preparation

Ni foils (99, 99%) with a thickness of 0.25 mm were purchased from Good Fellow, England and were used as a mould insert for further injection moulding processes. The microtextures were directly patterned on the Ni foils (Fig. 1 (a)) using a computer controlled RP-1AH microworking robot technique (Mitsubishi Electric) with the CR1 control and a feedback unit from Delta Enterprise, Ltd. The round shaped tungsten carbide working needles (Fodesco, Ltd.) with a tip diameter of 100 and 200 μm were assembled in a robot arm. The shape and size of the needle’s top diameter controlled the micropit design. The impact force of the needle controlled the depth of the microtexts. The working speed of texturing was 1000 mm/s. The arrangement of the micropatterns was controlled by manipulating distances between the microtexts. The microtexts were positioned in two different arrangements, square and hexagonal lattices. Furthermore, by controlling the position of the microtexts, microtextures of various sizes were produced. Both rectangular and circular shaped mould inserts were fabricated.

2.1.2. Metal injection moulding

Two different stainless steel granulated feedstocks were used in the metal injection moulding, including precipitation hardened 17-4PH SS and austenitic 316L SS (PolyMIM GmbH, Germany). A HAAKE® MiniLab II micro compounder (Thermo Fisher Scientific) was used for the MIM process. In the injection moulding process (Fig. 1 (b)) the Ni mould insert containing the designed microtexts was placed in the rectangular/ circular mould cavity, replicating the green part with micropillars. The feedstock granules were injected with a pressure of 450 bar and an injection time of 8 s for the rectangular mould cavity. For injection moulding of circular specimens, a pressure of 450 bar and an injection time of 4 s were used.

After the injection moulding, the green part was subjected to solvent debinding, drying, and sintering processes (Fig. 1 (c)). Debinding was
conducted in a distilled water bath at 60 °C for 10 h followed by further drying in the oven for 2 h in ambient conditions. The obtained brown part was sintered under hydrogen in the sintering furnace Carbolite GERO, model HTK 8 MO/16 (Carbolite Gero Ltd., United Kingdom). The sintering cycle for 316L SS was RT - 600 °C with 2 h holding time, 600–1380 °C for 8 h hold, and cooling from 1350 to 80 °C for 2 h. For 17-4PH the sintering cycle was RT – 600 °C with 2 h holding time, 600–1380 °C for 8 h hold, and cooling from 1350 to 80 °C for 2 h.

2.1.3. Stainless steel as a mould insert

Furthermore, the sintered SS surfaces with micropillars were used as mould inserts to reproduce the micropits (Fig. 1 (d)). For that, the SS containing micropillars was attached to the mould cavity followed by conventional injection moulding with the same parameters as used for fabrication of the 17-4PH SS samples. After the injection moulding, the green part with micropits was obtained. Then, debinding, drying, and sintering (Fig. 1 (e)) was applied, producing a SS substrate containing micropits.

2.1.4. Characterisation

Scanning electron microscope (SEM) images were captured with a Hitachi S4800 field emission SEM microscope. Acceleration voltages of 3–7 kV and an emission current of 5–10 A were used, and the working distance was 8 mm.

3. Results and discussion

3.1. Microtextured stainless steel samples

With the microworking robot texturing technique, it is possible to fabricate a mould insert with fine micropits for further metal injection moulding. A key to the successful fabrication of the final SS microtextures is complete filling of the metal powder feedstock within very small and narrow micropits. The feedstock viscosity and the powder particle size have a major influence on the complete filling of the feedstock within the micropits. Thus, the smaller the metal powder size, the smoother the surface and the better the surface roughness after the sintering. Therefore, to ensure a sufficient surface roughness, the choice of a small particle size is preferred. Typically, the powder size for MIM should be below around 20 μm [29]. Moreover, in order to fill cavities with size of 100 and 200 μm, the powder size should be sufficiently less than the size of the micropit. In our case, the D80 of the powder was 22 μm, and hence, it meets the requirements regarding the choice of powder size. Likewise, a complete demoulding of a green part from the mould insert permits creation of a sufficient aspect ratio of the micropillars during the replication. In this case, the choice of the mould insert material is crucial. Thus, for easy and complete demoulding, the feedstock should have low affinity for the mould insert material so that the feedstock does not stick to the mould surface. Moreover, the mould insert should possess high tensile strength to avoid mechanical damage. In this sense, the Ni foil, which was used for the microrobot surface texturing, successfully fulfilled the requirements for efficient replication of fine micropillars. For comparison, in addition an Al foil was also tested, which demonstrated incomplete demoulding of the green part from the micropits.

Fig. 2 (a1) shows the SEM image of the micropillars of the green part after the debinding and drying processes and its close-up image (Fig. 2 (b1)) and SS micropillars after the sintering (Fig. 2 (a2)) and its close-up image (Fig. 2 (b2)). The needle size of the microworking robot structuring was 200 μm and the hexagonal lattice was designed.

Fig. 2 (a1) and Fig. 2 (b1) shows the uniform micropillars, which proves complete filling of the feedstock with the metal feedstock and successful demoulding of it from the 200 μm-sized Ni mould micropits. Furthermore, it can be seen (Fig. 2 (b1)) that the micropillar has shape of the truncated cone and the size of the micropillars differs between the top and the bottom of the pillar. Thus, the micropillar fabricated with the 200 μm sized needle has an average size of 215 μm at the top and 240 μm on the bottom. The bottom size of the micropillar is controlled by the impact force of the needle when the needle hits the Ni foil. The larger the impact force of the needle, the deeper the micropits, and thus the larger the bottom size of the micropillar.

A sintering process is a crucial step for the creation of a solid stainless steel material. During the sintering, mass transport of the particles occurs, transforming them into grain boundaries and eliminating the pores between the particles. The pore elimination causes a shrinkage of the material, and therefore, results in a reduction of the specimen size. Thus, after the sintering (Fig. 2 (b2)), the average size at the top is 170 μm and on the bottom the size is 205 μm, which is smaller than the sizes of the micropillar before the sintering. The shrinkage of the sintered micropillars was calculated from parameters collected from 10 random micropillars, giving approximately 20% of shrinkage (calculated from a green part).

3.2. Variation of the microtextured stainless steel samples

Fig. 3 shows a schematic representation of the fabricated stainless steel surface with micropillars, which have square and hexagonal lattice arrangements, and their parameters.

In applications when there is a need for reducing the surface contact area with the counter body, for example in applications in microgears, surface microtexturing is an effective solution. The approximate surface fraction of the surface, which is covered with the micropillars is called a surface coverage (SC) and it can be calculated with the following equations for both the square and hexagonal lattice arrangements. As demonstrated in Fig. 3, W is the distance between the micropits and T is
Table 1
Designed and measured surface parameters.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Designed parameters</th>
<th>Measured parameters, after sintering</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Arrangement</td>
<td>Top diameter T (μm)</td>
</tr>
<tr>
<td>s1</td>
<td>square</td>
<td>100</td>
</tr>
<tr>
<td>s2</td>
<td>square</td>
<td>100</td>
</tr>
<tr>
<td>s3</td>
<td>square</td>
<td>200</td>
</tr>
<tr>
<td>s4</td>
<td>hexagonal</td>
<td>100</td>
</tr>
<tr>
<td>s5</td>
<td>hexagonal</td>
<td>100</td>
</tr>
<tr>
<td>s6</td>
<td>hexagonal</td>
<td>200</td>
</tr>
</tbody>
</table>

Fig. 4. SEM images of sintered 17-4PH stainless steel round shaped micropillars with a structure size of around (a1-b2) 100 μm sized with (a) low density and (b) high density, and (c) 200 μm with (1) square and (2) hexagonal arrangements, (d) square shaped micropillars with protective pillars, and (e) round shaped micropits.
the top diameter of the micropillar [27].

\[
SC_{sq} = \frac{\pi}{4} \frac{T^2}{W}
\]

(1)

\[
SC_{hs} = \frac{\pi}{2\sqrt{3}} \frac{T^2}{W}
\]

(2)

To reveal the influence of the designing parameters, which are the micropillar size, density of the micropillar and pattern lattice arrangement, the SC coverage values were calculated for both the designed and fabricated SS micropillars. The SC was calculated for the micropillars, which were fabricated with a 100 μm sized needle, varying the density of the micropillars and lattice arrangement, and for micropillars with the size of a 200 μm with various lattice arrangements. Table 1 shows the parameters of the designed microtextured surfaces with the SC value and the measured parameters after the sintering process and their SCs.

It can be seen from Table 1 that the SC reduces after introduction of the micropillars, considering that SS sample without micropillars has SC value equal to 100%. Moreover, the shrinkage of the micropillars caused by sintering additionally reduced the size of the microtextures, giving a difference in actual SC values compared to the designed micropillars. The hexagonal lattice arrangement gives a higher SC value compared to a square lattice arrangement.

The microworking robot needle size ranges from 5 μm to 500 μm, however, allows for the possibility of creating various microtextures sizes. However, when choosing a structuring needle, various factors should be taken into account. For example, texturing with a small sized needle, such as 5 and 10 μm, on the tough Ni foil, causes needle breakage. In addition, microtexturing with a large needle size, such as 500 μm, does not create enough local impact force to create a micropit. Thus, microtexturing with the 100 μm and 200 μm sized needles ensures the formation of fine micropillars.

Fig. 4 shows the SEM images of the variations of the microtextures that are possible to be manufactured using the microworking robot technique. Thus, Fig. 4 (a) and Fig. 4 (b) show SEM images of the 17-4PH micropillars fabricated with round shaped needle with size of 100 μm and Fig. 4 (c) demonstrates similar micropillars prepared with 200 μm sized needle. Fig. 4 (d) shows a SEM image of the complex-levelled surface textures fabricated with a 200 μm sized square shaped needle. Likewise, we demonstrated that the obtained micropillars can also be used as a mould insert. Fig. 4 (e) shows the SEM images of the SS micropits replicated from the SS mould insert containing round shaped micropillars.

It can be seen from Fig. 4 (a–c) that it is possible to fabricate various micropillars from a Ni mould insert containing 100 μm and 200 μm sized micropits, varying texture densities and arrangement. The micropillar-like textures significantly reduce the contact area with the counter body in a controlled way, which suggests potential applications in controlling surface friction between sliding materials. Apart from their tribological properties, these microtextured stainless steel substrates serve as a new platform for fabricating of substrates with controlled wettability, such as superhydrophic and ice-repellent surfaces. Moreover, microtextured SS substrates can act as durable substrate materials for further chemical and physical modifications, for example, deposition of inorganic materials, for the preparation of smart surfaces and interfaces with advanced surface properties.

Furthermore, in addition to single-levelled and single-sized topographies it is possible to replicate hierarchical surface topographies (Fig. 4 (d)). Fabrication of various levels can be performed by adjusting the impact force of the needle on the Ni foil either using the same program or by matching with another program and superimposing the positions of the designed coordinates. The hierarchy of the microtextures provides protection from mechanical wear and environmental damage.

Micropits (Fig. 4 (e)) have potential applications to act as reservoirs for both solid and oil lubricants for low friction applications [13,14]. Moreover, micropits can act as a protective vessel for locating and growing cells and various particles, and can be used as protection from environmental damage. In addition, micropits on a metallic support can be used as a corrosion resistant durable microreactor for loading of catalysts and transformation of exhaust gases [30].

Furthermore, while most of the conventional methods to prepare mould inserts are only available for manufacturing microtextures in lateral dimensions, with the use of these Ni mould inserts it is possible to fabricate not only planar but also curved shaped surfaces. Thus, Fig. 5 shows a photograph (Fig. 5 (a)) of a curved 316L SS containing micropillars and its SEM image (Fig. 5 (b)).

It can be seen from Fig. 5 that the quality of the replicated micropillars is relatively good. The filling of the feedback within the Ni mould micropits was complete. Therefore, the microtextured Ni mould insert can be used not only for fabrication of planar surfaces but also for surfaces with a curvature. In applications demands, for example, in bearings that have curved surfaces, these microtextures can reduce the surface friction between the moving parts.

The microworking robot texturing technique for mould insert fabrication has similarities with the LIGA lithographic technique, a fabrication process that includes complex highly specialized individual steps such as wafer assembly, PMMA resist exposure, and electrochemical deposition. Therefore, the LIGA fabrication technique is very time consuming and requires complex manufacturing facilities. With the proposed microworking robot texturing technique, it is possible to
fabricate mould inserts with only a single step. Moreover, the computer control facilitates flexibility and a variety of a surface designs. Hence, the microworking robot structuring technique opens a new approach for fabrication of miniaturized devices in a low cost and simple way.

4. Conclusions

In this study, we have demonstrated a new approach to fabricate various microtextured surfaces by combining Ni mould insert texturing with the metal injection moulding technique. With the computer controlled microworking technique, we were able to texture the Ni mould insert in one simple step. Micropillars, micropits, and multilevel micropits were prepared. The obtained surfaces have potential applications such as reducing surface contact and surface protection, thus enabling the production of low-cost and self-lubricating metal surface systems. The method is suitable for processing not only stainless steel feedstocks but also other injection mouldable materials, for example, hard metals, or even fabrication of multicomponent materials.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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