

Micromachining Lithium Niobate for Rapid Prototyping of Resonant Biosensors

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Abstract. Lithium niobate material is widely used in MEMS application due to its piezoelectric properties. This paper presents the micromachining process of lithium niobate to rapid prototype a resonant biosensor design. A high precision CNC machine was used to machine a sample of lithium niobate material at 5 different spindle speeds to find out the best conditions to machine this brittle material. A qualitative visual check of the surface was performed by using scanning electron microscopy, surface roughness was quantitatively investigated using an optical surface profiler and Raman spectroscopy to check the strain of the surface. Results show that the surface quality of the lithium niobate was significantly affected by the spindle speed with optimum conditions at 70k rpm giving a strained surface with 500 nm rms roughness.

1. Introduction

The design and fabrication of point-of-care biosensors attracts the attention of many researchers due to their applicability as they have high sensitivity, low price and the ability for being used outside the specialised laboratories. Mass biosensors are one of the most attractive designs for many applications especially the resonant based MEMS (microelectromechanical sensors) [1] which offers superlative sensitivity. As the main part of the diagnostic device, the resonant based MEMS biosensor needs to be designed and fabricated precisely ideally from crystalline material to give a high quality factor resonator.

The first attempt at developing a degenerate mode mass biosensor [2] at Newcastle University utilised microfabrication of crystalline silicon wafers to produce high quality factor resonators [3] although signal recovery from this capacitively driven and sensed device proved complex for the intended point-of-care applications [4]. To overcome this issue, a composite resonator was designed with thin film PZT for actuation leading to simplified signal recovery at the expense of resonator quality [1], thereby ultimately limiting device sensitivity. The group's aim is to further develop this sensor system, moving away from a silicon based technology to materials that give high mechanical and electrical performance.

The ability to produce MEMS based sensors using microfabrication techniques offers a low cost technology provided production volumes are high. The high overhead costs associated with microfabrication results in prototyping of designs typically costing in the range of £10000 to £100000 and with process flows not yet optimised for such designs, yields can be low. Currently a number of

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alternative micro manufacturing processes, such as micro milling, micro laser machining, micro water jet machining, can be used to produce micro components. Micro milling is the most flexible and versatile micro machining process and capable of generating a wide variety of complex micro components and microstructures due to its strengths in achievable precision and surface finish in addition to its simple set-up and versatility [5]. Micro milling as a newly emerged micro machining process has been successfully applied in processing various engineering materials [6].

The fabrication of piezoelectric based micro components is attracting more attention nowadays because of their properties such as compact size, high sensitivity and rapid response [7]. The crystalline form of lithium niobate coupled with its piezoelectric coefficients and chemical stability give this material excellent performance both mechanically and electrically. These characteristics make this material widely used in many applications especially in the field of sensor fabrication [8], applications include ultrahigh speed modulators and acoustic sensors [9]. The surface quality of the lithium niobate is very important aspect during any micromachining process especially when this material is used in precise devices such as biosensors [10]. However due to its low fracture toughness, lithium niobate has a lower material removal rate when machining compared to other materials and it is more likely to be scratched during the machining process [11]. Despite its mechanical and electrical properties, there are a limited number of publications available about micromachining lithium niobate for the purpose of manufacturing sensors.

Fabrication of micro scale devices made from other crystalline materials, such as silicon and graphite, by using conventional machining is reported by several groups. The reason for attempting to machine such brittle materials is to attempt to overcome the high costs and initial low productivity that can result from microfabrication approaches [12]. Mohammed Arif et al used a micro ball end machining process to machine silicon using a cubic boron nitride tool. Their results showed that there is a threshold of the tool feed rate, which had a significant effect on the surface quality, and they proved that it is possible to have a crack free surface by using this micromachining process on silicon [12]. Rusnaldy et al reported that even though there is a high possibility of surface damage when machining a brittle material such as silicon, it is possible to cut the silicon by using a micromilling process under various conditions [13]. They had implemented the micromachining process using diamond-coated end mills in order to machine grooves into silicon [13]. Kovalchenko presented a comprehensive report on machining brittle materials and showed that it is applicable in industry to machine silicon traditionally in order to increase the level of productivity and maintain the surface quality at acceptable levels [14]. In this paper, the micromachining of lithium niobate for the purpose of rapid prototyping a circular diaphragm resonant biosensor, akin to the design of Ismail et al [2], is assessed. The application of micro machining is expected to open new industrial avenues for prototyping biosensors.

2. Methodology

2.1 Micromachining lithium niobate using CNC machine

Experiments for this work were carried out on a NANOWAVE MTS5R – ML CNC machine as shown in figure 1. Standard CNC machine tools were used to ensure that the experimental results are applicable in industry. SAW grade, Z-cut lithium niobate samples of dimensions $10\text{ mm} \times 10\text{ mm} \times 0.5\text{ mm}$ thick were used in the experiment. As the samples were to be drilled from the back side down to thicknesses of around $50\text{ }\mu\text{m}$, the front surface required rigid supported to prevent the crystal from fracturing under the applied drilling pressure. Samples were glued to a holder by using a nail – lacquer glue and then left for three days to ensure they were rigidly bonded.

The tool used in this experiment was a 0.5 mm diameter cubic boron nitride (CBN) square end mill, manufactured by Union Tools. A G – code program was written and performed in order to machine holes in the lithium niobate samples with a depth of $450\text{ }\mu\text{m}$. Holes were machined at constant spindle speed starting from 40 k rpm to 80 k rpm in steps of 10 k rpm . The reason for choosing five different spindle speeds was to determine the most suitable condition for machining this brittle material and to

check the surface quality at each speed. Five holes were drilled at each speed. After finishing the machining process, the samples were removed from the support by submerging in acetone and then cleaned electronically by using a JPL Ultra-8060 Pro Ultrasonic Cleaner filled with water to ensure that the surface of each hole was cleaned before checking the surface quality.



Figure 1. Nanowave MTS5R – ML CNC machine.

2.2 Characterisation

2.2.1 Scanning electron microscopy (SEM)

A Hitachi TM3030 scanning electron microscopy was used to provide a qualitative visual check of the surface quality of each hole. A 15kV accelerating voltage was used in this experiment to provide high resolution topographical imaging at up to 30000x magnifications.

2.2.2 Optical surface profiler

To provide a quantitative evaluation of the quality of each machined surface, a Zygo NewView 5020 optical surface profiler with 10x objective was used. As lithium niobate is transparent, a 30 nm gold coating was evaporated onto the surface to improve measurement quality. A depth measurement of a hole confirmed a drill depth of 444 μm . Profiles were then taken of each drilled surface, figure 3 shows an example for a 60k rpm machined hole, and a plane mathematically subtract from the data. The rms value of the surface was then calculated and the number of points used in this analysis recorded, this number being indicative of the amount of visible (debris free) surface.

2.2.3 Raman system setup

It has been shown that Raman spectroscopy may be used to give a quantitative measure of crystalline quality and stoichiometry of lithium niobate after crystals are processed [11]. To quantify the micromachining process reported here, Raman measurements were taken of the samples using a Horiba Jobin Yvon FHR1000 system. The system utilised a beam from a 632.8 nm helium neon laser, this being focussed with a 50 \times long working distance objective giving a spot size of 4 μm . The Stokes Raman signal was collected, directed through the spectrometer and recorded on a liquid nitrogen cooled CCD camera set at -140 $^{\circ}\text{C}$. The system resolution was 0.25 cm^{-1} .

Spurious spikes (defined as greater than 5% increase in intensity from the neighbouring 6 points) were automatically removed from the spectrum and a Voigt profile (a convolution of a Gaussian and Lorentzian profile) was fit to the data using a Matlab nonlinear least-squares fitting routine. The Raman spectra of lithium niobate contain multiple peaks [15]. Work by Gallinetta et al [11] showed that for the 872 cm^{-1} line, the linewidth is indicative of composition whilst peak energy position reflects strains induced in the machined surface. However this peak is asymmetric showing a distinct

shoulder at around 900 cm^{-1} . For this work, only the lower energy side of the peak was fit, as shown in figure 2, between 820 cm^{-1} to 880 cm^{-1} to give an indicative assessment of peak position and width of this line resulting from the machining process.

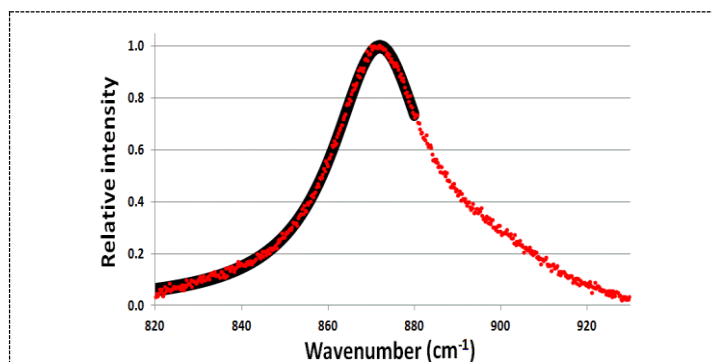


Figure 2. The 872 cm^{-1} spectral line shows a pronounced shoulder at higher energies. The peak was fit using the data points recorded on the lower energy side.

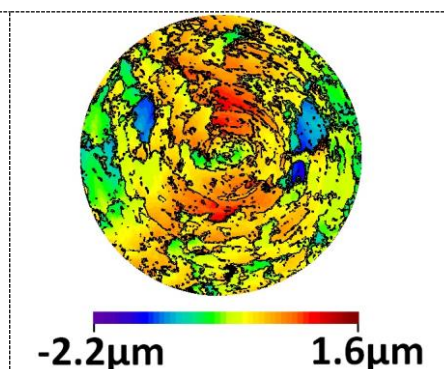


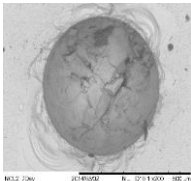
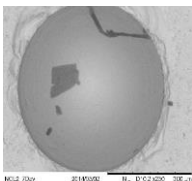
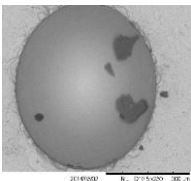
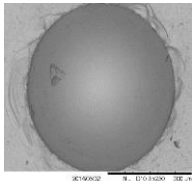
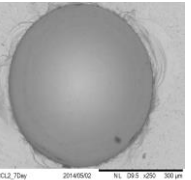
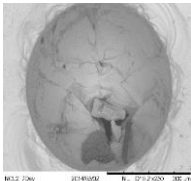
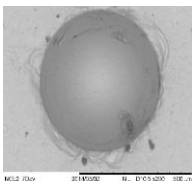
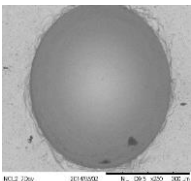
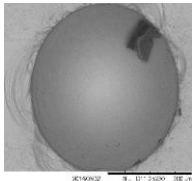
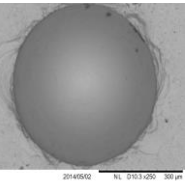
Figure 3. Surface profile measurement of the lithium surface machined at a speed of 60k rpm.

3. Results and Discussion

3.1 SEM results

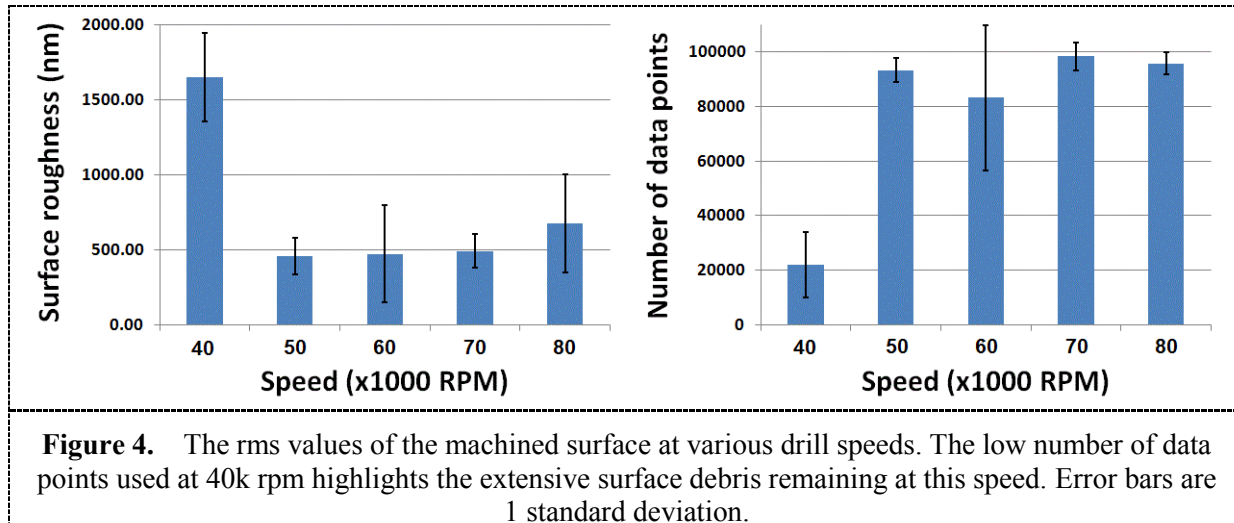
After finishing the micromachining process to drill 25 holes ($5\text{ holes} \times 5\text{ speeds}$) in the lithium niobate, the scanning electron microscopy was used to check the surface quality of each hole. Table 1 contains representative images of the surfaces obtained for each speed. The most significant damage occurred at a speed of 40K rpm, the results demonstrating that the slower machining speeds are unsuitable for this material. Above this, surface quality showed more consistency with no cracks being evident on the surface. Despite the ultrasonic clean, residual debris remained on the surface, this appearing to reduce as the machine speed increased.

Table 1. A qualitative comparison of the results of various machine speeds was performed with scanning electron microscope images of the surfaces.

Machining speed	40k rpm	50k rpm	60k rpm	70k rpm	80k rpm
					
					

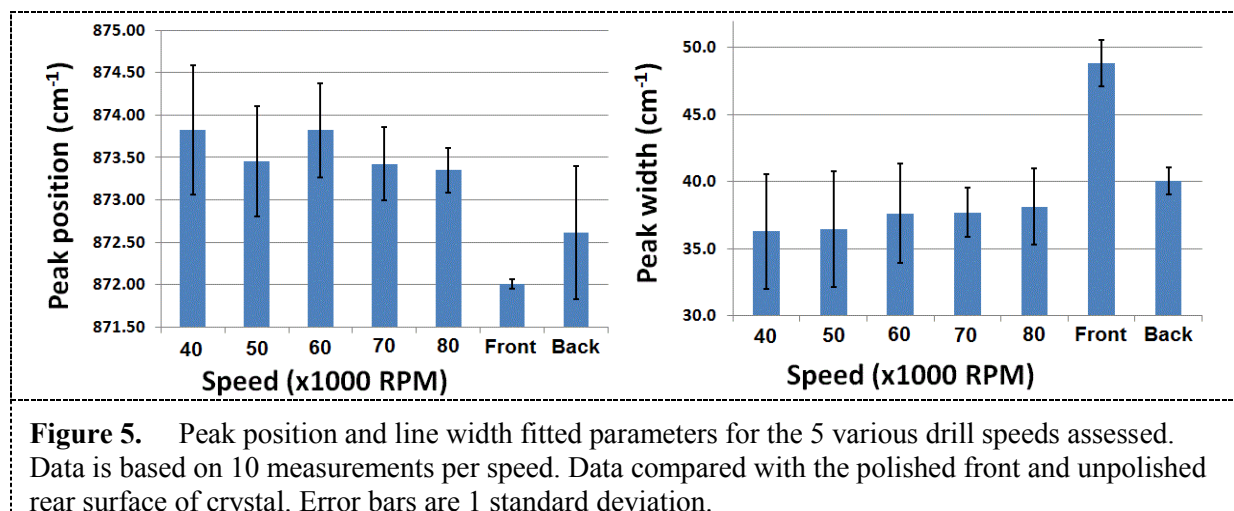
3.2 Surface profiler results

The results of the surface profiler analysis are shown in figure 4. At 40k rpm surface roughness is excessive at over 1500 nm and the large amount of debris after processing is evident from the limited amount of surface that can be measured. At 50k rpm and above, results remain quite consistent with rms values around 500 nm with residual debris minimising at the higher speeds. Whilst most surfaces demonstrated a random variation of height across their surfaces, at 80k rpm, some of the machined surfaces exhibited a cos 2 θ undulation resulting in the larger rms.



3.3 Raman results

The results of the Raman fitting is given in figure 5 with 10 measurements at each of the different drill speeds compared to the polished front side of the lithium niobate crystal and the unpolished back face. Unstrained lithium niobate shows a Raman peak at 872 cm^{-1} , the peak position measured indicates that the fabricated surface becomes increasingly strained at the slower machining rates. The line widths of the Raman peak resulting from the drilled surfaces indicate that stoichiometry remains consistent to within measurement accuracy and is comparable to the unpolished side of the lithium niobate crystal.



4. Conclusions

Comparing the results of varying the spindle speed from 40k rpm to 80k rpm, it can be clearly seen that the spindle speed has a significant impact on the surface quality of the lithium niobate. Low spindle speeds are redundant for machining this material due to the excessive damage that results. Above 50k rpm, results are generally consistent. Quantification by surface profilometry implies a machining speed of 70k rpm is optimum resulting in an rms value of surface roughness of 500 nm with minimal debris. Raman spectroscopy of the 872 cm^{-1} indicated that the surface is strained due to this process. Work is continuing in optimising the drilling process and further work on analysing the Raman spectra in conjunction with additional surface analysis techniques will aim to better quantify the process. To reduce debris and strain in the surface, post machining treatments will be investigated.

Acknowledgements

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References

- [1] Hu Z, Hedley J, Keegan N, Spoors J, Waugh W, Gallacher B, et al. Design, fabrication and characterization of a piezoelectric MEMS diaphragm resonator mass sensor. *Journal of Micromechanics and Microengineering*. 2013;23(12).
- [2] Ismail AK, Burdett JS, Harris AJ, Suarez G, Keegan N, Spoors JA, et al. The fabrication, characterization and testing of a MEMS circular diaphragm mass sensor. *Journal of Micromechanics and Microengineering*. 2008;18(2).
- [3] Ortiz P, Burnett R, Keegan N, Spoors J, Hedley J, Harris A, et al. Issues associated with scaling up production of a lab demonstrated MEMS mass sensor. *Journal of Micromechanics and Microengineering*. 2012;22(11).
- [4] Burnett R, Harris A, Ortiz P, Hedley J, Burdett J, Keegan N, et al. Electronic detection strategies for a MEMS-based biosensor. *Journal of Microelectromechanical Systems*. 2013;22(2):276-84.
- [5] Chae J, Park SS, Freiheit T. Investigation of micro-cutting operations. *International Journal of Machine Tools and Manufacture*. 2006;46(3-4):313-32.
- [6] Cheng K, Huo D. *Micro Cutting: Fundamentals and Applications*. Wiley: Wiley; 2013.
- [7] Lalinský T, Hudek P, Vanko G, Dzuba J, Kuti V, Srnánek R, et al. Micromachined membrane structures for pressure sensors based on AlGaIn/GaN circular HEMT sensing device. *Microelectronic Engineering*. 2012;98:578-81.
- [8] Zhang Z, Yang S, Xu C, Wang B, Duan N. Deformation and stress at pop-in of lithium niobate induced by nanoindentation. *Scripta Materialia*. 2014;77:56-9.
- [9] Kurihara R, Nakamura M, Yoshida M, Sumomogi T, editors. *Study on micro machining of lithium niobate using single abrasive grinding* 2006; Monterey, CA.
- [10] Jeong S, Lee H, Cho H, Lee S, Kim H, Kim S, et al. Effect of additives for higher removal rate in lithium niobate chemical mechanical planarization. *Applied Surface Science*. 2010;256(6):1683-8.
- [11] Galinetto P, Marinone M, Grando D, Samoggia G, Caccavale F, Morbiato A, et al. Micro-Raman analysis on LiNbO₃ substrates and surfaces: Compositional homogeneity and effects of etching and polishing processes on structural properties. *Optics and Lasers in Engineering*. 2007;45(3):380-4.
- [12] Arif M, Rahman M, San WY. An experimental investigation into micro ball end-milling of silicon. *Journal of Manufacturing Processes*. 2012;14(1):52-61.
- [13] Rusnaldy, Ko TJ, Kim HS. An experimental study on microcutting of silicon using a micromilling machine. *International Journal of Advanced Manufacturing Technology*. 2008;39(1-2):85-91.
- [14] Kovalchenko AM. Studies of the ductile mode of cutting brittle materials (A review). *Journal of Superhard Materials*. 2013;35(5):259-76.
- [15] Schaufele RF, Weber MJ. Raman Scattering by Lithium Niobate. *Physical Review*. 1966;152(2):705-8.