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Scanning Probe Microscopy for roughness analysis of complex shape samples

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1. Introduction

The continuous technological progress presently experienced by many fields of engineering is posing new challenges for the development of diagnostics suitable for process and product assessment.

An interesting example is represented by the fabrication of fuel injector nozzles in the automotive industry. The increase of engine efficiency and the forthcoming stringent regulations aimed at reducing pollution require an unprecedented accuracy in defining the geometry of the nozzle [1]. While diameter and size can be effectively controlled in the machining stage, other details which can strongly affect the fluid-dynamics of the fuel injection such as, the edge angles and the shape of the corners, require a careful process engineering, while preserving the requirements of economic sustainability needed in large scale production.

Moreover, in order for the nozzle to properly operate, surface finishing must be controlled as well. Surface roughness plays in fact a relevant role in the complex phenomena taking place during fuel injection and vaporization [1]. On the other hand, surface roughness typically depends on many factors related to both material and process parameters. Therefore, the use of advanced techniques to measure the workpiece morphology and to assess its roughness is mandatory in order to improve the quality of the produced items.

Micro-Electro Discharge Machining (μ-EDM), or fs-pulse laser drilling (Fs-LD) are, respectively, the present standard and the forthcoming standard approach in the automotive industry for mass production of fuel injector nozzles. The ability to capture a whole view of the inner surface of the machined hole achieves a crucial role in the R&D of this key point sector, leading to a valuable route for process improvement and optimization [2].

We have imaged the morphology of micro-sized holes drilled by μ-EDM or Fs-LD by using a specifically conceived scanning probe microscopy (SPM) technique. The technique allows us to measure the surface roughness in true non-contact mode, in order to compare the finishing properties of surfaces produced with different process parameters or methods.

2. The drilling process

At present, μ-EDM is well established in the field of manufacturing metal pieces with complicated geometries, such as the drilling of nozzles for diesel and gasoline injection systems. The high flexibility with respect to workpiece geometry and material is a distinguishing advantage of the μ-EDM process. The technique remains the method of choice for drilling hard conductive materials of diameters down to 5 μm. The geometry of the holes is very important to the injection nozzle market [2], which has to improve quality and product performances.

The surface finishing properties of a μ-ED machined piece are governed by the physical mechanism involved in the process. Discharge energy produces very high temperatures at the point of spark on the surface of the specimen removing the material by melting and vaporization. The top surface of the workpiece re-solidifies and cools extremely quickly. This process causes a ridged surface and may induce micro-cracks due to the high tensile residual stresses in the surface layer [3]. Process parameters (e.g., pulse duration, energy delivered) can affect the roughness; in particular, smoother surfaces are attained with low energy pulses [4]. However, non-negligible roughness are typically obtained.

3. Morphological analysis of recessed surfaces

The advent of SPM, notably of atomic force microscopy (AFM), offered an extremely valuable tool for reconstructing the surface morphology with nanometer accuracy [5]. AFM, originally applied to analyze atomistically flat surfaces, can be adapted to measure sizeable roughness like those typically found in machined workpieces, providing with a diagnostics complementary to other, more conventional, microscopy techniques, either optical or electronic. Compared for instance to scanning electron microscopy (SEM), AFM offers as an important added value the ability of measuring, i.e., quantitatively determining, the topographical variations responsible for surface roughness. Such an ability stands at the basis of reliable comparisons between different techniques or samples processed with different parameters.

However, despite of its wide diffusion in the research environment, application of AFM to the analysis of workpieces, showing in particular recessed surfaces as those considered in the present work, presents several critical issues.

First of all, even though a variety of strategies has been developed to prevent contact between the AFM tip and the surface, wearing of the tip or, in the case of soft materials, surface scratching represent an important issue. As a matter of fact, probing the surface is typically accomplished in AFM by applying a fast oscillation of the tip along the vertical direction, that can easily lead to intermittent interactions producing probe blunting during the scan. A true non-contact operation is on the contrary strongly desired especially when corrugated surfaces must be analyzed.

Moreover, there are other relevant technical limitations hampering exploitation of conventional AFM for the investigation of machined workpieces. The physical dimensions of cantilevered probes pose serious limitations for accessing recessed surfaces, since, when landing the tip over the region of interest, the cantilever can interact with the uppermost borders of the workpiece. In addition, the optical lever method generally exploited in AFM is prone to artifacts due to scattering of the stray light from the corrugated surface. Finally, large travel capabilities are needed when workpieces with complex shapes must be analyzed.

In summary, highly versatile SPM techniques has to be deployed, able to approach and analyze in true non-contact mode recessed surfaces minimizing the needs for mechanical preparation of the specimens.

3.1. Shear-force microscopy

Shear-force microscopy (ShFM) represents a viable route to circumvent most of the above mentioned limitations of conventional SPMs. Shear-forces are effectively used to sense the surface in scanning near-field microscopy (SNOM) [6]. They are mostly based on the viscous interaction of the air layers imprisoned between the tip, kept in oscillation parallel to the surface, and the surface itself. Such an interaction is known to produce a strong damping of the oscillation when the tip-to-surface gap decreases at the few nanometers level [7].

Since the tip oscillations occur only along the parallel direction, any form of contact is avoided, at least if the oscillation amplitude is kept small enough. This prevents tip wearing allowing for reliable repeated scans useful for comparison purposes.

Furthermore, in the absence of vertical oscillations cantilevered probes are not needed and can be replaced by needle-like tapered tips, perfectly able to approach recessed surfaces. This remarkably widens the applicability of the technique to workpieces with complex shapes.

Finally, sensing the oscillation amplitude can be accomplished by non-optical methods, therefore removing any problem related to stray light scattering. In particular, commercial quartz tuning fork can be used, by gluing the needle-like tip to one of the fork arms, according to a method originally introduced by Karrai et al. [8].

4. Experimental setup

The custom design of the instrument implements all above advantages of ShFM. Core of the setup is the probe assembly, which includes the tip, glued to one tuning fork arm, and a piezoelectric transducer needed to put the fork/tip system into oscillation parallel to the specimen surface, as schematically depicted in **Errore. L'origine riferimento non è stata trovata.**

Tips are made by electrochemical etching a tungsten wire (0.125 mm dia.) in a KOH solution. The anisotropic conditions established at the wire end lead to the formation of a tapered tip. By properly setting the etching parameters, tips with an apical diameter on the order of one hundred nanometers are achieved. Tips are then epoxy glued on one tuning fork arm: the length of the tip exceeding the arm can be adjusted to the millimeter range, that enables probing recessed surfaces.

The oscillation, driven by a piezoelectric transducer, occurs at a frequency chosen close to the resonance of the mechanical system, close in turn to the resonance frequency of the bare tuning fork (around 32 kHz). The signal produced by the tuning fork, after low noise preamplification, is synchronously demodulated in order to produce a dc signal proportional to the mechanical oscillation amplitude.

Such a dc signal is sent to a feedback electronics, included in a commercial SPM controller (RHK), where is continuously compared to a set-point value, typically set around 80% of the free oscillation value. The output of feedback circuit, after proper conditioning, controls the vertical displacement of the sample. Therefore, constant tip-to-sample gap operation is achieved, preventing any accidental contact eventually leading to tip wearing. The control signal is acquired by the SPM controller in order to build the topography map.

The sample is placed on a 3-direction piezoelectric nanopositioner (Physik Instrumente) which operates in closed loop. Beside vertical displacement, required to ensure the constant gap operation, the nanopositioner allows for raster scanning the sample under the tip. The maximum travel enabled by the nanopositioner is 100µm x 100µm wide, whereas the maximum vertical displacement is 20 µm. Joined with the large physical accessibility to the specimen under investigation, this allows to analyze specimens with complex geometries and relatively large size.

The ShFM reconstructs the topography map of a sample as a h(x,y) matrix. The h (height) value is achieved from the feedback system upon the requirement of the constant gap. The number of points along each row and column of the scan can be adjusted up to a maximum of 2048 pixels.

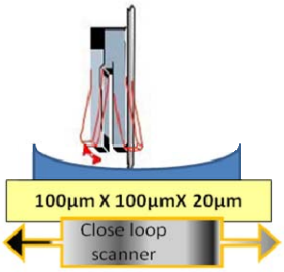

Figure 1 Sketch of the tip and sample arrangement

5. Narrow hole analysis

As already mentioned, a general advantage of SPMs compared to other microscopy tools (e.g., optical microscopy, SEM) is the availability of quantitative measurements in addition to imaging. Being the maps composed of numerical matrices, data can in fact be easily manipulated in order to quantitatively determine the morphological parameters of the surface, that can be carried out by using commercial software [9].

Prior to the roughness analysis, a macroscopic best fit is applied to remove the global curvature due to the circular shape of the hole, hence to project the surface over a flat plane (flattening). The topography maps can then be displayed, as shown for instance in Fig. 2, where a pseudo-3D representation is used (maps refer to a μ-ED machined hole in stainless steel).

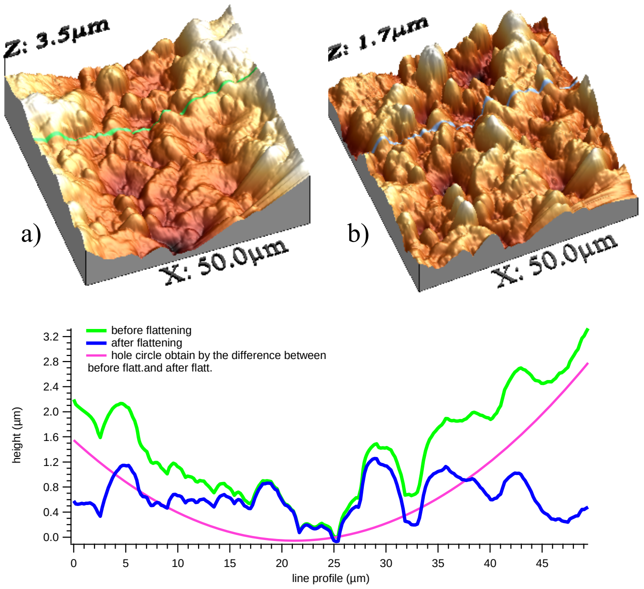

Figure 2 Pseudo 3D representation before (a) and after (b) the flattening process described in the text. The graph shows the cross sections of the topography maps, drawn along the lines superposed on the maps. The curvature obtained by the best fit procedure is also shown.

Using the cross section tool it is possible to extract the topography profile along one segment. This can be useful to compare different samples, for instance machined with different experimental conditions, as shown in Fig. 3. In this example, holes produced with different energy of the discharge pulse are compared, demonstrating that low energy leads to reduced roughness.

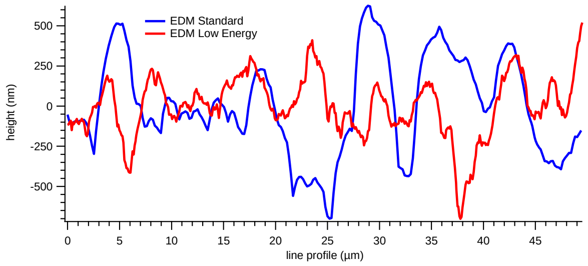

Figure 3 Cross sections of holes drilled with two different parameters sets: standard energy in blue and low energy in red.

5.1. Roughness parameters

Conventional roughness parameters [10] can be readily extracted from the h(x,y) matrices representing the sample topography. The analysis can be carried out either averaging over the whole map, leading for instance to surface-averaged total or root-mean-square roughness, S_A and S_Q, respectively, or choosing a direction and determining the total or root-mean-square roughness, R_A and R_Q, along such direction. In particular, vectors $R_{Qi,\ i=1,n}$, where n is the number of rows (or columns) of the matrix and the element R_{Qi} is the R_Q value evaluated over i^{th} row (or column) can be obtained.

The row by row evaluation offers the unique capability to understand the evolution, if any, of the roughness along the drilling direction. For instance, Fig. 4 shows the plots of three R_{Qi} vectors corresponding to different maps acquired in different regions along the hole axis (out area, central area, step hole). The three plots are joined together in order to show the roughness evolution along the whole hole length.

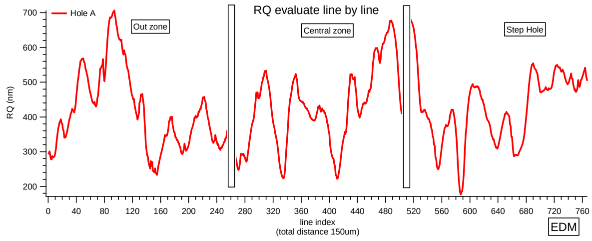

Figure 4 Plot of the vector R_{Qi} in the three regions along the hole axis. The drilling direction proceeds from right to left.

Evaluation of R_Q column by column and its comparison with the row by row analysis can instead be useful to highlight anisotropies induced by the machining process, giving rise, for instance, to morphological sub-structures.

5.2. Edge analysis

The ShFM turns out helpful also in another key point in the fabrication of injector nozzles, that is the study of the hole edge radius, expected to strongly affect the fluid-dynamics of the injection process. The design edge angle is typically 90 degrees, with a radius ranging from the hundreds nm up to few micrometers depending on the drilling technique and parameters.

The ShFM is able to scan the area across the edge providing with a direct measurement of the edge angle and radius. By subsequently scanning several rectangular areas across the edge, the evolution of the radius along the hole circumference can be investigated. Remarkably, such a kind of measurement does not require any modification to the instrumental setup, which is hence able to carry out a complete characterization of the workpiece.

6. Conclusions

In the original implementation we have developed, the ShFM is a powerful tool for the roughness investigation of complex shape surfaces and the geometrical characterization of the hole edges. The topography maps are obtained by true non contact operation and in the absence of technical limitations and artifacts often found with conventional AFM.

Therefore, the ShFM can acquire a relevant role as a complementary technique to SEM and optical microscopy, with the added value of providing the user with reliable quantitative data. in order to characterize the morphology features of a sample at the nano scale. Besides process and product optimization, mapping the topography can open the possibility, upon replicating over the whole hole surface, to feed numerical analysis of the injection and vaporization process with semi-true bordering conditions.

Moreover, the possibility to exploit the tip oscillation along the direction parallel to the surface which is at the basis of the shear-force mechanism in order to acquire tribological information, able for instance to discriminate between different materials, will be explored in the future to further extend the potential of the instrument in terms of nanoscale investigations of machined surfaces.

7. References

[1] Geometry of the holes A. Kufferath, S. Berns, J. Hammer, R. Busch, M. Frank, A. Storch, The EU6 Challenge at GDI – Assessment of feasible systems solutions, Proceedings of the 33rd Internationales Wiener Motorensymposium (2012).

[2] C. Diver, J. Atkinson, H.J. Helml, L. Li, Micro-EDM drilling of tapered holes for industrial applications, Journal of Materials Processing Technology, 149 (2004) 296-303

[3] Y.C. Lin, B.H. Yan, F.Y. Huang, Surface improvement using a combination of electrical discharge machining with ball burnish machining based on the Taguchi method, Int. J. Adv. Manuf. Technol. 18 (9) (2001) 673–682.

[4] G. Cusanelli, M. Minello, F. Torchia, W. Ammann, P.E. Grize, Properties of Micro-Holes for Nozzle by Micro-EDM, Proc. 15th Int. Symp. Electromach. (ISEM XV), (2007) 241 -245.

[5] For a review, see, e.g., B. Bhushan (Ed.), “Springer Handbook of Nanotechnology” (Springer-Verlag, Berlin Heidelberg, 2010).

[6] See, for instance L. Novotny and B. Hecht, Principles of Nano-optics, Cambridge University Press, New York, 2006.

[7] K. Karrai and I. Tiemann, “Interfacial shear force microscopy”, Phys. Rev. B 62, 13174-13181 (2000).

[8] K. Karrai, & Grober, R.D. (1995). Piezoelectric tip-sample distance control for near field optical microscopes, Appl. Phys.Lett., Vol 66, pp. 1842-1844

[9] I. Horcas, R. Fernandez, J.M. Gomez-Rodriguez, J. Colchero, J. Gomez-Herrero and A. M. Baro, Rev. Sci. Instrum. 78, 013705 (2007)

[10] Degarmo, E. Paul; Black, J T.; Kohser, Ronald A. (2003), Materials and Processes in Manufacturing (9th ed.), Wiley, p. 223, ISBN 0-471-65653-4