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ADVANCED MANUFACTURING

A Study on the Surface Chemistry of Laser Textured Parts

The research investigates the changes in the surface properties and surface chemistry following laser microtexturing of stainless steel parts, in relation to their applications in the energy sector. In particular, the material compositions of the laser surface textured (LST) parts, together with the oxide compound formation on them are evaluated with respect to their wettability property. Wettability is crucial for heat exchange processes as it affects the efficiency of heat transfer by influencing surface contact and the formation of droplets on the heat exchanger surface. Here, two simple LST geometries, viz. channel and cross-hatch, were produced using a nanosecond fibre laser micromachining system. The wettability of the textured surfaces was then examined over a period of 45 days with an interval of 15 days. It was observed that the former geometry rendered a hydrophilic surface initially, which transformed to a hydrophobic surface after 45 days, whereas the latter LST design exhibited hydrophobic characteristic over the entire duration of the assessment. The material compositions and the oxide compound formation on the LST parts were analysed via energy dispersive spectroscopy, X-ray photoelectron spectroscopy and X-ray diffraction techniques and the results were correlated with the measured wettability data.

Keywords:
Microtexturing, laser manufacturing, micromachining, wettability, surface chemistry.

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INTRODUCTION

The British Petroleum Statistical Review of World Energy 2021 highlights the need of increasing renewable energy production by ~50% before 2050 [1]utilization, and storage; and nuclear power development. We used the annual ‘British Petroleum statistical review of world energy 2021’ report as our primary database. Globally, fossil fuels, renewable (primarily hydro, wind and solar. A viable approach to reach this goal is via reduction of energy consumption. In regard to the energy sector, microtextured surfaces can offer a viable passive mechanism for decreasing energy consumption by reducing drag and harnessing greater heat recovery in a turbulent and/or humid environment [2, 3]biomedical, transportation and aerospace sectors. In relation to the energy sector, microtextured surfaces provide an energy efficient and cost-effective passive mechanism for increased heat transfer during matter’s phase change in energy recovery systems. This study explores the viability of laser microprocessing as an attractive manufacturing route for generating textured surfaces and compares with the results from a previous study involving microgeometries created by micro-wire electro discharge machining (µWEDM.

One innovative approach to generate renewable energy from wind, gales, hurricanes, etc, is via developing High-Peak Perishable Energy Recovery systems that are capable of enhancing condensation heat transfer [4]. Such systems can be developed by producing microtextured surfaces on the components using various micromanufacturing techniques, e.g. electro-discharge machining, chemical machining and laser surface texturing (LST). The microtexture geometries, such as ridges grooves, and dimples can enhance the thermal capacity of the parts under isothermal fluid flow [5]. In wind energy applications, microtextures can improve the aerodynamic performance of wind turbine blades and increase the efficiency of energy generation [2]. As a passive energy-harvesting technology that requires no additional energy input or moving components, use of microtextured surfaces represent a promising pathway toward a sustainable future for the energy sector [6].

Over the past decade, manufacture of the microtextured surfaces has witnessed rapid growth. The correlation between microtextures and the boundary layer on the surfaces under turbulent fluid flow conditions has demonstrated the potential of minor surface modifications to significantly impact the fluid dynamics [7]. The primary aim of these microtextures is to alter surface properties, including wettability properties that can also be influenced by the surface chemistry [8, 9].

Previous research has proposed and analysed various computational fluid dynamics (CFD) software tools to identify the most effective simulations based on channel designs; however, the effects of the surface chemistry on the surface properties of the textured parts has not been considered [3]. Other studies have investigated the short- and long-term wetting behaviour of laser-textured stainless steel surfaces using femtosecond pulses and normal pulse mode, with applications in self-cleaning and anti-icing through homogeneous hydrophobic chemical modification [10]. The relationship between the surface microtexture and surface chemistry remains a topic of debate, as researchers strive to achieve desired wettability. Investigations into the surface chemistry of 2507 super duplex stainless steel in aerated, deaerated, and acidified artificial seawater have shown that the percentages of oxidised Cr, Fe(II), and hydroxides increase while film thickness decreases with acidification [11]. A deeper understanding of the interactions among dispersive and non-dispersive chemical properties is still imperative [12]. A thorough analysis of the chemical compositions of the microtextured surfaces can enhance and contribute to the development of innovative surface modification techniques for other sectors, such as biomedical, marine, aerospace and automotive sectors [13, 14].

MATERIALS AND METHOD

Laser surface texturing was carried out on 316L stainless steel samples, of 35 mm × 35 mm area and 2 mm thickness, on a Lasertec DMG-40 nanosecond fibre laser system.

To achieve a uniform and smooth surface prior to the laser texturing, the samples were cleaned with acetone and isopropyl alcohol, followed by drying in air. The nominal dimensions of the LST geometries are shown in Fig. 1 (a) while the CAD designs of the Channel and the Cross-hatch textures are displayed in Figs. 1 (b) and 1 (c), respectively. A 0.25 mJ laser energy at 80 kHz frequency were used to produce the textures, together with a 800 mm/s beam scanning speed and 100 µm hatch distance. 50 scanning passes were given along machine X-axis to generate the Channel texture, whereas 50 passes were utilised along both X and Y-axes to produce the Cross-hatch pattern. The nominal laser beam diameter in all cases were fixed to 32 µm.

The surface wettability tests were carried out by dropping 10 µL of water from a syringe on the LST surfaces. The surfaces’ wetting property was assessed over a duration of 45 days, with an interval of 15 days. Thus, the contact angles of the droplets were measured on Day 0, Day 15, Day 30, and Day 45, by using ImageJ software.

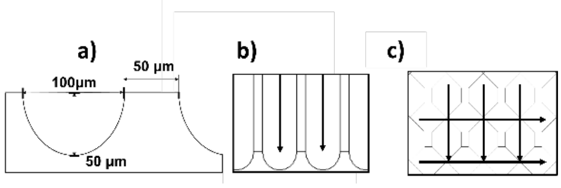

Fig. 1. (a) Texture dimensions, (b) Channel and (c) Cross-hatch designs.

The changes in the material compositions and the oxide compound formations on the LST surfaces over the duration of 45 days were analysed using different methods such as energy dispersive spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS) and X-ray diffraction (XRD). A Thermo Fisher Scientific K-alpha+ spectrometer was utilised to conduct the XPS analysis. Samples were examined using a micro-focused monochromatic Al X-ray source (72 W) in the ‘400-micron spot’ mode, where an analysis defining an elliptical X-ray spot of ~400 µm × 600 µm was provided. The data was captured with pass energies of 150 eV for survey scans and 50 eV for high resolution scans, with step sizes of 1 eV and 0.1 eV, respectively. Data analysis was conducted using CasaXPS v2.3.24 software [15]discussions arising from a series of workshops have been a significant source for developing the overall XPS data processing concept and are the motivation for creating this work. These workshops organized by the Institut des Matériaux Jean Rouxel (IMN. The quantification was determined utilising a Shirley type background and Scofield cross-sections with an electron energy dependence of -0.6. XRD analysis was carried out using a Siemens D5000 system using a CuKα X-ray target, from 20° to 90°, with a step angle of 0.02° and a scan step time of 1 s.

RESULTS AND DISCUSSION

The EDS analysis reveals that the LST specimens were prone to oxidation as they were produced under atmospheric conditions. As seen from Table 1, the oxygen content increased to 9.6 weight% in the Channel textured regions on Day 0, from a 5.3 wt% of oxygen content in the untextured areas. The same raised to 8.61 wt% in the Cross-hatched textures from 4.11 wt% in the untextured fields. The data also showed that the Fe content decreased in the textured areas, possibly due to the increased oxygen content. The higher oxygen content in the textured areas is attributed to the recast materials. Oxygen content was found to be less than 0.01% at the bottom of the Channels and 0.48% at the intersection of the grooves in the Cross-hatch design. However, the oxygen contents were ~24.54% and ~19.60% at the top of the riblets in the Channel and Cross-hatch textures, respectively. By comparing the chemical compositions of the Channel and Cross-hatch LSTs between Day 0 and Day 15, only minor changes were observed, except for the carbon content, which increased by over 1% in the Channel textures, but by only 0.46% in the Cross-hatch textures after 15 days.

	C	O	Fe	Si	Cr	Mn
Channel Day 0	5.77	9.60	57.87	0.34	16.02	1.38
Channel Day 15	6.89	9.86	57.80	0.36	15.79	1.39
Cross-hatch Day 0	4.13	8.61	60.35	0.33	16.93	1.76
Cross-hatch Day 15	4.59	8.02	60.53	0.34	16.76	1.62

Table 1. EDS results in wt% on the Channel and Cross-hatch textures after Day 0 and Day 15.

The SEM images of the textures are shown in Figs. 2 and 3. For the Channel specimens, the method of manufacturing was by removing layers along the X-axis, whereas for the Cross-hatch, the laser scanning was repeated along the Y-axis after completing scanning along the X-axis. This resulted in deeper craters in the Cross-hatch pattern, see Fig. 4.

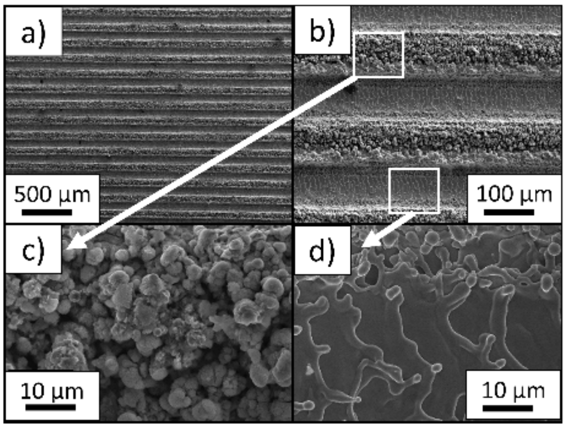

Fig. 2. SEM images of the Channel texture: Day 0.

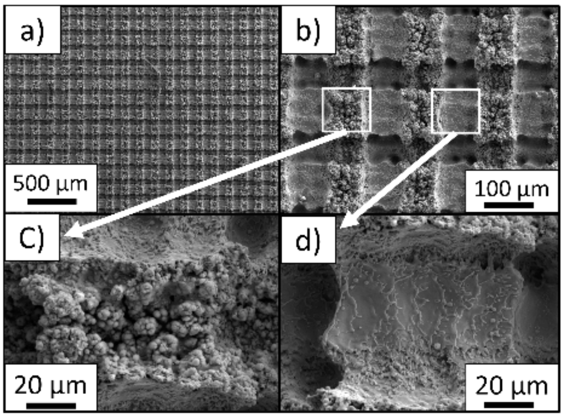

Fig. 3. SEM images of Cross-hatch texture: Day 0.

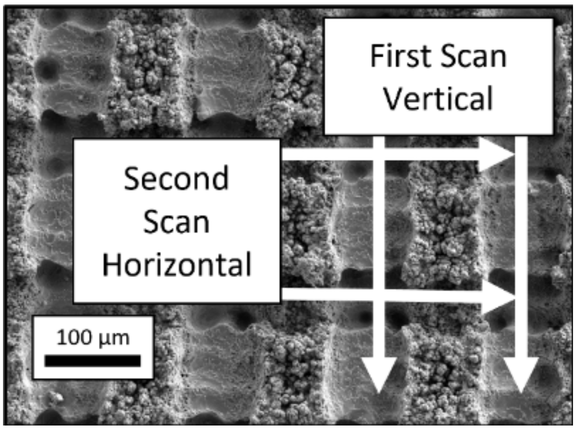

Fig. 4. Cross-hatch crater merge.

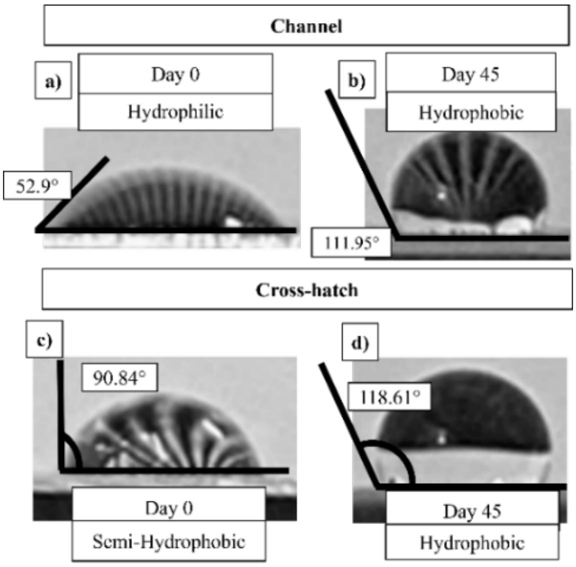

Fig. 5. Wettability contact angle results.

The images of the water droplets on the LST surfaces after Day 0 and Day 45 are shown in Fig. 5, together with the corresponding contact angles obtained via the ImageJ software. The changes in the measured contact angles over the period of 45 days are displayed in Fig. 6 overleaf. It is found that the contact angles for both textures increased considerably, by ~111.34% and ~30.57% for the Channel and Cross-hatch patterns, respectively.

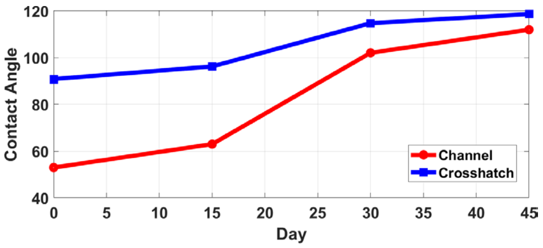

Fig. 6. Contact angles measured after Days 0, 15, 30, and 45.

The XRD spectra obtained from both Channel and Cross-hatch textures are shown in Fig. 7. The results show that iron (Fe), as well as the presence of Fe_2O_3 and Fe_3O_4, is in agreement with the EDS results, which indicates surface oxidation.

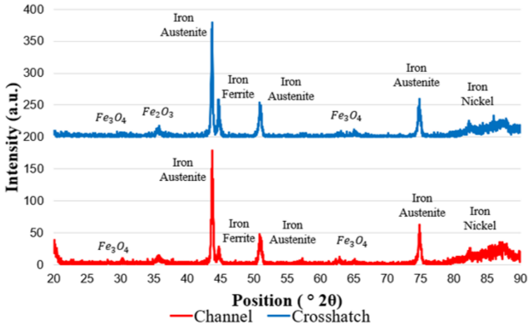

Fig. 7. XRD phase analysis on the Channel and Cross-hatch LST specimens.

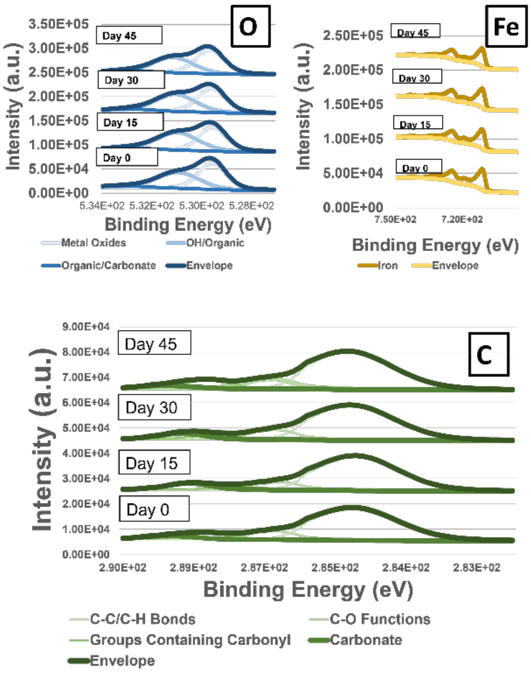

Fig. 8. XPS analysis of oxygen (O), iron (Fe) and carbon (C) on the Channel LST specimen.

The XPS results are on the Channel design are shown in Fig. 8. Possibly the minor variation in the percentage of carbonyl groups attributed to the change in the wettability property of the Channel textures, i.e. from hydrophilic after Day 0 to become hydrophobic after Day 45.

CONCLUSIONS

The paper reports material composition and surface chemistry analysis of LST parts over a duration of 45 days after texturing. The effects of these two factors on the wettability property of the textured surfaces are evaluated. The data suggests that surface oxidation as well as the presence of certain carbon-based compounds (in particular, carbonyl groups) might play a crucial role in determining the hydrophilicity or hydrophobicity of the textured surfaces. Future work will involve an in-depth study investigating the effects of other elements present in the material, such as Cr, Mo, Ni, Mn and N, on the wettability property. A deeper insight in understanding of how surface chemistry changes via laser texturing will immensely aid in designing bespoke textured surfaces with controlled surface and material properties.

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Conflicts of interest

The authors declare no conflict of interest.

REFERENCES

[1] J. L. Holechek, H. M. E. Geli, M. N. Sawalhah, and R. Valdez, ‘A Global Assessment: Can Renewable Energy Replace Fossil Fuels by 2050?’, *Sustainability*, vol. 14, no. 8, p. 4792, Jan. 2022. doi.org/10.3390/su14084792

[2] H. Martinez-Zavala, D. Bhaduri, P. Petkov, A. Valera-Medina, and S. Bigot, ‘Effects of laser microtextured surfaces in condensation heat transfer’, *Procedia CIRP*, vol. 95, pp. 927–932, 2020. doi.org/10.1016/j.procir.2020.01.184

[3] A. Yarden Escudero-Ornelas, D. Bhaduri, H. Martinez-Zavala, A. Valera-Medina, and S. Bigot, 'On the performance evaluation of microtextured surfaces using computational fluid dynamics: A comparative study', *World Congress on Micro and Nano Manufacturing - WCMNM 2022*, Leuven, Belgium.

[4] H. Martinez Zavala, 'High peak, perishable energy recovery – foundation phase', PhD thesis, Cardiff University, 2022.

[5] A. J. Mazaltarim, J. J. Bowen, J. M. Taylor, and S. A. Morin, ‘Dynamic manipulation of droplets using mechanically tunable microtextured chemical gradients’, *Nat Commun*, vol. 12, no. 1, p. 3114, May 2021. doi.org/10.1038/s41467-021-23383-7

[6] A. Sharma, D. Marla, S. S. Joshi, and R. Bathe, ‘A Study of Femtosecond Laser Processed Microtextures on Silicon Wafers to Enhance Optical Absorption’, *Lasers Manuf Mater Process*, vol. 9, no. 3, pp. 277–291, Sep. 2022. doi.org/10.1007/s40516-022-00176-4

[7] M. A. H. Al-fahham, S. Bigota, and A. Valera Medina, ‘A Study of Fluid Flow Characteristics Using Micro Structured Surfaces Produced by WEDM’, in *4M/IWMF2016 The Global Conference on Micro Manufacture : Incorporating the 11th International Conference on Multi-Material Micro Manufacture (4M) and the 10th International Workshop on Microfactories (IWMF)*, 2016, pp. 247–250. doi.org/10.3850/978-981-11-0749-8_708

[8] F. Schell, S. Alamri, T. Steege, C. Zwahr, T. Kunze, and A. Lasagni, ‘On the wetting behavior of laser-microtextured stainless steel using Direct Laser Interference Patterning’, *Surface and Coatings Technology*, vol. 447, p. 128869, Oct. 2022. doi.org/10.1016/j.surfcoat.2022.128869

[9] S. Y. Misyura, G. V. Kuznetsov, D. V. Feoktistov, R. S. Volkov, V. S. Morozov, and E. G. Orlova, ‘The influence of the surface microtexture on wettability properties and drop evaporation’, *Surface and Coatings Technology*, vol. 375, pp. 458–467, Oct. 2019. doi.org/10.1016/j.surfcoat.2019.07.058

[10] G. Giannuzzi *et al.*, ‘Short and long term surface chemistry and wetting behaviour of stainless steel with 1D and 2D periodic structures induced by bursts of femtosecond laser pulses’, *Applied Surface Science*, vol. 494, pp. 1055–1065, Nov. 2019. doi.org/10.1016/j.apsusc.2019.07.126

[11] Z. Cui *et al.*, ‘Passivation behavior and surface chemistry of 2507 super duplex stainless steel in artificial seawater: Influence of dissolved oxygen and pH’, *Corrosion Science*, vol. 150, pp. 218–234, Apr. 2019. doi.org/10.1016/j.corsci.2019.02.002

[12] A. Samanta, Q. Wang, S. K. Shaw, and H. Ding, ‘Roles of chemistry modification for laser textured metal alloys to achieve extreme surface wetting behaviors’, *Materials & Design*, vol. 192, p. 108744, Jul. 2020. doi.org/10.1016/j.matdes.2020.108744

[13] C. Hu, D. Ashok, D. R. Nisbet, and V. Gautam, ‘Bioinspired surface modification of orthopedic implants for bone tissue engineering’, *Biomaterials*, vol. 219, p. 119366, Oct. 2019. doi.org/10.1016/j.biomaterials.2019.119366

[14] S. Arango-Santander, ‘Bioinspired Topographic Surface Modification of Biomaterials’, *Materials*, vol. 15, no. 7, p. 2383, Jan. 2022. doi.org/10.3390/ma15072383

[15] N. Fairley et al., ‘Systematic and collaborative approach to problem solving using X-ray photoelectron spectroscopy’, *Applied Surface Science Advances*, vol. 5, p. 100112, Sep. 2021. doi.org/10.1016/j.apsadv.2021.100112

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