

Advancing Electrochemical Jet Machining through Process Monitoring and Control

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September 2022

A thesis submitted to the University of Nottingham for the
degree of Doctor of Philosophy

Abstract

Electrochemical jet machining (EJM) is a non-conventional surface texturing process based on the localised anodic dissolution of a metallic work piece under the influence of an impinging electrolyte jet and an applied electrical potential. Although the surface modification of metallic components through EJM has been widely researched, there is a limited understanding of the electrical response of the system and unexploited opportunities to utilise this for process monitoring and integrated surface measurement.

The research presented in this thesis addresses this knowledge gap and investigates the development of on-machine metrology and advanced process control through electrical measurements of the electrolyte jet. The application of a high-frequency electrical excitation allowed the measurement of work piece surface topography through mapping jet resistance as a function of spatial coordinates without affecting the surface. The performance of this novel measurement system was evaluated by comparison against established measurement techniques and an analytical model describing the response was proposed.

This was further applied to investigate the resistance during machining, and it was demonstrated that the removal depth can be correlated to the measured resistance in-process. This provides a route for real-time monitoring of the live process and implementation of closed-loop control. A method for automated tracking of the geometry of the work piece during machining based on the resistance was developed.

The resolution of electrochemical jet machining and the jet-based surface characterisation methods presented in this thesis is constrained by the diameter of the jet. To address this limitation, a novel end-effector was developed that can constrict the electrolyte jet through the hydrodynamic effect of flow focusing, and its machining and measurement performance was characterised. It was demonstrated that the jet diameter can be reduced 79% with corresponding 54% reduction of machine kerf width. The tool size can be continuously varied in-process through a simple variation of process parameters as part of the machine control programme.

Publications

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Acknowledgements

I would like to thank my primary supervisor, Professor Adam Clare, for his invaluable input and guidance throughout the PhD, and for his unceasing enthusiasm about this research. Working with him has significantly contributed to my professional and personal development. Thanks go to Dr Alistair Speidel for the many discussions about experiments, electrochemistry, and non-work-related matters that helped me get to where I am today. I am indebted to Mr Alexander Jackson-Crisp who was always available to brainstorm design ideas and rapidly come up with solutions when the practical realities of working in the lab got in the way of progress.

I would also like to thank Dr Sidahmed Abayzeed of the Optics and Photonics Research Group for contributing with his expertise in optics and his different perspective, and for introducing me to electrochemical impedance spectroscopy, which became instrumental for this thesis. I am extremely grateful to Dr Jonathon Mitchell-Smith of Texture Jet Ltd – the early work with him kickstarted this research and gave me the technical skills necessary to complete this work. Thanks also go to Mr Mohamed Zubayr, who will continue this research in new directions and to Dr Adam Thompson of the Manufacturing Metrology Team for his assistance with stylus profilometry.

I would like to thank Franziska Wohlgemuth who unwaveringly supported me through many highs and lows of this journey and Simon Taylor with whom I shared much of my spare time. Finally, I am grateful to my parents and my family who stood right behind me the whole time.

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Nomenclature

All symbols in bold represent vector quantities.

A_{cross}	Cross-sectional area of machined channels	m^2
A_{jet}	Cross-sectional area of the jet	m^2
A_{noz}	Cross sectional area of the nozzle	m^2
c	concentration	mol/m^3
c_b	Bulk concentration	mol/m^3
C_{dl}	Double layer capacitance	F
c_p	Specific heat capacity	$J/(kg.K)$
c_s	Surface concentration	mol/m^3
D	diffusion coefficient	m^2/s
d_{jet}	Jet diameter	m
d_{noz}	Nozzle internal diameter	m
d_p	Syringe plunger diameter	m
E	Electrode potential	V
E_{eq}	Equilibrium potential	V
F	Faraday's constant	C/mol
f	Frequency	Hz
h	Throat thickness for flow focusing end effector	m
H	Tip-to-orifice distance for flow focusing end effector	m
I	Electric current	A
J	Current density magnitude	A/m^2
J	Current density vector	A/m^2

j	Imaginary unit	-
J_0	Exchange current density	A/m ²
J_{lim}	Limiting current density	A/m ²
k	Electrochemical equivalent	Kg/C
L	Stand-off distance	m
l	Local vertical distance between nozzle exit plane and surface topography	m
L_b	Jet break-up length	m
L_{eq}	Equivalent distance obtained from resistance measurement of an impinging electrolyte jet	m
m	Mass	kg
M	Molar mass	kg/mol
N	Ionic flux	mol/(s.m ²)
$P(x, y)$	Removal rate function	m/s
p_g	Air pressure	Pa
q	Electric charge transferred	C
Q	Liquid flow rate	m ³ /s
R	Universal gas constant	J/(K.mol)
r	Radial distance from nozzle centre	m
R_{jet}	Resistance of the electrolyte jet	Ω
Re	Reynolds number of the jet	Dimensionless
S	Sensitivity of jet resistance to change in stand-off distance	Ω /m
s	Arc-length	m
S_d	Sensitivity of jet resistance to change in jet diameter	Ω /m

T	temperature	K
t	Time	s
t_p	Period of rotation of syringe pump	s
U	Potential difference applied between the nozzle and work piece	V
u	Ionic mobility	$\text{m}^2/(\text{V.s})$
$\mathbf{v}$	Flow velocity vector	m/s
$\mathbf{v}_d$	Dissolution velocity of the work piece	m/s
v_f	Feed rate	m/s
v_g	Velocity of coaxial gas	m/s
v_{jet}	Average flow velocity	m/s
V_q	Charge specific material removal	m^3/C
$w(x, y)$	Weight function	Dimensionless
We_g	Aerodynamic Weber number	Dimensionless
$x' = (x', y')$	Spatial coordinates relative to nozzle position	m
x_p	Pitch of lead screw	m
$x = (x, y)$	Spatial coordinates	m
$z(x, y)$	Surface height	m
Z	Electrical impedance	Ω
α	Charge transfer coefficient	Dimensionless
γ	Surface tension of liquid-air interface	N/m
δ	Nernst diffusion layer thickness	m
η	Current efficiency of anodic dissolution	Dimensionless
ν	Kinematic viscosity	m^2/s
ρ	Density of workpiece material	kg/m^3

ρ_g	Density of gas	kg/m ³
ρ_l	Density of electrolyte	kg/m ³
σ	Electrical conductivity	S/m
ν	Experimental uncertainty	Quantity dependent

Abbreviations

AC	Alternating current
CC	Constant current
CV	Constant voltage
DC	Direct current
EBSD	Electron backscatter diffraction
ECM	Electrochemical machining
EJM	Electrochemical jet machining
EJP	Electrochemical jet processing
FVM	Focus variation microscopy
IEG	Inter-electrode gap
LPBF	Laser powder bed fusion
MRR	Material removal rate
NA	Numerical aperture
OER	Oxygen evolution reaction
SECM	Scanning electrochemical microscopy
SICM	Scanning ion conductance microscopy
TEM	Transmission electron microscope
TMEMM	Through-mask electrochemical micromachining
UV	Ultraviolet

1. Introduction

Surface texture, comprising the local irregularities of a surface relative to its nominal shape, is often critical for the functional performance of engineering components as it determines the interaction of the surface with its environment [1] . Manufacturing processes that enable control and modification of surface texture are hence essential for the delivery of optimised end products. For example, the fabrication of surface features through texturing processes is indispensable for the manufacturing of solar cells, where microscale surface features are imparted to minimise reflection. In the mould tool industry, texturing is commonly applied to the mould to transfer an aesthetic pattern onto the plastic component. Textured surfaces are used in biomedical implants to promote better integration with the bone. As such, our ability to engineer surfaces for application is critical. This provides an overriding driver for the present work.

Electrochemical jet machining (EJM) is an emerging surface texturing technology for metal components that realises localised material removal by passing an electric current through a jet of conductive liquid (electrolyte) impinging on the work piece surface. A positive potential with respect to the nozzle producing the jet is applied to the work piece, thus making it the anode of the electrochemical cell, and resulting in controlled dissolution in the impingement region of the jet. Electrochemical material removal is unique in the regard that it exerts no mechanical or thermal effect on the work and as such it is favoured in scenarios where surface integrity is critical.

Despite the advantages that the process offers compared to established surface texturing methods, such as high-quality surface finish, absence of heat input to the work piece and low capital cost of the equipment, EJM still has a limited adoption in industry. Reasons behind this include the complex interaction with the work piece material, leading to lengthy optimisation of process parameters and difficult prediction of resulting surface geometry, and the limited machining resolution.

This thesis intends to address some of these limitations and augment the capabilities of the process through the development of on-machine metrology for EJM and improvement of machining resolution. The integration of measurement techniques within manufacturing processes is considered highly beneficial for achieving high-quality and low-cost end products, as it enables rapid inspection and quality control without taking the part outside of the production line. Furthermore, in-process metrology, meaning that the measurement is performed simultaneously with the manufacturing process, enables data acquisition in real-time and identification of problems or defects as they arise. The underlying physics of EJM presents opportunities to explore implementation of the above based on the measurement of the electrical resistance of the inter-electrode gap (IEG), which is known to be dependent on the distance between the nozzle and the work piece, without the need for additional sensors. Aided by mathematical models of the manufacturing process and the output signals, this can enable intelligent closed-loop surface processing where the machine tool can adapt to changing conditions of the IEG. In addition, improvements in resolution of the process are sought in this thesis, as this is identified as a key limitation of EJM. This is explored through the

application of flow focusing, which is a hydrodynamic method for miniaturising liquid jets previously reported in fundamental fluid dynamics studies [2]. It is hoped that this research will further mature EJM for industrial applications and increase the readiness of the process for current trends towards intelligent and digitalised manufacturing.

1.1. Aims and objectives

The overall aim of this thesis is to advance electrochemical jet machining for surface texturing of engineering components through the development of integrated on-machine metrology and process monitoring. To achieve this aim, the following research objectives are defined:

1. Investigate the electrical behaviour of the inter-electrode gap in EJM and specifically the electrical response to variation of work piece surface topography for the purpose of on-machine measurement
2. Understand the influence of system parameters on this response
3. Assess the feasibility of performing on-machine measurement based on the electrical response and evaluate the performance against conventional metrology
4. Understand the relationship between the electrical response during machining and the machined surface geometry to enable live process monitoring for quality control
5. Develop mathematical models for the surface geometry evolution and the electrical signal during the manufacturing process
6. Demonstrate the application of the developed process monitoring approach to closed-loop control of the machining process

7. Explore approaches to improve the currently achievable spatial resolution for electrolyte jet methods for the purposes of both machining and on-machine measurement

1.2. Thesis outline

The thesis is structured in the following chapters:

In Chapter 2, the state-of-the art in EJM is reviewed along with the fundamental electrochemical phenomena underpinning the process and current limitations of the technology. Based on this, the research gaps to be addressed in this thesis are identified. In addition, the application of on-machine metrology to electrochemical machining (ECM) and other manufacturing processes is discussed.

Chapter 3 details the experimental methods and data analysis used for this thesis.

Chapter 4, the first results chapter, explores experimentally the electrical behaviour of the impinging electrolyte jet and more specifically the response of electrical resistance to work piece surface geometry. The realisation of scanning measurement of surface geometry by mapping jet resistance as a function of spatial coordinates is demonstrated. This presents a novel method for non-destructive on-machine metrology within EJM.

Chapter 5 builds on the results of Chapter 4, by applying resistance measurements during EJM surface processing. A mathematical model is developed to simulate the evolving surface geometry during machining and predict the corresponding resistance response. This allows to utilise in-process resistance monitoring for the detection of errors in the machining process. A

resistance-based approach for automated tracking of 3-dimensional work piece surfaces with unknown geometry is also presented.

In Chapter 6, a method to improve the spatial resolution of electrochemical jet techniques for both machining and measurement is explored. The design of a novel end effector, which allows to constrict the jet through the principle of flow focusing, is reported. The new tooling is characterised through optical imaging and electrical resistance measurements of the jet and the machining performance is investigated.

The overall conclusions of the work undertaken and the implications of the developed innovations for EJM are presented in Chapter 7, while potential future research directions are outlined in Chapter 8.

2. Literature review

2.1. Overview and fundamentals of EJM

2.1.1. Overview of EJM

Electrochemical jet machining techniques emerged from conventional electrochemical processes like electrochemical machining (ECM) [3] and electropolishing [4]. The same apparatus can be used to realise different modes of electrochemical surface modification such as electrodeposition [5], hence the term electrochemical jet processing (EJP) is used in this thesis to refer to the broader family of jet-based electrochemical techniques. Ultimately, a potential difference is applied between two electrodes (tool and workpiece) with a conductive fluid medium (electrolyte) in the inter-electrode gap (IEG), which enables continuous charge transfer during processing [6]. For machining, the work piece is positive with respect to the nozzle (Fig. 2.1a), while jet electrodeposition requires the opposite polarity (Fig. 2.1c).

EJM differs from other techniques in the mode of electrolyte delivery to the workpiece, which occurs as a free jet emanating from a nozzle, which is usually translated over a fixed workpiece (Fig. 2.1a). A free cylindrical jet impinging on the workpiece surface experiences rapid deceleration (stagnation) in the impingement zone and spreads radially. At sufficiently high jet velocity, a radial thin film (wall jet) region of electrolyte can form, of supercritical flow (Fig. 2.1b). The radial supercritical flow transitions to subcritical, lower velocity thick film electrolyte at a hydraulic jump. It is understood that the radial thin film electrolyte region possesses a greater resistance than the free jet, restricting charge transfer to the impingement region. This enables the manipulation of

both the process parameters at a given point on the surface [7], and the surface electrochemistry at a given point in the process cycle [8] (Fig. 2.1c). This is the governing principle upon which the high degree of localised process control in EJM is derived. The central region of highest mass transfer (and highest current

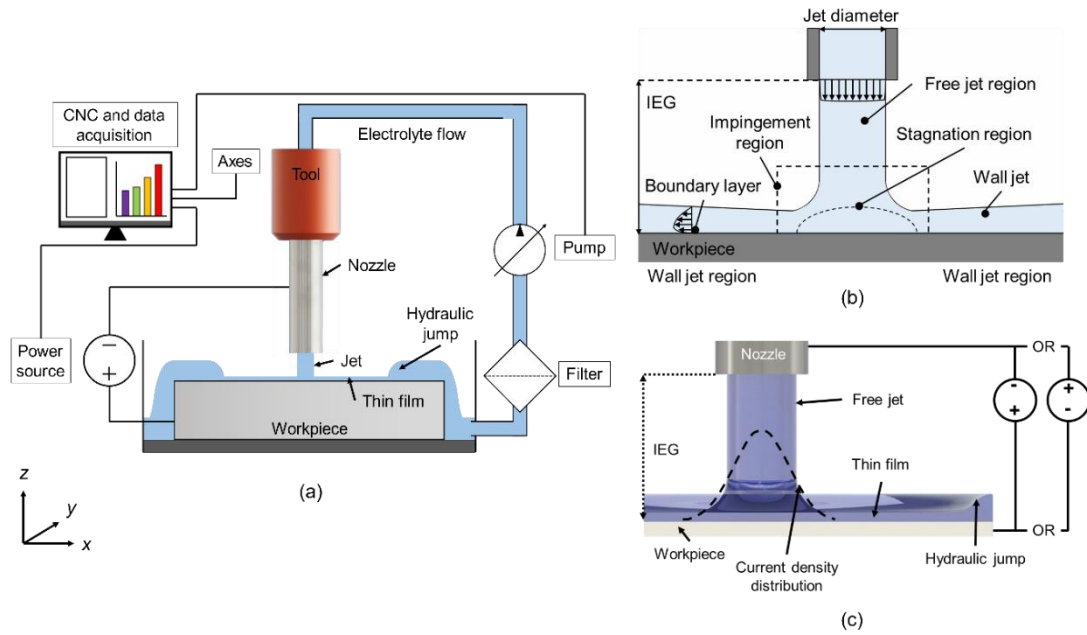


Fig. 2.1. (a) Schematic of typical EJM and EJP apparatus, with recirculating electrolyte. (b) Schematic of the hydrodynamic regions of the impinging jet. Electrolyte is ejected from a nozzle electrode towards a workpiece electrode at such a velocity to create a radial, wall jet in the impingement region. (c) Section of the IEG at onset of processing, showing a typical current density distribution profile across the jet and the different polarities applied for machining and electrodeposition density) may be further restricted by the application of a coaxial compressed air feed to the jet [9, 10], which stabilises the thin film region.

The primary function of the electrolyte is to provide a medium through which charge can be passed between the nozzle and the workpiece. As such, most electrolytes are aqueous solutions of dissolved salts that are highly conductive and Newtonian in nature under the process conditions. This reduces the power consumption and the probability of dielectric breakdown. Beyond this, the electrolyte is responsible for maintaining the stability of the electrochemical

process. This is achieved by clearing machining debris and evolved gases from the IEG, and controlling the mass-transport of reactants to, and products away from, the workpiece. The latter is responsible for determining the resulting microscale surface texture from the electrochemical jet interaction. Furthermore, high electrolyte flow rates reduce the impact of Joule heating in the gap, which can be significant at high current densities (up to 10^3 A/cm^2 [11]) and lead to boiling if uncontrolled [12].

2.1.2. Fundamentals of electrochemical mass transfer

All electrochemical manufacturing processes involve a change in the oxidation state of the workpiece and/or electrolyte atoms. Eq. (2.1) is the overall electrode reaction for metal dissolution or deposition in EJP driven by the applied potential.



where M denotes an atom of workpiece material. Eq. (2.1) shows an oxidation reaction of an anodic workpiece to effect one of two prescribed outcomes: anodic dissolution or passivation. The former is the mechanism through which metal atoms are oxidised from an anode through an applied potential difference and current, and solvated or suspended in the electrolyte – the machining process. In the latter, charge transfer leads to oxide layer growth, inhibiting machining. This is relevant for aqueous electrolyte systems having a source of OH^- anions and is the underlying principle of the industrially significant process of anodisation. Alternate reactions, such as oxygen evolution, can also occur at certain parameters. In the reverse direction (2.1) shows the cathodic reduction of a solvated cation and is the governing electroplating reaction,

depositing reduced metal at the workpiece. Again, alternate reactions, like hydrogen evolution, can occur concurrently or at certain parameters. Hydrogen evolution is also the main reaction occurring at the cathodic tool electrode in ECM. The processes at the workpiece and nozzle electrodes could in practice be more complicated than (2.1) involving multiple reaction steps or adsorption at the surface. The mass transferred at the electrodes m is proportional to the quantity of electric charge passed through the cell q according to Faraday's laws of electrolysis:

$$m = \frac{Mq}{Fz}, \quad (2.2)$$

where F is the Faraday constant, M the atomic mass of the reacting species, and z is the valency. The ratio $k = \frac{M}{Fz}$ defined as the electrochemical equivalent of the substance. In electrochemical machining and deposition, current efficiency η is defined as the ratio between the observed mass (removed or deposited) and the theoretical mass according to (2.2). This quantity effectively indicates what proportion of the current flowing through the system is carried by electrochemical reaction of interest as opposed to competing reactions. The current efficiency is not usually constant, but a function of applied current or potential.

Considering the equivalence between mass and charge transfer, localised mass transfer rates correspond to the local current density $\mathbf{J}$ at any point in the electrolyte solution:

$$\mathbf{J} = F \sum_i z_i \mathbf{N}_i \quad (2.3)$$

where $\mathbf{N}_i$ is the flux of ions of species i (number of ions passing per unit area per unit time) and the right-hand side represents the contributions of all species involved in the process. Symbols in bold here and throughout the thesis represent vector quantities. For practical purposes, in EJP the average magnitude of the current density J is often quoted as the ratio between total applied current and the nozzle cross-sectional area. However, the form of the current density distribution near the work piece and tool electrodes is non-uniform (Fig. 2.1c). The ionic flux in the system is given by the Nernst-Planck equation:

$$\mathbf{N}_i = -u_i c_i \nabla U - D_i \nabla c_i + c_i \mathbf{v} \quad (2.4)$$

where c_i is the ion concentration, u_i is the ionic mobility, U is the electric potential, D_i is the diffusion coefficient and $\mathbf{v}$ is the velocity of the electrolyte solution. The three terms in (2.4) correspond to contributions to the ionic flux from electric migration, diffusion and convection respectively. Substituting into Eq. (2.3), the expression for current density is:

$$\mathbf{J} = -F \nabla U \sum_i z_i u_i c_i - F \sum_i z_i D_i \nabla c_i + F \mathbf{v} \sum_i z_i c_i \quad (2.5)$$

In the bulk electrolyte solution away from surface of the electrodes, there is an excess of electrolyte ions which are continuously refreshed, so concentration gradients can be neglected. The electrolyte is assumed to be electroneutral $\sum_i z_i c_i = 0$ and Eq. (2.5) reduces to the generalised Ohm's law:

$$\mathbf{J} = -\sigma \nabla U \quad (2.6)$$

where $\sigma = F \sum_i z_i u_i c_i$ is the conductivity of the electrolyte solution. Charge transport in the bulk occurs only due to migration of the electrolyte ions. Near the surface of the electrode, concentration gradients become significant due to

the presence of reaction products or depletion of electroactive species from the electrolyte. In this region, convection and the diffusion are the dominating factors determining overall mass transport. A common theoretical approximation of the near-electrode region is the Nernst diffusion layer, according to which there is a layer of finite thickness δ on the electrode surface (Fig. 2.2a) where the concentration of the species of interest varies linearly with distance from the surface. The thickness of this layer is determined by the flow of the impinging jet. Outside the diffusion layer the bulk concentration is c_b . Following these assumptions, the magnitude of the current density due to an electroactive species is:

$$J_i = - \frac{Fz_i D_i (c_{ib} - c_{is})}{\delta} \quad (2.7)$$

where c_{is} is the surface concentration. Transport through the diffusion layer determines the availability of reactants or the removal of products at the electrode-electrolyte interface and hence it is often the limiting step for electrochemical reactions at the electrode (Fig. 2.2b-c). The rate of transport through the diffusion layer has significant effects on the microscale surface topography in EJP [13]. An important condition occurs when, dependent on the mechanism, the surface concentration of reacting ions reaches saturation (if the process is limited by product removal) or is reduced to zero (if the process is limited by supply of reactants). The Faradaic process at the electrode is then progressing at the fastest rate possible and the corresponding current density is known as the limiting current density J_{lim} . Semi-empirical expressions for the

limiting current density and diffusion layer thickness for the stagnation region of the impinging jet have been proposed [14, 15].

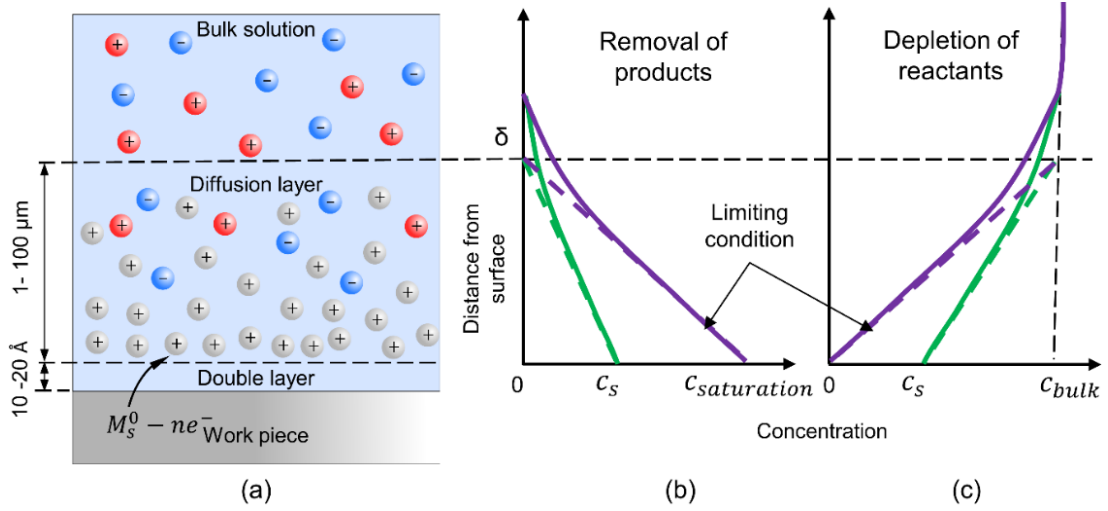


Fig. 2.2. (a) Diagram of the electrolyte environment adjacent to the workpiece electrode in an electrolyte. Typical diffusion layer and double layer thicknesses taken from [16] Typical concentration profiles of a (b) product-removal limited and (c) reactant depletion limited reaction.

At the electrode-electrolyte interface the electron transfer reaction (2.1) causes a potential shift of the electrode from its equilibrium value E_{eq} at zero current, which is known as an overpotential. Assuming a simple single-step reaction and both anodic and cathodic processes occurring at the same electrode, the general relationship between current density and overpotential is described by the Butler-Volmer model of electrochemical kinetics [17]:

$$J = J_0 \left\{ -\frac{c_{O_s}}{c_{O_b}} \exp \left[-\frac{\alpha F}{RT} (E - E_{eq}) \right] + \frac{c_{R_s}}{c_{R_b}} \exp \left[\frac{(1 - \alpha) F}{RT} (E - E_{eq}) \right] \right\} \quad (2.8)$$

where c_{O_s} and c_{O_b} are the concentrations of the oxidant (M^{n+} in Eq. (2.1)) at the electrode surface and the bulk respectively, c_{R_s} and c_{R_b} are concentrations of the reductant, R is the universal gas constant, T is absolute temperature, α and J_0 are the charge transfer coefficient and the exchange current density,

which are treated as empirical constants. The two terms of the above equation correspond to the cathodic and anodic components of the current at the electrode. In the case of EJP, one of these components is dominating dependent on the selected operating mode and the other can be considered negligible. Eq. (2.8) can then be reduced to the Tafel equation:

$$E - E_{eq} = a + b \log J \quad (2.9)$$

where a and b are constants. Electrochemical kinetics plays an important role in EJP for example when competing reactions occur the electrode surface such as the dissolution of discrete phases in the workpiece or oxidation of electrolyte ions [18]. Reactions with lower overpotentials occur at faster rates and consume a larger proportion of the processing current. Slow electrochemical reactions with large overpotentials require more energy input to achieve a certain current.

2.1.3. Electrical behaviour of the IEG

The non-linear electrochemical kinetics, defined by Tafel's law (Eq. (2.9)), and mass transport effects lead to non-linear potential-current relationships for electrodes in electrochemical systems. Fig. 2.3 illustrates qualitatively the current response of a dissolving metal anode upon increase of applied potential as outlined by Datta [19]. At low potentials, the current obeys Tafel's law and the electrode undergoes 'active' dissolution. At some potential range a limiting current plateau can be observed (blue curve in Fig. 2.3) associated with the saturation of the near-electrode region and the formation of surface films. Some materials exhibit passivation (red curve in Fig. 2.3) where over a certain potential range the surface is covered with an oxide film preventing dissolution leading to a significant reduction of current flow. Further increase of potential

beyond the passivation region leads to a renewed increase of current in the ‘transpassive’ region which may involve pitting of the passive film, oxygen evolution or transpassive dissolution of the metal. The fact that the current density distribution in EJM is non-uniform means that different portions of the affected region of the workpiece can be in a different regime locally.

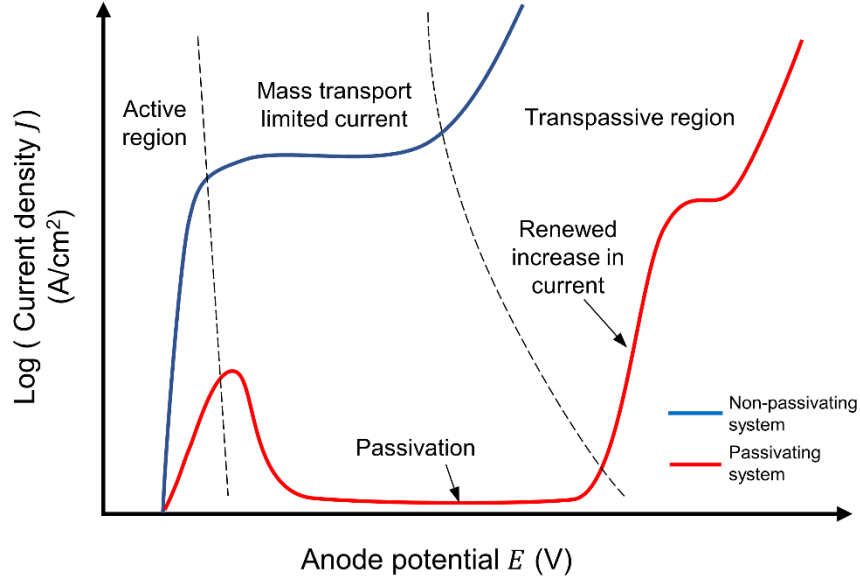


Fig. 2.3. Polarisation behaviour of an anodic work piece showing regions of active dissolution, passivation and transpassive dissolution. The blue curve represents the response of a metal, which dissolves in the ‘active’ regime according to Tafel’s law. The red curve is typical of a metal that forms passivating oxide film hindering dissolution and current flow. The anodic behaviour is dependent on the material – electrolyte system. Reproduced from [19]

Considering the overall voltage of the IEG in EJM, this also includes contributions of the process occurring at the nozzle and the Ohmic voltage drop across the jet due its resistance R_{jet} . The Ohmic drop is linear with respect to current according to Ohm’s law $U_{\Omega} = IR_{jet}$ and the jet resistance has been proposed [20] to approximate that of a cylindrical conductor with

$$R_{jet} = \frac{L}{\sigma A_{jet}} \quad (2.10)$$

where σ is the electrolyte conductivity, A_{jet} is the cross-sectional area of the jet and L is the vertical distance between the nozzle and the work piece (stand-off distance).

Electrochemical cells also exhibit capacitive behaviour due to the charge redistribution occurring at electrode interface. A simple representation of the interfacial structure, known as the electric double layer is shown in Fig. 2.4a. Ions accumulate near the electrode surface to balance excess charge near the surface within the metal. The charge is distributed in two regions – the compact region, consisting of adsorbed ions on the electrode surface, and the diffuse region consisting of solvated ions at a distance from the electrode. This separation of charge is equivalent to a capacitor and is important under transient electrical conditions. The model electric circuit for the IEG in Fig. 2.4b. shows the double layer capacitance occurring in parallel with an impedance associated with the Faradaic reaction, which is in general non-linear. The two overall electrode impedances are in series with the resistance of the jet. Oxide films on the surface of metal electrodes also contribute to the capacitance.

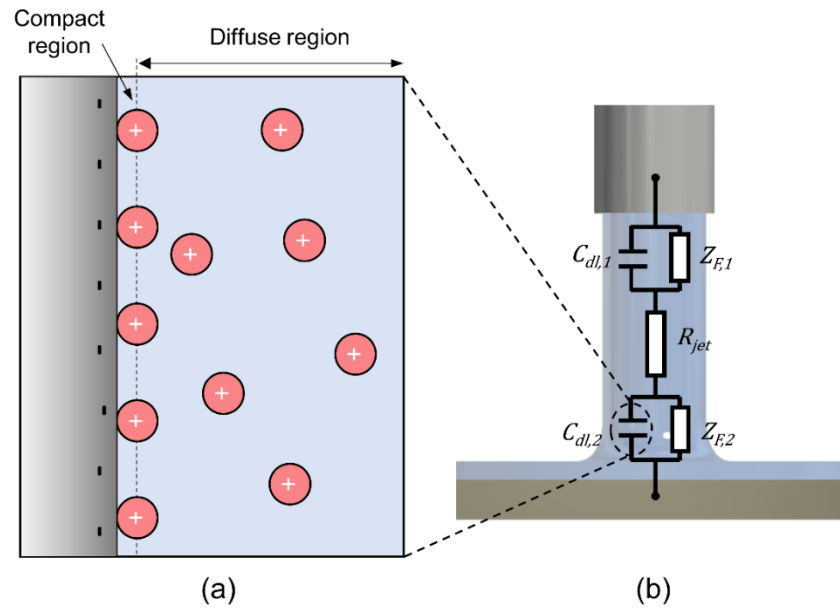


Fig. 2.4. (a) A representation of the electric double layer at the interface. (b) Electrical circuit analogy for the IEG.

2.1.4. Alternative operating modes

Electrochemical surface reactions that have been applied through a jet for both anode (red) and cathode workpieces (black) are shown in the process tree in Fig. 2.5. While this thesis is concerned primarily with the machining mode of EJP, the alternative surface modification mechanisms exploited using the apparatus are adaptations of industrially relevant techniques, hence they are summarised here.

Fig. 2.5a shows the mechanism of anodic dissolution, the underlying principle of ECM technologies and the subject of significant research and commercial activity in electrochemical processing. The overall observed reaction is the liberation of workpiece material due to electrochemical oxidation, according to Eq. (2.1). The specific reactions that occur during processing are complex and dependent on the workpiece material, the current density and the electrochemical environment.

Anodization also is dependent on the oxidation of a surface atom of metal (Fig. 2.5b), where the anion responsible for charge transfer is generally OH^- . The mechanism is understood to be reliant of the electrical migration of a hydroxyl anion through the passive oxide layer, where charge transfer occurs upon chemical contact with a lattice metal atom (M^0) to form an oxide of composition MO_n . Here, n is determined by the possible oxidation states of the metal and the process parameters at which the workpiece was applied. The electrical migration of hydroxyl anions through insulating oxide layers requires high electrical fields, therefore limiting the effective oxide film thickness achievable by anodization to sub-micron levels.

Plasma electrolytic oxidation (PEO) has recently been demonstrated using electrolyte jet apparatus [21], and the method is capable of depositing thicker oxide films than anodization alone (Fig. 2.5c). However, the mechanism is not purely electrochemical and is reliant on discrete electrical discharges, rather than continuous current flow. For PEO on an anode workpiece, an initial thin film is grown through an anodization (Fig. 2.5b). As the film grows, the electrical field strength across this oxide grows until it exceeds that of dielectric breakdown of the oxide, therefore forming an electrical discharge. The oxidation of the metal occurs upon the cooling of the discharge plasma (Fig. 2.5c). Localised plasma electrolytic polishing (PEP) has also been demonstrated within a jet-based system [22]. Similarly, this is mechanistically dependent on the formation of a gas envelope over the workpiece (anode), across which forms a plasma.

Electrodeposition refers to the electrochemical reduction of a metal cation onto a cathode workpiece to build up a layer of the coating material (Fig. 2.5d) [23],

and is industrially relevant in many fields of macroscale coating and microfabrication in electronics and micro-mechanical systems. In EJP, the metal cation is supplied by the electrolyte to the workpiece surface. EJP can be exploited for the generation of metal matrix composite coatings, whereby suspended particulate matter within the electrolyte is deposited with the matrix former to generate a co-deposit, denoted by the yellow particulates shown in Fig. 2.5e [24].

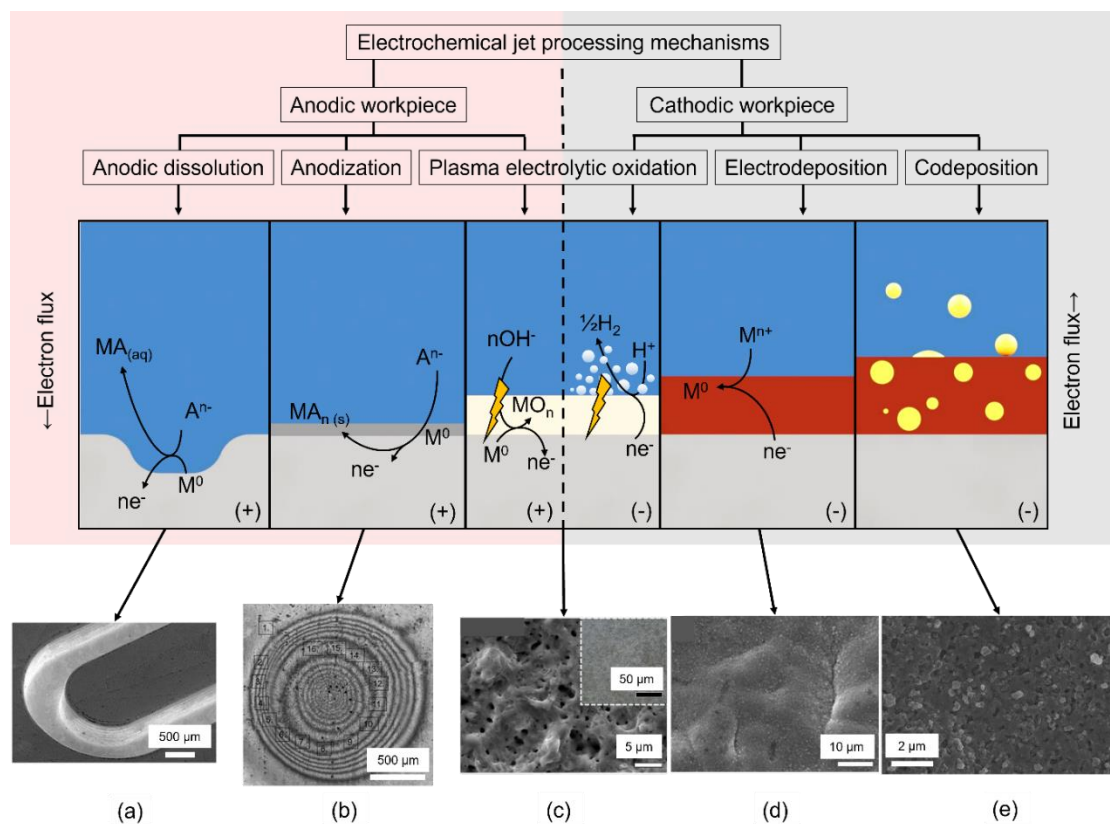


Fig. 2.5. Process tree of electrochemical micro-machining and surface modification methods applied experimentally through an electrolyte jet, based on the work piece polarity. Anodic workpiece: (a) anodic dissolution, (b) anodization, (c) plasma electrolytic oxidation (opposite polarity also possible). Cathodic workpiece: (d) electroplating, (e) particle codeposition. SEM micrographs: (a) taken from Hackert et al. [25], (b) LSM taken from Morgenstern et al. [26], (c) taken from Clare et al. [21], (d) taken from Speidel et al. [27], (e) taken from Ning et al [28].

Cathodic hydrogenation of Nb has been demonstrated using EJP apparatus [29], with the objective of improving conventional machinability of the material

by controlling the ductile-to-brittle transition in a selective manner. Nb was selected due to the low temperature embrittlement behaviour.

2.2. Process design and parametric influence in EJM

The performance of EJM is defined by a large number of process parameters. The influence of these is discussed here along with advancements in process design.

2.2.1. Tool path strategy

Translating the jet over the work piece is necessary to generate complex features and to modify large areas. While altering the translation velocity (feed rate) will affect local mass transferred at a given current (Eq. (2.2)), non-uniform current density distributions also affect the surface and the profile. In static machining (i.e. drilling), the surface condition at the feature centre is defined by the peak current density, which decreases radially from the jet centre. Consequently, there is a difference in surface finish between the centre and the edges of the machined feature – a highly polished surface is easily achieved within the central region of the pit under the influence of the high current density, but the roughness increases in the low current density region where the regime of dissolution is different (Fig. 2.6a).

Typically, in EJM pit machining, a power law dependence on final roughness at the pit centre with increasing current density has been reported for most non-passivating materials, regardless of composition or present phases. This has been well reported for single phase materials, such as stainless steels [30-32], Cu [33, 34], and Ni [35], as well as multiple phase materials, such as Ni-superalloys, WC-Co composites [7], and dual-phase brasses [18]. In some

materials, the surface finish has been observed to deteriorate upon further increases to the current density (Fig. 2.6b), such as processing steels with NaNO_3 based electrolytes [36]. This could be related to concurrent oxygen gas evolution in this electrolyte upon operation in the high transpassive dissolution

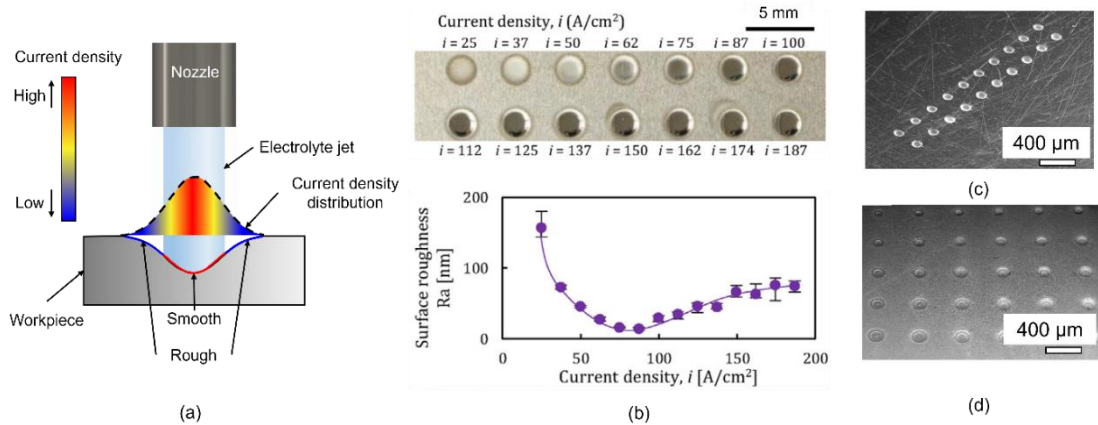


Fig. 2.6. Pit machining in EJM. (a) Pit centre finish is determined by the peak current density. (b) This leads to predictable finishes and similar roughness-current density curves for most materials. As such, (c) steels and (d) WC-Co materials can be machined. b. from [36], c. from [25] d. from [7].

state. Controlled finishes can be imparted into steel and even WC-Co composites, with appropriate electrolyte selection (Fig. 2.6c-d).

Translating the nozzle is necessary to achieve complex microfeatures like those shown in Fig. 2.7a-c. However, nozzle translation enables lower current density regions to interact with the surface, which deteriorates finish if not controlled. Pits and groove features were directly compared by Ikeda et al. [31], who showed that translation increases roughness at a given current density. In addition, the resulting roughness relationship is removed from the power-law relationship observed in drilling; translation reduces the effect of changes to current density on the resulting surface.

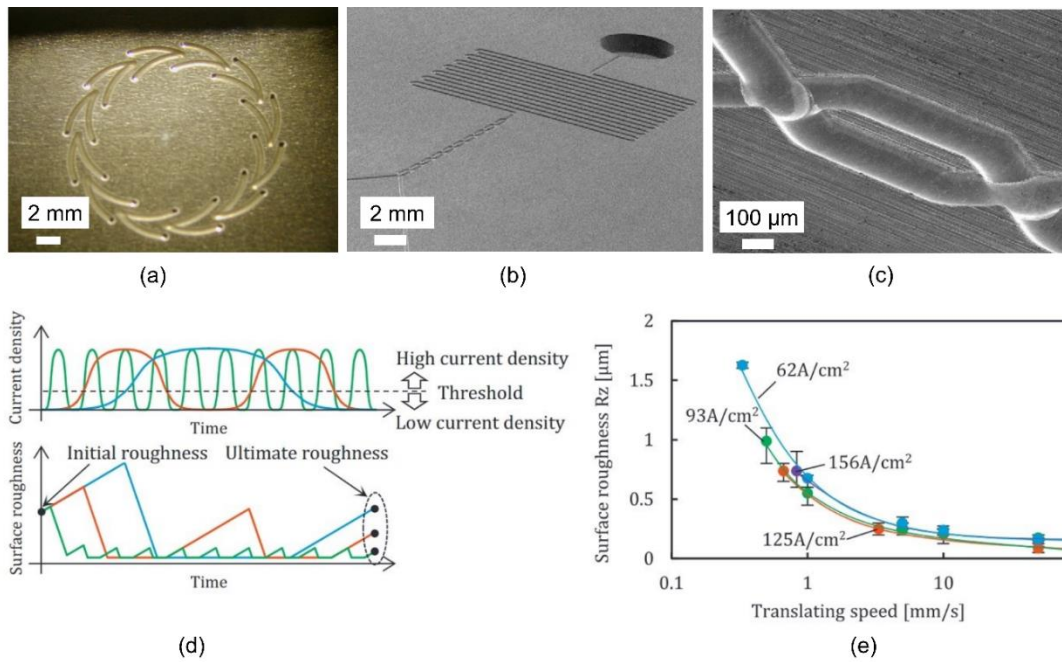


Fig. 2.7. Surface structuring with EJM. (a) Translating the jet allows low current densities to interact with the surface on translation, leading to 'keyhole' phenomena. (b-c) Translation is necessary to generate complex shapes. (d) Increasing feed rate decreases low-current density interaction time, (e) leading to smoother surfaces. (a) adapted from [32], (b-c) taken from [25], (c) and (d) taken from [36].

The extent to which the surface finish deteriorates upon nozzle translation in EJM is dependent on the nozzle feed rate. For example, Sen and Shan [30] found negligible influence of feed rate on surface finish at slow feed rates (0.2 – 0.8 mm/min). Higher feed rates have been shown to decrease surface roughness in different steels (12 – 60 mm/min) [37]. Kawanaka and Kunieda [36] proposed that smaller exposure times (increasing feed rate) of the surface to the low current density regions could reduce dull finishes, exploiting slow dissolution rates and strong time dependence in the low current density regions (Fig. 2.7c-d). Polishing effects were observed at high translation velocities (up to 6,000 mm/min). At high feed rates, multiple passes are superimposed to preserve total material removal [32]. The relationship between translation velocity and surface amplitude is dependent on the workpiece composition and

the electrolyte chemistry, for example strongly passivating materials (like Ti-alloys) behave in a different manner due to the requirement to break down surface oxide films prior to removal. Complex nozzle translation strategies are employed to remove material to target 3-dimensional profiles and to increase dimensional accuracy. For example, Natsu et al. [38] used a layer-by-layer profile superposition approach to generate a large (1100 μm in diameter) spherical cavity using a small nozzle (internal diameter 50 μm).

2.2.2. Nozzle and work piece geometry

Local reaction rates are defined by the current density experienced at any point on the workpiece. Using cylindrical nozzles, the current density is typically observed to have a bell-shaped spatial distribution [12, 39] analogous to energy beams such as lasers and electron beams. This limits dimensional accuracy and challenges consistent surface finishing, manifesting as tapered holes or grooves in EJM.

Nozzle diameter generally dictates feature scales in EJP by determining electrolyte jet diameter and therefore the minimum radius of the electrolyte thin film. This represents the limit at which sufficient current density can evolve to effect material removal, where the high-velocity resistive thin film localises mass transfer [40]. In preliminary studies, the hydrodynamic stagnation region for impinging jet electrodes was found to correspond to a radius of approximately 0.6 – 1.4 nozzle diameters [41]. Multiple studies in EJM have subsequently agreed with this, where the feature size is often observed to be between 1.5 - 3 nozzle diameters [32, 37, 42, 43]. Enhancing the resolution of EJP methods is hence reliant on the capability to manufacture small nozzles to be used as the tool electrode. Li et al. developed a method for in-situ

preparation of nozzles whereby copper or platinum is electrodeposited on the internal surface [44] resulting in a reduced jet diameter (Fig. 2.8a). Platinum was chosen for jet deposition applications due to its nobility compared to copper to prevent dissolution of the tool. A nozzle size of 20 μm was reported after 10 mins of deposition time. By applying this constricted jet to micromachining and electrodeposition, the authors were able to machine micro-grooves with widths down to 20 μm and fabricate freestanding Cu columns. Reduction of feature size relative to nozzle diameter can be achieved by adapting power supply conditions (Section 2.2.4), electrolyte chemistry and supply (Section 2.2.5), as well as adapting the nozzle profile and angle of address. Alongside cylindrical nozzles, slit nozzles are applied in EJM for the purpose of time-efficient machining, finishing, etching, and turning of larger surface areas [36, 45] (Fig. 2.8b).

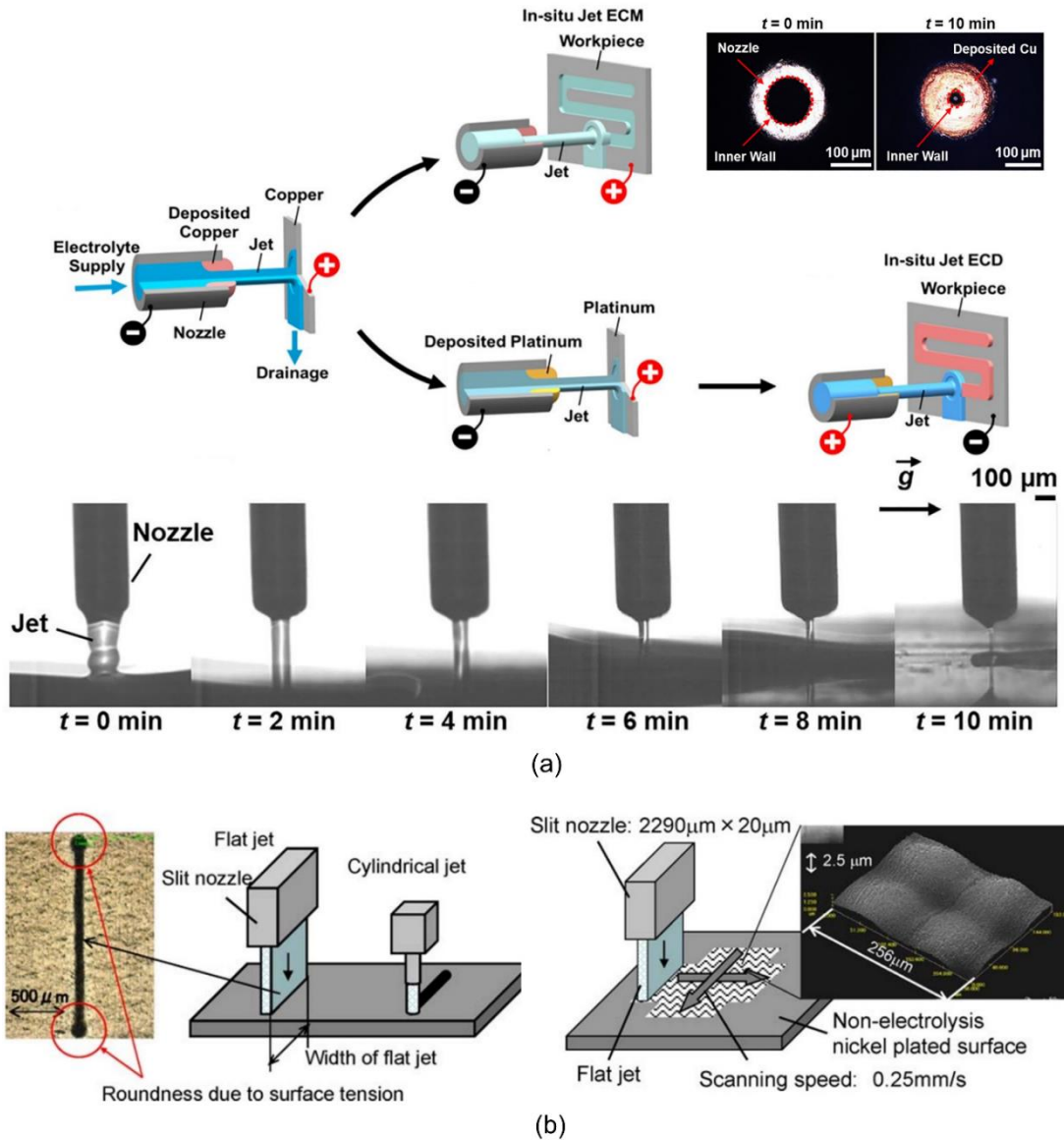


Fig. 2.8. Processed feature dimensions are closely related to nozzle size. (a) Fabrication of cylindrical nozzles for EJP through in-situ electrodeposition as reported by Li et al. [44] (b) Slot jets designed for rapid texturing of large areas. Taken from [45].

In order to modify the conventional axially-symmetric current density distribution, Mitchell-Smith et al. [46] proposed adapting the nozzle geometry (Fig. 2.9a-c) exploiting the idea that current flow will tend to focus along the shortest distances between the nozzle rim and the surface. By modifying the rim contour at appropriate length scales, control over the resulting feature geometry was realised which was correlated to an increase in resistance of the IEG dependent on the specific nozzle design (Fig. 2.9d). Higher aspect ratio

features than those achievable with standard cylindrical nozzles were obtained.

Modifying nozzle geometry can also improve the evacuation of electrolyte and

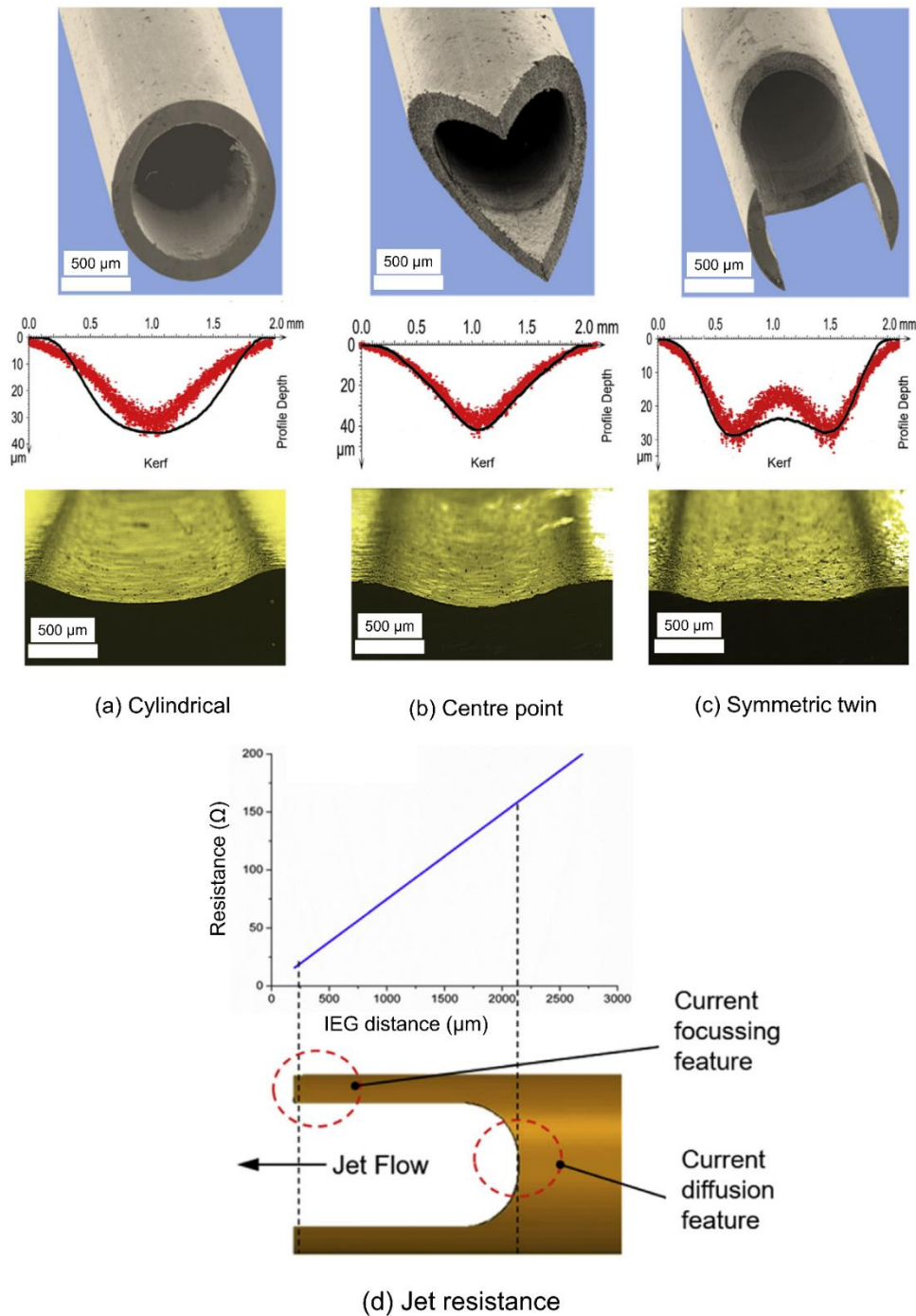


Fig. 2.9. Current density distributions can be modified by nozzle geometry. (a) Cylindrical nozzle and resulting profile, (b) centre-point nozzle and higher aspect ratio profile, (c) symmetric twin nozzle and 'w'-shaped profile. (d) At appropriate length scales, nozzle geometry can affect the resistance across the jet and therefore the local current density at the workpiece. Adapted from Mitchell-Smith et al. [46]. Scale bars are 500 μm , unless indicated otherwise.

electrolytic products from the IEG, especially at low IEG distances. Liu and Qu [47] showed that inclined angles of the nozzle end could prevent the nozzle from contacting the evacuating electrolyte stream which led to improvements in stray machining and surface roughness. This also resulted from the restriction of the current density distribution to the forward end of the nozzle, which enabled higher current densities and material removal rate (MRR) (at 40 V applied voltage).

When processing more complicated features than grooves using non-axisymmetric nozzles, it is necessary to integrate a rotational axis to maintain the desired orientation between the nozzle relative to the workpiece (Fig. 2.10a). Applying a rotational setup, Mitchell-Smith et al. were able to machine circular features with a symmetric-twin profile (Fig. 2.10b) [48].

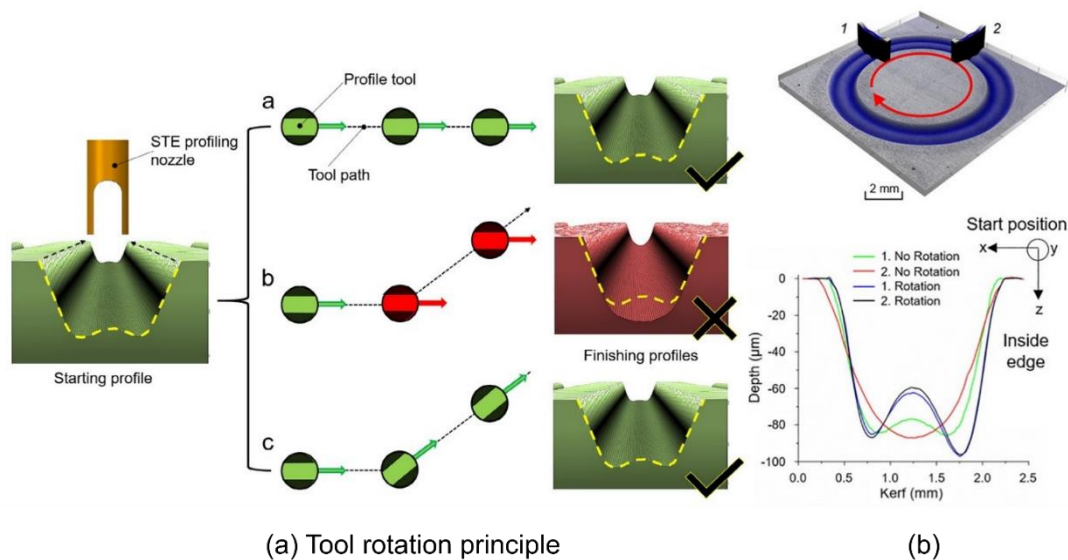


Fig. 2.10. (a) To retain desired profiles in complex toolpaths, rotational axes can be employed, (b) for example circular toolpaths. Taken from [48].

Analogous to modifying the nozzle, adapting the angle of address of the nozzle relative to the workpiece also causes redistribution of current density on the

work piece surface and can therefore increase the aspect ratio of resulting profiles (Fig. 2.11a-b) [20]. In addition, the focusing of current to the proximal workpiece locations to the nozzle enables bright surface finishes, where focused current densities are the last to interact with the workpiece ('push mode', Fig. 2.11c-e). Altering the angle of address also alters the hydrodynamic flow conditions and increases mass transport in the adjacent areas (see Section 2.3.2). Recently, Zhang et al. [49] investigated adapting the angle of address of a slot jet setup, in which one of the nozzle walls was electrically insulating, on the resulting surface and material removal characteristics. The authors showed that an address angle of 30° in 'pull mode' could enable mirror-like finishing, where the insulating wall was the last section of the nozzle to interact with the surface. In the opposite case, where the conductive wall was the last section of the nozzle to interact with the surface, surface quality was shown to be significantly worse than that prepared using the conventional setup.

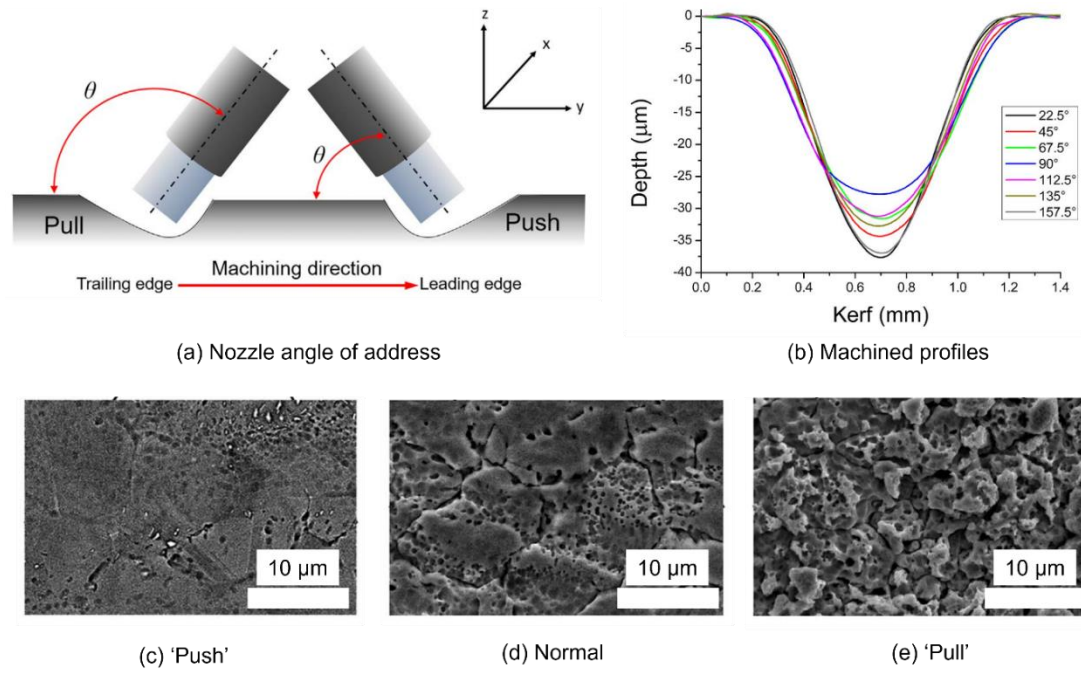


Fig. 2.11. Altering angle of address modifies the surface current density distribution. (a) The electrolyte jet can either be 'pulled' or 'pushed'. (b) Profile aspect ratios increase upon tilting the jet. (c) The high surface-defining current density in 'push' mode overcomes normal translation effects in EJM (d). (e) Operating in 'pull' mode deteriorates the surface. Adapted from [20].

Similarly, workpiece geometry can alter processed features. For example in EJM turning of steel rods, the profile widths and lengths were observed to decrease compared with planar surfaces, resulting from a change in the current density distribution [50]. Workpiece orientation relative to the nozzle was explored for non-planar geometries by Zhao and Kunieda [51]. The authors demonstrated that sharp corners and small radii of workpiece curvature enhance local current density distribution and removal depth (Fig. 2.12).

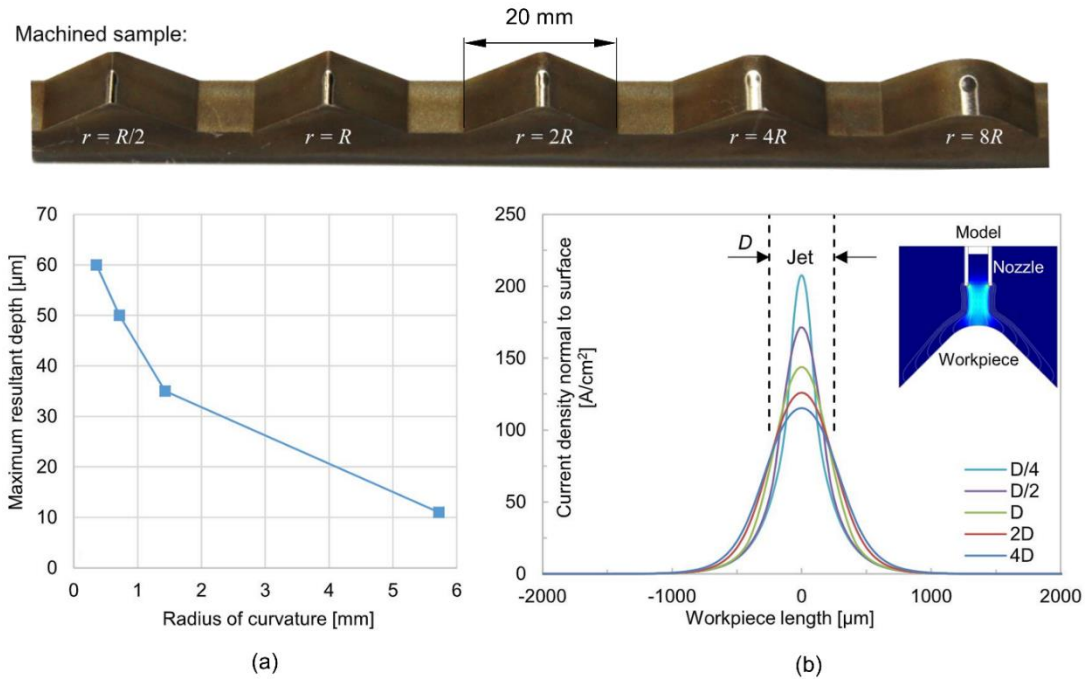


Fig. 2.12. Work piece geometry modifies the surface current density distribution. (a) Smaller radii of curvature increase the resulting dissolution rates, focussing current densities (b) over complex geometry surfaces. Adapted from [51].

An underexplored area relating to tool geometry for EJP is the design of the coaxial air pathway around jet. Xinchao et al. [10] proposed the compressed-air film encircling jet electrodeposition to reduce the jet diameter and homogenise the electrolyte flow velocity across the jet cross section. Approximately 30% reduction of jet diameter was reported through their air nozzle design and a corresponding reduction of diameter of circular copper deposits. The effects that the design of the coaxial air supply has on EJM has not been investigated in detail, apart from it being used to prevent electrolyte accumulation in the IEG and aid the formation of the thin film region.

Applying multiple electrolyte jets enables faster areal processing compared with single nozzle setups and is relevant for repeating patterns over large flat areas and is key to scaling up productivity. Ikeda et al. [31] investigated multiple nozzles in EJM, however the process is complicated by the interaction between

the discrete impingement regions of each nozzle. This is important, as both surface finish and dimensional accuracy are susceptible to changes in the electrolyte flow condition. When processing surfaces with a multi-nozzle approach, Luo et al. [52] observed stray corrosion in inter-nozzle areas, caused by delocalised current density distributions. Furthermore, the authors observed that interacting electrolyte jets led to droplet collection on the nozzles and rolling off into the IEG, temporarily disrupting the thin film region and delocalising material removal. As a solution, the authors used insulator-coated nozzles, reducing current density distributions between the nozzles, and accordingly stray corrosion. In addition, the authors observed that variation in dimensional accuracy was reduced at inter-nozzle gaps >2.50 mm, when using 0.5 mm diameter nozzle arrays. Fig. 2.13a shows micro-groove arrays generated by offsetting the nozzle array to overcome this limit (4.00 mm inter-nozzle gap).

In addition to using multiple nozzles, Chen et al. [53, 54] applied flexible insulated masks to a slot jet in EJM, to enable the creation of micro groove arrays (Fig. 2.13b-e). This approach significantly reduces the interaction between electrolyte flow fields, reducing shape variation and enabling smaller inter-groove spacing. The authors showed that the insulating mask reduced stray corrosion on the groove edges; however, the dimensional accuracy was affected by poor electrolytic product evacuation from the gap using this setup. This was overcome by the application of a pulse power supply, which also increased dimensional accuracy (Section 2.2.4).

Scale-up problems need to be addressed to apply EJP industrially, and the multiple nozzle approach is one such solution. However, this is challenged by complex flow field interactions, and non-planar workpiece geometries.

Therefore, this approach is currently limited to simple toolpaths on planar workpieces.

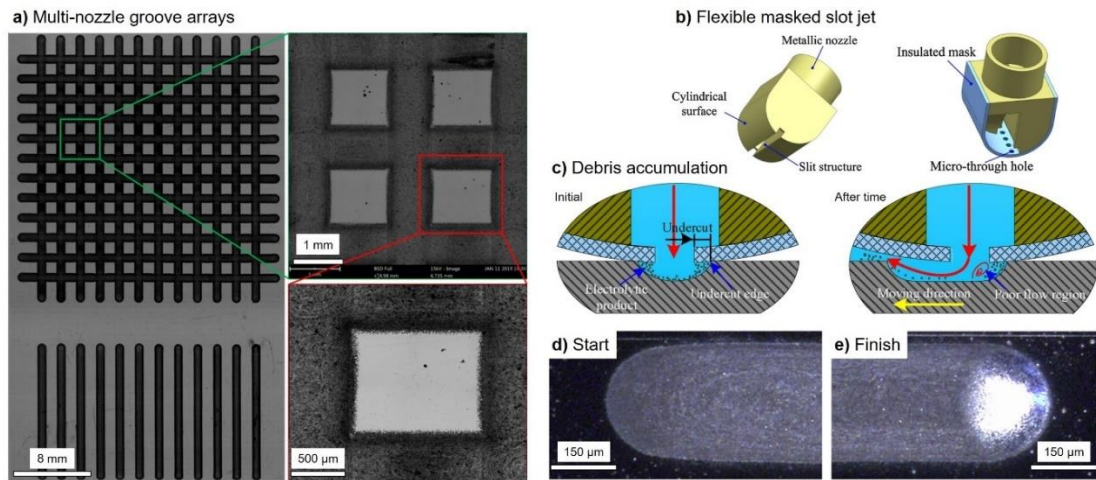


Fig. 2.13. Multiple nozzles enable rapid feature replication. (a) Multi-nozzle approach with insulating shielding, while inter-nozzle separation was 4.00 mm, passes were offset to generate features with 1.00 mm spacing and limited stray corrosion. (b) Flexible mask slot jet approach limits stray corrosion, (c) but results in debris accumulation from poor flow conditions. (d)-(e) This can be overcome by application of pulse power, leading to good surface finishes. (a) Taken from Luo et al. [52], (b)-(e) Taken from Chen et al. [53, 54].

2.2.3. Masked EJM

The influence of nozzle diameter and stray corrosion or stray deposition in EJP can be overcome by applying masks. Masking is applied in other electrochemical microfabrication methods, such as LIGA (*Lithographie Galvanoformung Abformung* – lithography, electroplating, moulding) [55], and through-mask electrochemical micromachining (TMEMM) [56], as the principle through which charge transfer can be localised. Briefly, in the LIGA process, a photoresist material is applied to the substrate surface and selectively irradiated by X-rays or ultra-violet (UV) light. After this, a solvent is used to degrade the irradiated areas of the photoresist, so that metal can be plated in the produced voids. Following the removal of the remaining photoresist, the patterned metal components can be used in replication processes such as injection moulding

and stamping. The process steps are similar in TMEEM, but instead of electrodeposition, substrate material is removed where uncovered by the photoresist mask. While EJP is selective by hydrodynamic restriction, applying masks can further miniaturise feature scales, disconnecting the process from the nozzle diameter. Masked impinging jet methods achieve greater mass transfer rates in comparison to lateral flow electrochemical processing [57], but at the expense of additional tooling and generally limiting the process to flat surface geometries.

By applying high-fidelity UV photoresist masks prior to EJM, arrays of pits and protrusions with significantly smaller diameters (0.1 mm) than the supply nozzle (2 mm) can be rapidly achieved on a repeatable scale (Fig. 2.14a-d) [58]. Protrusions are generated with the negative of the mask. Conductive masks have been applied to increase aspect ratios of micro-dimples [59] and grooves [60] from those achievable using insulating masks. Conductive masks were reported to stabilise the electrical field strength over the rim zone of the mask, preventing current density focusing on the rim contours, and reducing the growth rate in this area.

Masking therefore presents a practical solution to overcome nozzle diameter feature size limitations. However, efficiency is reduced at the expense of mask creation, which is often a multi-step process. Ultimately, masked EJM becomes a shape-replication method rather than a shape-creation method. This is a highly effective strategy for repeating pattern generation; however, it is challenging to apply the method to complex surface processing.

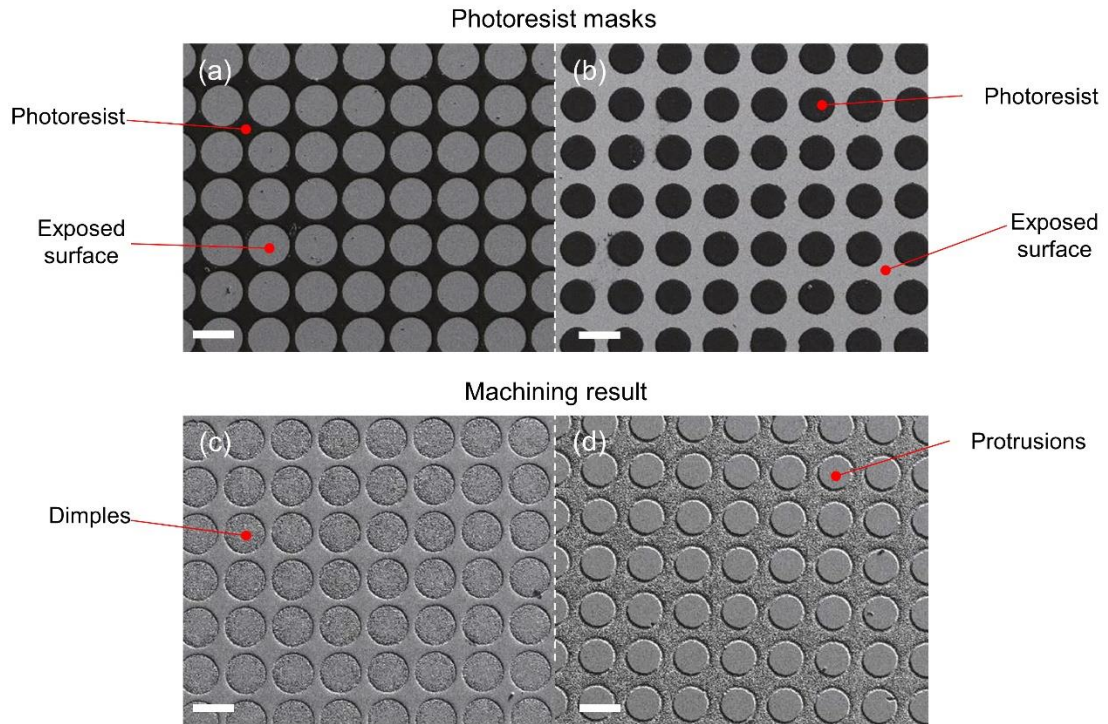


Fig. 2.14. Masking in EJM. (a)-(b) Photoresists ($100 \times 1.3 \mu\text{m}$) and resulting (c) dimples and (d) protrusions from EJM using a 2 mm nozzle. Scale bars correspond to $100 \mu\text{m}$. Taken from Wu et al. [58].

2.2.4. Power supply modes

The majority of EJM studies have applied direct current (DC) power, because continuous charge flow enables the greatest material removal rates, this is the simplest mode of power supply, introducing the fewest variables, and therefore steady-state phenomena can be appraised. Two distinct modes of power supply control are used by EJM researchers namely controlled current (CC) and controlled voltage (CV) mode dependent on the imposed electrical excitation to the IEG. The application of controlled current ensures constant mass transfer rate during processing, while under controlled voltage mode, the current density at the workpiece and respectively the mass transfer rate is sensitive to the geometry of the IEG and variation of the electrolyte conductivity. This can be beneficial dependent on the application, as under CV the removal

volume in EJM could be self-limiting with the removal rate decreasing when the depth of cut increases [7].

Pulsed power supplies have been shown to localise mass transfer and enhance electrochemical phenomena in the IEG in other electrochemical manufacturing methods, for example electrochemical micromachining to localise removal [61], and bath electrodeposition [62], and adaptation of the excitation waveform is beginning to be explored in EJP. The formation of the near-surface diffusion layer and the charging of the electric double layer are transient phenomena that can be modulated with a time-varying electric signal. In addition to DC current, pulse current and pulse-reverse current (bipolar) modes have been applied to EJP.

In EJM, the effect of changing power supply conditions on dimensional accuracy is relatively underexplored. Ye et al. [63] applied a pulse current to drill holes in a stainless steel sheet (0.5 mm thickness). While the authors observed no change in entrance hole diameters, aspect ratios of through holes were improved at the pulse parameters (50% duty, 2 kHz frequency). This was attributed the evacuation of dissolution products from the gap in the off cycle. Decreasing taper on application of pulse current has also been observed when processing Ni-superalloys [64]. Where flow conditions are poor, such as when applying masks, pulse current waveforms enable dissolution product evacuation from the anode surface, preventing stray machining [53].

Changing power supply condition has been exploited to enable EJM of WC-Co composites. Conventionally, this is achieved using caustic hydroxyl-based electrolytes, such as NaOH [7]. Hydroxyl anions dissolve WO_3 films that readily

form upon anodic polarisation of tungsten-containing materials. However, caustic electrolytes present additional health and safety concerns and are therefore less desirable in terms of handling and waste disposal. DC EJM of tungsten-containing materials is assisted by the electrolyte flux passing through the cathode nozzle. As the cathodic reaction generates both hydrogen and hydroxyl anions, these are transported to the anode surface through forced convection in the flow of the electrolyte jet where, oxide dissolution and subsequent machining is enabled, albeit at low rates (Fig. 2.15a). Removal rates were increased by Mizuagi et al. [65], who applied a bipolar pulse during processing. At the cathode polarisation, hydroxyl anions can be generated in-situ at the workpiece, activating the surface and enabling ECM on the inverse polarisation at appropriate pulse parameters (50% duty, 5 Hz) (Fig. 2.15b). Increasing duty factor was determined to increase removal selectivity towards the Co-matrix and decreasing duty factor increased removal selectivity towards WC-phases, attributed to greater hydroxyl concentrations generated on the cathode pulse. Bipolar current processing leads to anodic dissolution of electrically conductive nozzles, which can alter the current density distribution (Section 2.2.2), therefore the authors recommend insulating nozzles in this setup. Bipolar processing of WC-containing materials was also explored by Martin et al. [66] to achieve designed surface waviness (60-90% duty, 10 Hz frequency). EJM turning of WC-Co materials was applied to create micro-rods of minimum diameter 36 μm using a slot jet setup (50% duty, 10 ms pulse) (Fig. 2.15c) [67].

Kawanaka and Kunieda applied both pulse and bipolar currents to achieve mirror-like finishing [36]. In this study, the authors observed mirror finishes

when applying pulse currents (duty 10%, 100 μ s pulse, 1 mm/s), however process efficiency was limited by the low duty cycles required. By applying bipolar pulsing, the duty factor (and MRR) could be readily increased (duty 67%, 100 μ s pulse, 1 mm/s), while retaining a mirror-like finish at relatively low translation velocities. This approach was adopted using a slot jet to mirror finish large areas in stainless steel (67% duty, 100 μ s pulse) (Fig. 2.15f). The authors attributed the polishing effect to the higher double layer charging times associated with greater resistance in the low current density regions to reduce low current density effects. Bipolar currents were assumed to enable faster discharging of the double layer. However, the diffusion layer and surface concentration gradients are also pulsating [68] so it is challenging to discriminate the exact mechanism leading to surface texture improvement under time-varying excitation and hence further study is required here.

The cathodic pulse of the AC waveform has been demonstrated to allow the dissolution of certain oxide films. For example, Xue et al. [69] recently showed this could enable the machining of highly passivating Nb with a passivating electrolyte (NaNO_3). In this novel approach, the anodic pulse anodized the surface (converting the surface to Nb-oxide phases), while the cathodic pulse allowed the electrochemical reduction and removal of this film, in addition to H_2 gas evolution that aids the process through mechanical exfoliation of the oxide. This approach could have significant implications in the machining of alternative passivating materials such as Ti-alloys that feature widespread application in industry.

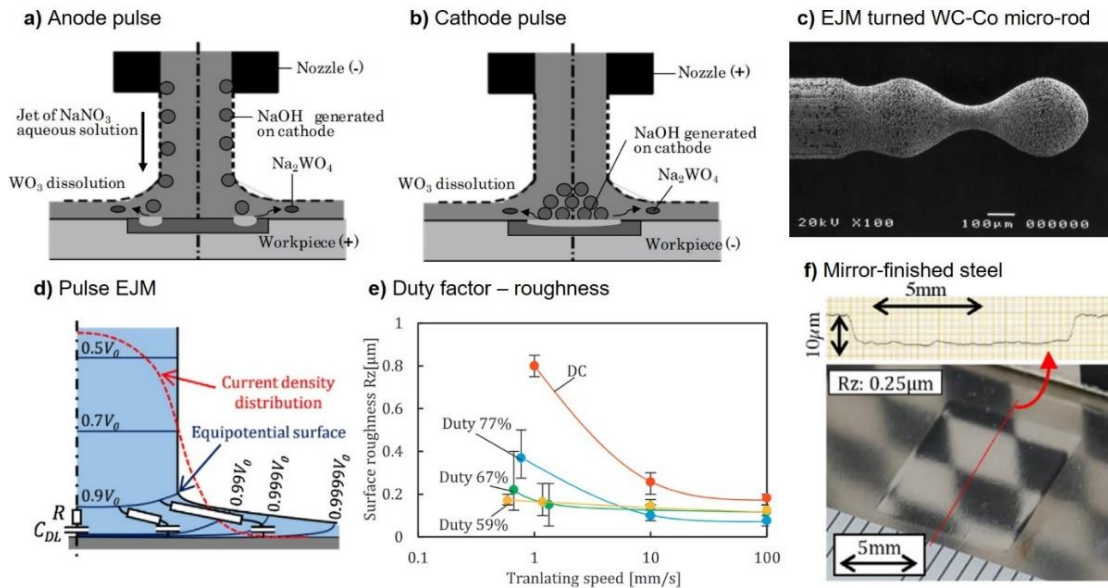


Fig. 2.15. Pulsed power adapts IEG electrochemical phenomena. (a) Cathode-generated hydroxyl anions are transported by the electrolyte jet to the workpiece in anodic cycles, or when applying conventional DC EJM. (b) Bipolar pulsing enables hydroxyl generation at the workpiece on the cathode pulse, enabling WO_3 film dissolution. (c) This approach was applied to turn WC-Co micro-rods. (d) At sufficiently low pulse on times, double layers cannot fully charge in low current density regions, enabling mirror-finishing when translating the jet (e)-(f). (a)-(b) Taken from [65], (c) taken from [67], and (d)-(f) taken from [36].

2.2.5. Electrolyte delivery and composition

The physical and chemical properties of the electrolyte along with the flow hydrodynamics have significant effects on EJM and the resulting surface quality. The electric conductivity of the electrolyte is typically modified by changing salt concentrations. For controlled voltage EJM, increasing conductivity increases current and therefore MRR in accordance with Ohm's law (Fig. 2.16a-c) [70]. This has been shown for stainless steels, Ti-alloys [71], and Ni-superalloys [30]. In fixed current processing, increasing electrolytic conductivity does not change MRR in most cases [72], but is associated with a drop in power consumption resulting from decreased resistance. Under controlled voltage conditions, current density varies with conductivity having

significant influence on surface finish. Sen and Shan [30] observed an increase in roughness with increasing electrolyte concentration, which was more marked at higher applied potentials (Fig. 2.16d). Even at a fixed current density, modifying electrolyte concentration would affect the near-surface concentration gradients and is expected to influence surface finish. Experimental studies to isolate such effect of concentration in EJM are limited. Dimensional accuracy of ECM processes generally decreases upon increasing conductivity [73, 74] under controlled voltage. This is the result of increasing local current densities in the extremities of the central impingement region, which increases overcut, stray corrosion, and roundness error (Fig. 2.16e). This is not observed in all cases, for example when processing composite materials such as WC-Co with alkaline electrolytes [7].

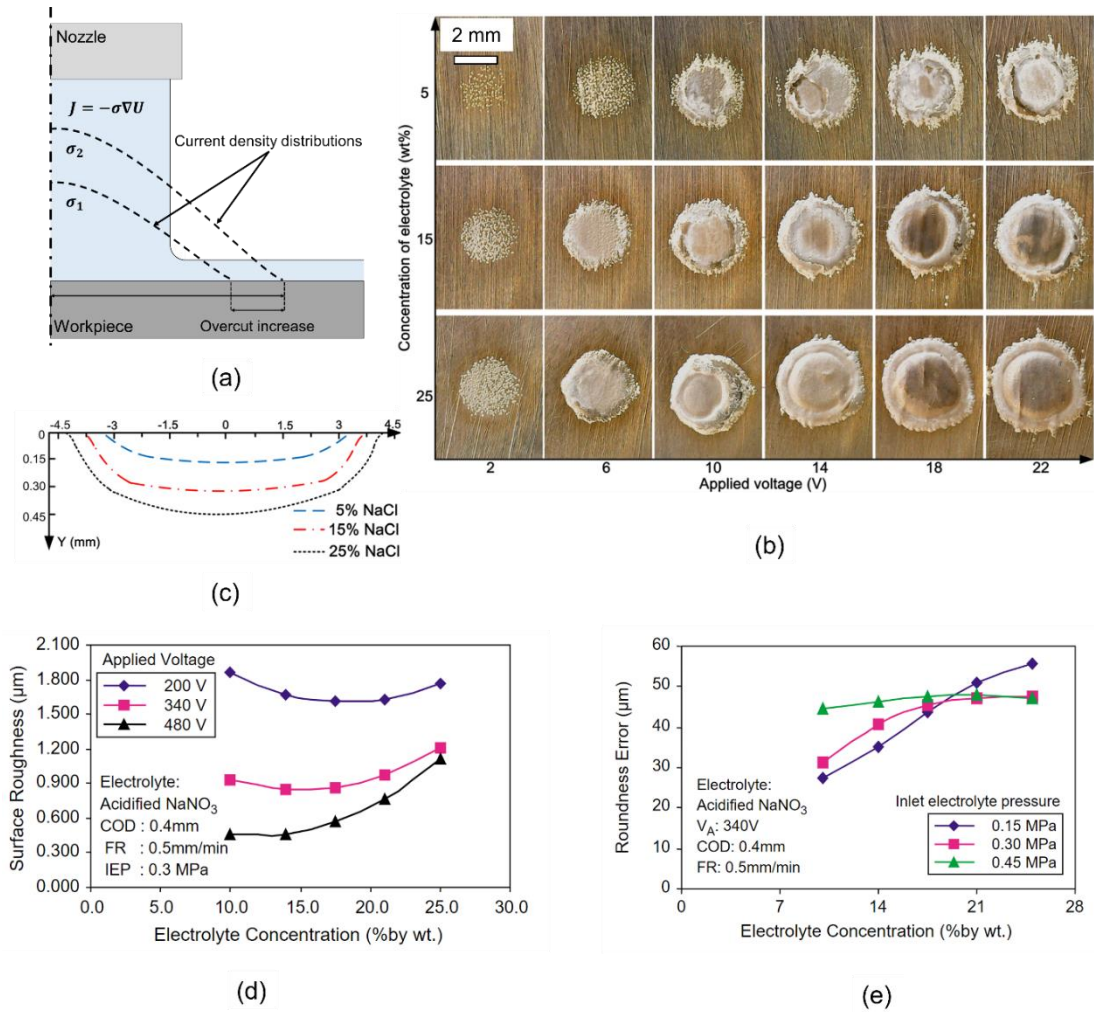


Fig. 2.16. Effects of electrolyte concentration on EJM. (a) Increasing electrolyte conductivity directly affects the primary current density distribution under controlled voltage conditions (b) Photographs of pits machined in Ti-alloy TB6. (c) selected pit profiles in the same material, for different electrolyte conductivities. (d) Surface roughness and (e) roundness error shown to increase in Ni-superalloy on increasing conductivity. (b)-(c) Taken from [70] (d)-(e) taken from [30].

Electrolyte viscosity directly affects mass transport rates in electrochemical processing. Increasing electrolyte viscosity decreases diffusion rates, which enhances anodic film formation and associated brightening effects. However, there is also a reduction in the efficacy of product removal, where precipitates such as metal hydroxides may influence the current density distributions in the IEG. This can lead to unstable removal profiles, especially in high-rate removal processes. Furthermore, viscosity affects the formation of the thin film

electrolyte region, in turn affecting selectivity. Ultimately, where the associated hydraulic jump cannot be generated at sufficient radial distance from the nozzle, mass transfer delocalises from the central impingement zone such that selectivity and process control is lost. However, Liu et al. [75] showed that increasing electrolyte temperature stabilises the thin film (by reducing the viscosity) in EJM when applying viscous ethylene glycol based electrolytes (Fig. 2.17a-b).

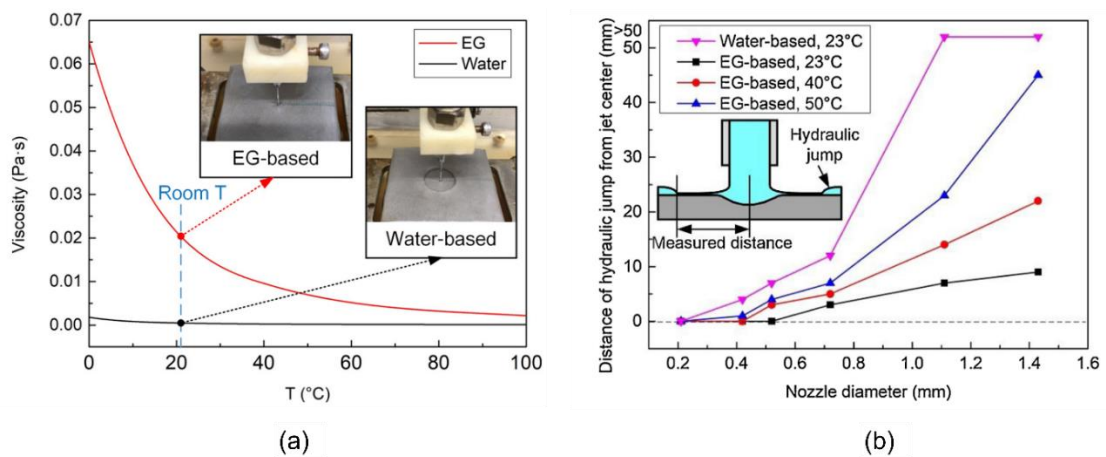


Fig. 2.17. Viscosity influences the thin film region. (a) Viscosity-temperature graph for aqueous and non-aqueous electrolytes in EJM, showing no thin film at room temperature with ethylene glycol. (b) Localisation can be retained by raising electrolyte temperature or increasing nozzle diameter. Taken from [75].

Jet velocity affects product and gas evacuation, and selectivity. Typical average flow velocities applied for EJM are usually in the range of 5 – 20 m/s [20] which translates to different flow rates dependent on nozzle cross-sectional area (e.g. 2.4 ml/min – 10 ml/min for an 0.1 mm diameter nozzle, 240 ml/min – 1000 ml/min for a 1 mm nozzle). Poor control over electrolyte velocity in ECM often leads to flow-affected surface structures [76], however such effects are largely unreported in EJM, likely due to unidirectional electrolyte evacuation through prior-machined grooves during translational processing. Jet velocity can affect

the resulting profile as shown in machining trials in Inconel 718 [20] although the difference is slight when applying velocities >10 m/s (Fig. 2.18a). The jet velocity also changes the shape of the reflected jet in EJP, which affects the removal profile. Hackert et al. [11] modelled and experimentally verified the fluid dynamics of the electrolyte jet upon anode surface evolution in pit EJM. The authors observed that upon machining to sufficient depths, the electrolyte thin film domain separates from the anode workpiece and can contact the nozzle (Fig. 2.18b-c). This reflected jet was shown to induce secondary machining, leading to increased pit diameters.

Recent attempts to understand hydrodynamic regimes in EJM involve the manipulation of the shape of this reflected jet. For example Wang et al. [77] correlated jet shape with linear charge density in Inconel 718, using a 0.8 mm nozzle. The authors observed different reflected jet regimes that had a significant effect on stray corrosion, where the reflected jet shape is itself dependent on the removal geometries (Fig. 2.18d-e). Care must be taken when interpreting these results however, as the investigation neither studied the influence of incident jet velocity, nor nozzle diameter. Jet velocity, along with removal profile, underpin the shape of the reflected jet, while nozzle diameter dictates the removal profile for a given linear charge density. As such, absolute correlation of linear charge density with jet shape is challenging. It is likely the study of areal charge density, incorporating the nozzle area, will allow a better correlation of removal profile to jet shape. Liu and Qu also exploited beneficial jet reflection to improve machining accuracy and surface texture when processing Ti-alloys in a sinking-scanning process [78]. Furthermore, Zhang et al. [79] reported that the orientation of the nozzle relative to gravity, but normal

to the workpiece, affected both roughness and overcut in pits. The authors reported that when applying the electrolyte jet in the horizontal mode, the reflected jet separated from the workpiece more readily, leading to further localisation of mass transport in comparison to vertical downstream (conventional EJM) and vertical upstream operational modes.

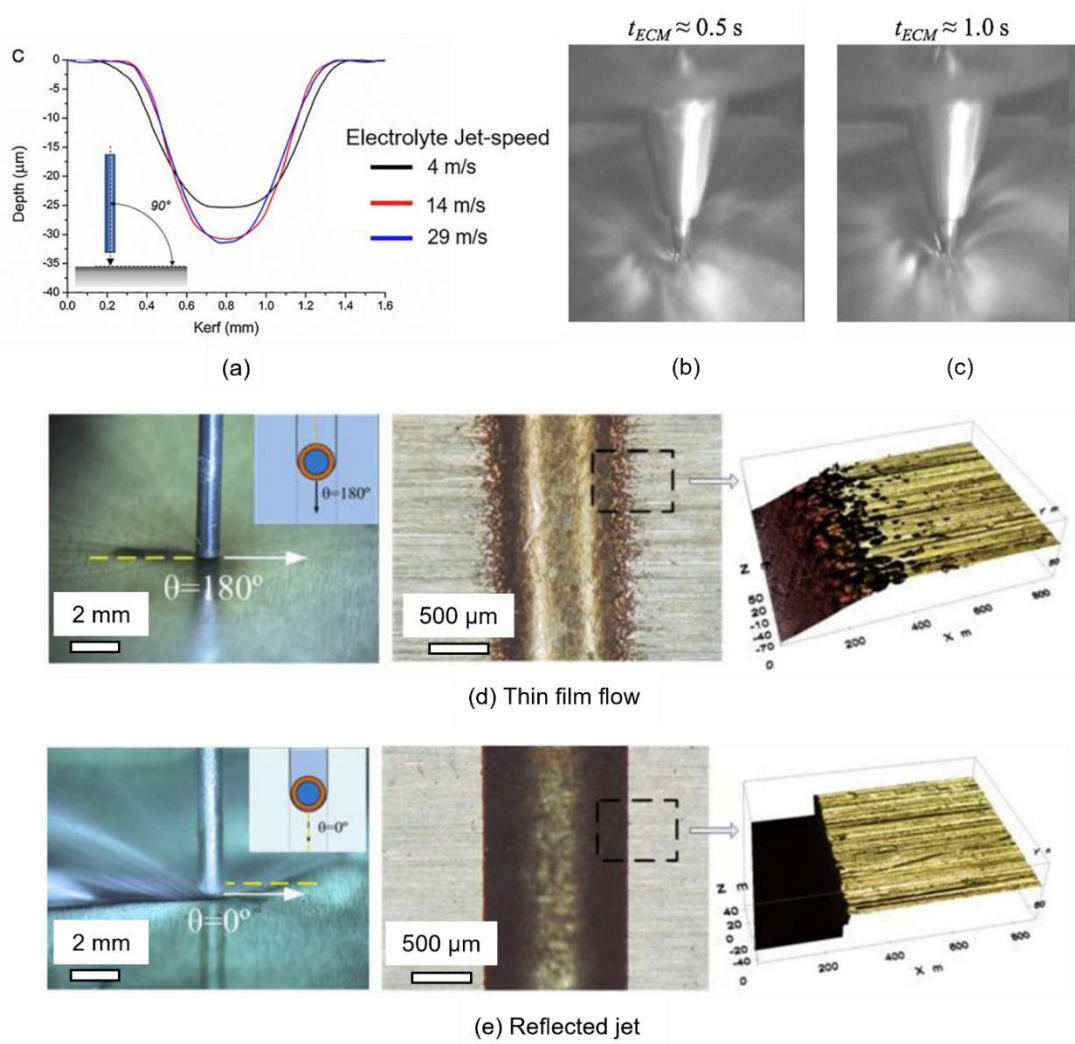


Fig. 2.18. Electrolyte velocity affects profile and stray corrosion. (a) Profile broadening at low jet velocity, likely caused by poor flushing of machining products. Images of the jet at (b) the start of machining and (c) once a certain depth is attained causing jet reflection. (d) Without jet reflection, stray corrosion can result at groove edges, (e) jet reflection arrests this phenomenon. (a) Taken from [20], (b)-(c) taken from [11], (d)-(e) taken from [77].

Electrolyte chemistry defines the reactions that occur at both electrodes and the stability of surface oxide films. Dissolution products, evolved gases, and sludge precipitation in the IEG influence local current density distributions and must be removed in ECM processes. High jet velocities and local turbulence aid dissolution product evacuation in EJP. This is assistive and enables EJM of most relevant engineering materials (e.g., steels [80], stainless steels [37], Ni-superalloys [20, 46], and Al-alloys [43], metal matrix composites [81] etc.) to be conducted in neutral aqueous salt electrolytes, predominantly NaNO_3 and NaCl .

In ECM, a common distinction is drawn between ‘passivating’ and ‘aggressive’ electrolytes, where the former enables the formation of a beneficial oxide film that serves to brighten the surface finish. Aggressive electrolytes destabilise oxide films by electrostatic migration of the anion (like Cl^-) through the oxide, which can lead to pitting. Oxide film formation in passivating electrolytes enables a degree of oxygen evolution, causing lower current efficiencies compared with aggressive electrolytes, as well as a strong dependence of current efficiency on current density. In aggressive electrolytes, current efficiency is less dependent on applied current density; in practice, in EJM, this means that material removal is promoted across the current density distribution. This leads to increased overcut (the ratio between nozzle diameter and feature diameter) when machining with chloride-based electrolytes in comparison to nitrate-based electrolytes.

Overcut has been further decreased by chemical means in EJM, for example, Mitchell-Smith et al. [82] applied iodide-doped electrolytes towards Inconel 718. The authors exploited the facile nature of anodic iodide oxidation: $\text{I}_{(aq)}^- \rightleftharpoons$

$\frac{1}{2} \text{I}_{2(l)} + e^{-}$, forming a poorly soluble supersaturated iodine film at the anode.

This controlled secondary reaction eliminated low current density areas through an effective chemical filter mechanism, reducing taper, albeit at the expense of MRR. Film thickness was correlated with the incident dopant concentration (mol/s) by resistance measurements, studied by adapting the jet velocity. Clare et al. [83] complemented this approach with other precision-enhancing adaptations, like profiled nozzles and manipulated angles of address. As different electrolytes influence surface finish, dimensional accuracy, and removal rate, it may be beneficial to dynamically modulate impinging jet chemistry. Mitchell-Smith et al. [8] demonstrated that this can be achieved by drawing electrolytes from multiple tanks during processing.

ECM is challenging in some key engineering materials (e.g., Ti-alloys and WC-Co composites), which readily form oxide films on anodic polarisation. These materials are difficult to micro-machine and process through conventional shear-based means; therefore, non-conventional methods present an opportunity in this regard. WC-Co materials can be processed using NaNO_3 by application of bipolar pulse power (Section 2.2.4), avoiding the requirement for alkaline electrolytes that dissolve the passivating WO_3 anodic films [84].

EJM of Ti-alloys is typically performed in chloride electrolytes, as nitrate electrolytes generate thick TiO_2 surface films that disrupt dissolution [72, 85, 86]. This can however, be partially overcome by high applied currents and extended dwell times (e.g. 140 A/cm^2 , 60 s) [87], or increasing the nitrate concentration [72]. Regarding concentration, Liu et al. [70] reported a decrease in oxide film breakdown time with increasing chloride concentration, however it

is unclear if this effect is related to corresponding increases in current density at the fixed voltages (2 – 24 V).

Other halide anion-containing electrolytes (bromide and iodide), have been observed to improve ECM performance of Ti-alloys by destabilising the oxide layers [88, 89]. Lu and Leng [90] applied a high concentration (5 M) NaBr electrolyte to machine pits in Ti-6Al-4V at high applied potentials (>100 V) on a curved surface. Speidel et al. [72] showed that neutral Br-based electrolytes reduce pit widths and secondary machining effects at high current densities (>400 A/cm²), in comparison to neutral NaCl electrolytes, albeit for a reduction in MRR (Fig. 2.19a-b). Further work investigated the role of doping halide electrolytes together to improve machining performance [91]. The authors demonstrated reductions in overcut and stray machining when applying iodide-doped chloride electrolytes at high current densities (>200 A/cm²) (Fig. 2.19c-d). The oxide breakdown mechanism was proposed to be accelerated by aggressive halide electrolytes (Br⁻, I⁻), which promoted pitting corrosion ahead of the translating nozzle. This leads to periodic potential oscillations and highly regular pitting phenomena indicative of undesirable machining regimes at lower current densities (<150 A/cm²).

Liu et al [75] applied a non-aqueous electrolyte (5 wt.% NaCl in ethylene glycol) towards Ti EJM with a pulse current power supply, reporting performance improvements. While the authors observed opposite current efficiency relationships compared with aqueous NaCl electrolytes under equivalent parameters, improvements in finish, and reduced surface oxidation was reported (Fig. 2.19e-g). The high electrolyte viscosity, enables brightening conditions, aiding surface finishing. This is supported by surface observations

under pulse current conditions, where short on-times relative to off-times lead to rough surfaces.

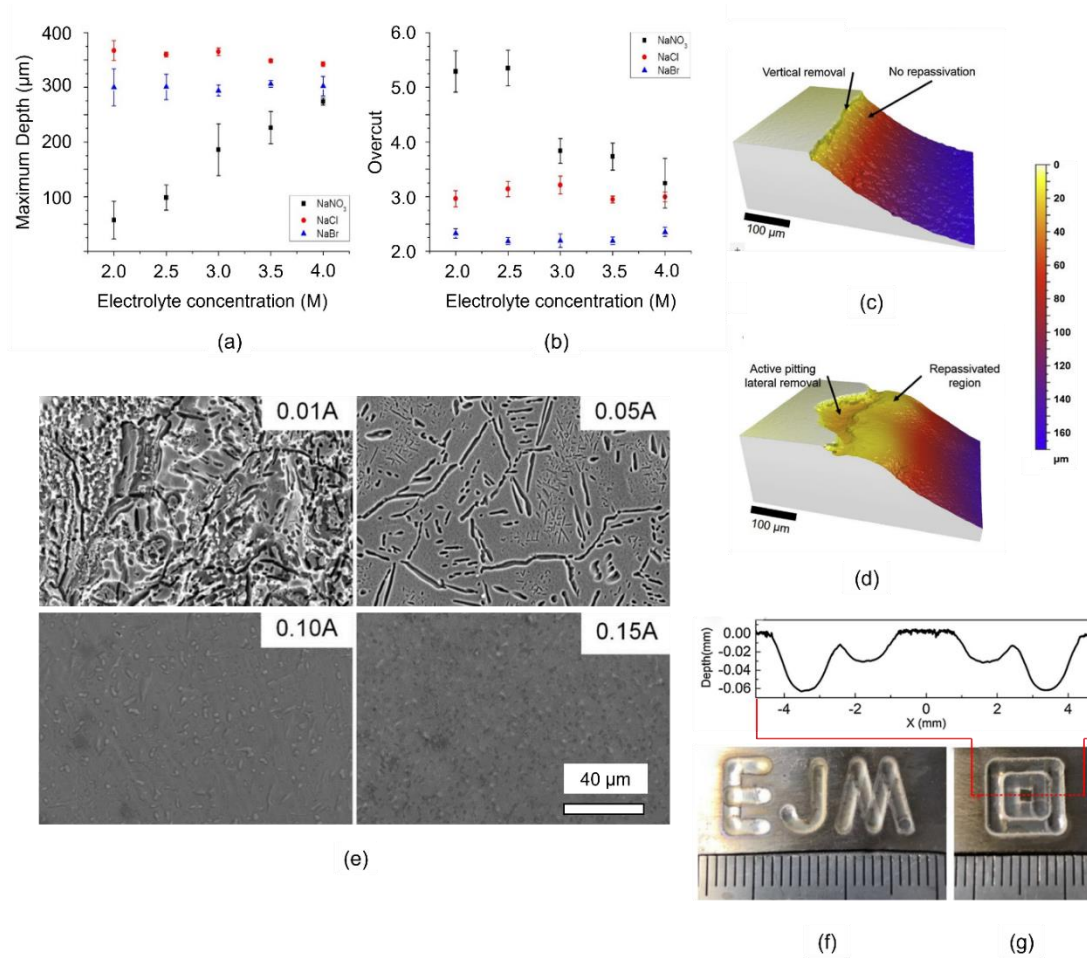


Fig. 2.19. EJM of Ti-alloys in different electrolytes. (a) Pit depths and (b) overcut in Ti-6Al-4V (408 A/cm^2 , 10 s). (c) Application of iodide-doped electrolytes reduces overcut, compared with NaCl and (d) NaF-doped electrolytes at high current densities (300 A/cm^2). (e) Non-aqueous electrolytes promote polishing (0.01-0.15 A, 2 mm/s) and (f)-(g) can enable direct writing and shape generation in Ti-1023 alloy. (a)-(b) taken from [72], (c)-(d) adapted from [91], (e)-(g) taken from [75].

2.3. Modelling of EJM

Significant research efforts have been devoted to the modelling of EJM in order to improve understanding of the process mechanisms and inform parameter selection.

2.3.1. Electrical simulations

The main objective of process simulation of EJM is the prediction of the surface geometry resulting from the machining operation. The surface evolution of the anode is governed by

$$\mathbf{v}_d = \eta \frac{k}{\rho} \mathbf{J} \quad (2.11)$$

where $\mathbf{v}_d$ is the dissolution velocity, $\mathbf{J}$ is the current density at the anode surface, η is the current efficiency, k is the electrochemical equivalent of the work piece material and ρ is its density. Since the current density vector is normal to the surface of the work piece, the velocity given by the above equation is also in the direction normal to the evolving work piece surface. For the purposes of shape prediction in EJM the electrode overpotentials are often considered negligible compared to the overall potential difference across the IEG [12] and no distinction is made between the bulk of the electrolyte and the near-electrode region assuming that Ohm's law (Eq. (2.6)) is valid everywhere in the electrolyte. The current density and potential distributions can then be obtained by solving Laplace's equation

$$\nabla^2 U = 0 \quad (2.12)$$

within the electrolyte domain with the appropriate boundary conditions (Fig. 2.20). This a non-trivial task and necessitates the application of numerical methods such as the finite-difference [12] or more commonly the finite element method [39]. Knowledge of the shape of the impinging jet is required for this approach. In early EJM modelling studies [12, 39] a static shape is assumed for the electrolyte jet. Making use of the rotational symmetry about the jet centre line, Eq. (2.12) was solved numerically over a two-dimensional domain to

obtain current density on the work piece surface as a function of radial position from the jet centre (Fig. 2.20).

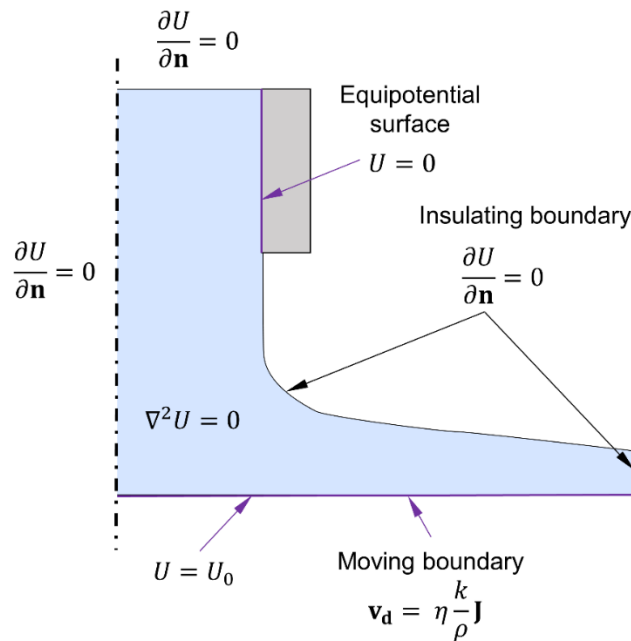


Fig. 2.20 Typical computational domain and boundary conditions for electrical modelling of EJM. Laplace's equation for electrical potential holds everywhere within the electrolyte with the anode and cathode considered as equipotential surfaces. The rotational symmetry allows solution in cylindrical coordinates.

Yoneda and Kunieda [39] simulated the process under controlled current, where the current density distribution on a flat surface was initially calculated from assumed initial potential at the boundaries and then integrated to obtain the total current. This process was iterated, adjusting the potential to achieve the desired set current. Following experimental calibration of the electrochemical equivalent and current efficiency for anodic dissolution of iron in sodium nitrate, it was demonstrated that this model can predict machined pit depth within 10 %. Kozak et al. [12] simulated the current density distribution at different ratios between IEG and nozzle radius, showing that when this ratio is smaller than 0.5, the peak of the current density distribution resulting from a cylindrical nozzle occurs under the nozzle rim instead of at the centre. The

authors also proposed a model to account for conductivity changes due to Joule heating of the electrolyte and estimate the maximum achievable removal rate for a given allowable temperature increase of the solution. The static electrolyte boundary simulation approach has remained a widely-used tool to predict current density distributions and inform the design of tooling and process variants such as through-mask [59] and multiple-electrode EJM [52].

2.3.2. Fluid dynamic and multiphysics simulations in EJM

More recently, the availability of commercial multiphysics simulation software has allowed for accurate modelling of the hydrodynamics of the impinging jet through the Navier-Stokes equations before solving for the electrical potential and current density distributions. Hackert et al. [11] investigated the change of jet shape during a pit machining operation. The simulation of jet shape is a two-phase flow problem, where the Navier-Stokes equations are solved for the level set function ϕ which takes a value from 0 to 1 within the solution domain, with 0 corresponding to electrolyte and 1 corresponding to air (Fig. 2.21a) and $\phi = 0.5$ at the interface. The subsequent current density calculation provides an insight into the influence of the evolving surface geometry on the material removal rate, which becomes more uniformly distributed over the affected area as the depth of the machine pit increases (Fig. 2.21b). A decrease in current is also seen as the process is simulated in the controlled voltage mode. The results also confirm the experimentally observed phenomenon at large depth of cut when the electrolyte is ejected from the side walls of the machined feature re-contacting the nozzle.

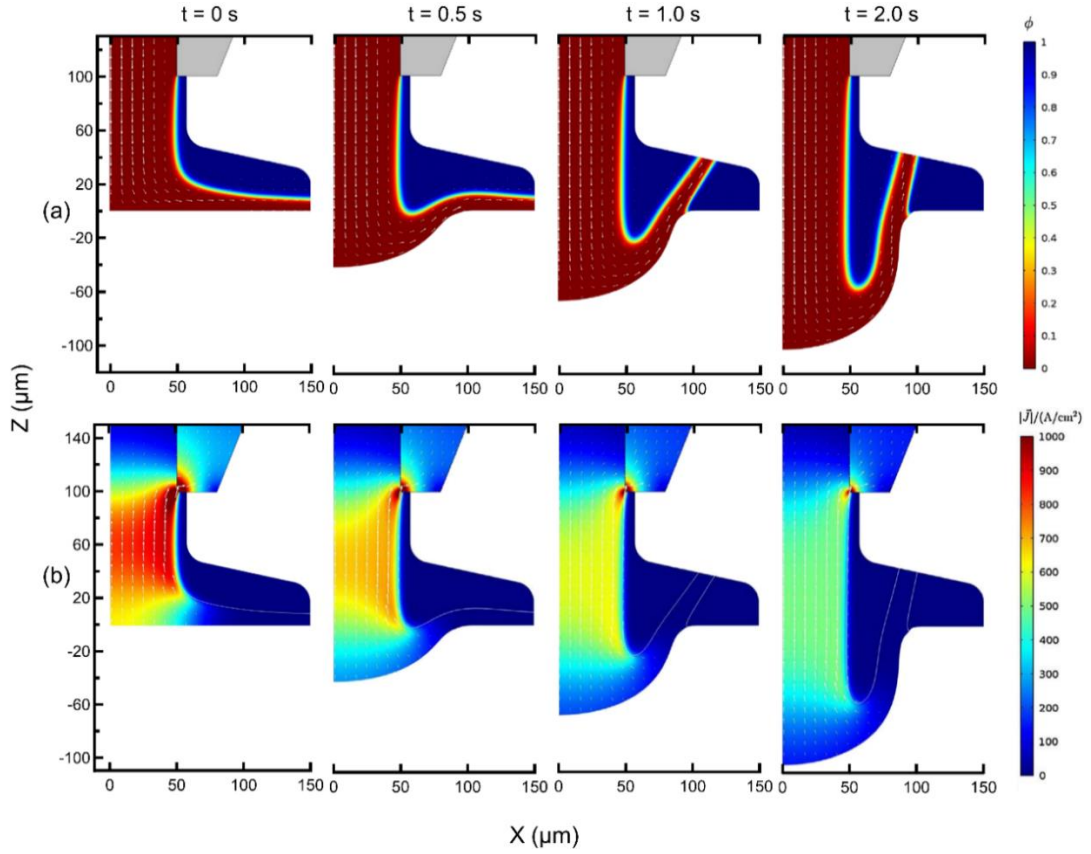


Fig. 2.21. Multiphysics simulation of static machining with electrochemical jet machining performed by Hackert et al. [11]. (a) Computational fluid dynamic simulation of the jet shape as function of time t . (b) Corresponding current density in the jet. Reproduced from [11].

Zhao and Kunieda [51] employed a similar simulation approach to understand the interaction of a flat jet with non-planar work piece surfaces and the alteration of current density distribution resulting from the slope and radius of curvature of the surface. Three-dimensional simulations of fluid flow have been performed by Liu et al. [92] of the inclined nozzle configuration of EJM to investigate the alteration of the thin film and the stagnation region of the jet. As the angle of address to between the jet and the surface decreases (Fig. 2.22a-c) the thickness of the thin film region reduces behind the jet and increased in front of the jet with an observed shift in the stagnation point. The velocity distributions within the computational domain also demonstrate that the region of low electrolyte velocity is reduced at low angles of address leading to

more effective mass transport. The simulated current density distributions agree with the cut geometries observed by Mitchell-Smith et al. [20].

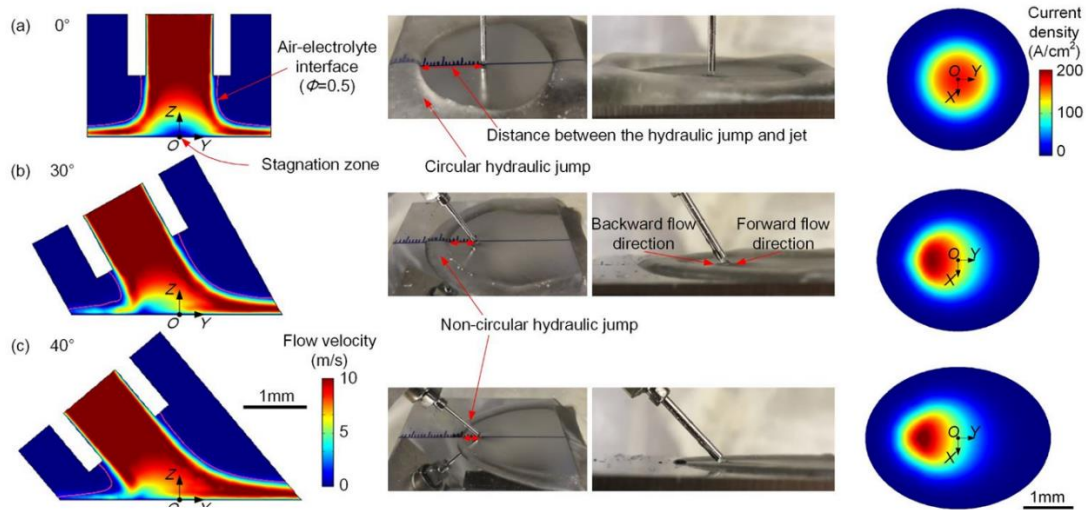


Fig. 2.22. Electrolyte velocity distribution and corresponding current density distribution for inclination angles of (a) 0°, (b) 30° and (c) 40° measured from the work piece surface normal.

2.3.3. 3-dimensional removal simulations in EJM

The multiphysics models discussed above provide valuable insights to the fundamental mechanisms of EJM, but they investigate the current density distribution and material removal without nozzle movement, which is not reflective of most practical machining operations. Guo et al. [93] first applied a 3-dimensional finite element model with a moving nozzle to simulate the shape of three dimensional channels machined with both standard and shaped nozzles in EJM. A hydrodynamic model was not considered, so a simplified static shape was used. To improve the accuracy of the simulated channel cross-sections, experimentally obtained current efficiency data as function of current density. An interesting result of their work is the insight into the current density distribution in translation and the advancement of dissolution in the direction of motion (Fig. 2.23a). Focusing of current density is observed near

the front of the moving jet where the removal depth is lower. This has been also demonstrated in the two-dimensional simulation of channel machining with a masked slot nozzle performed by Chen et al. (Fig. 2.23b) [53].

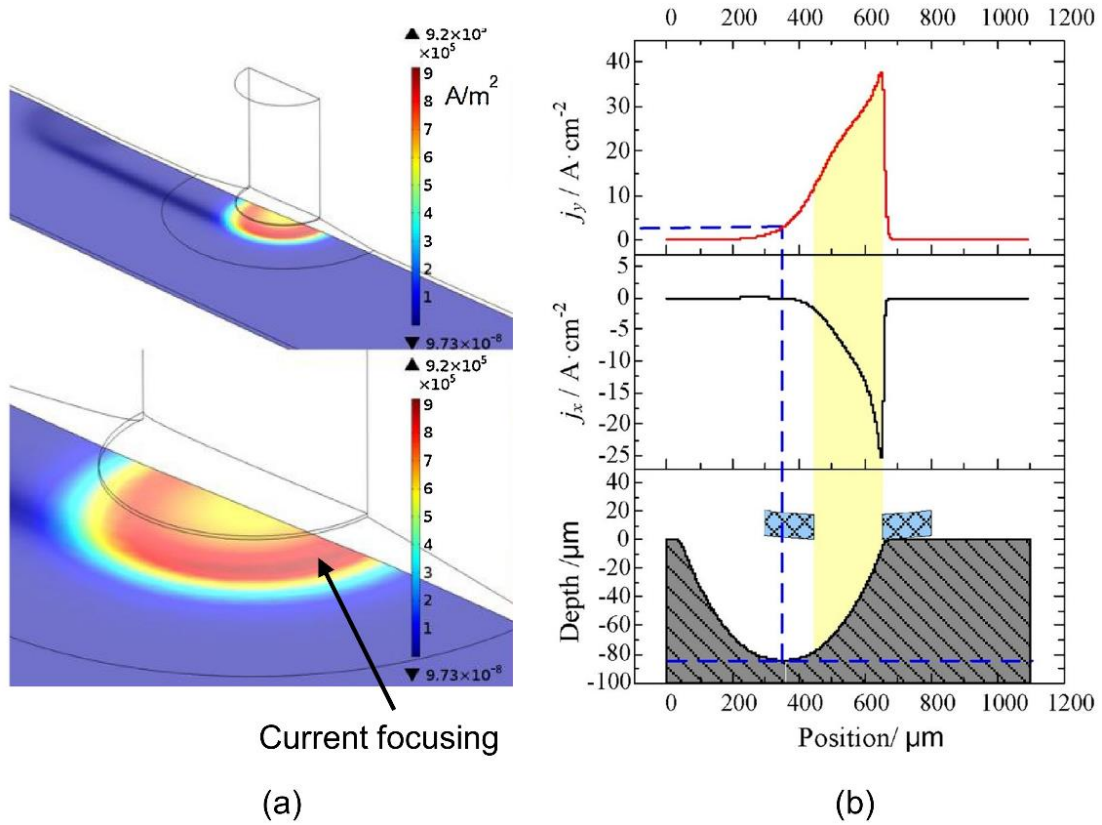


Fig. 2.23. Current density distribution during channel machining in: (a) EJM as simulated by Guo et al. [93]. (b) through-mask EJM as simulated by Chen et al. [53].

The simulation of longer and more complicated tool paths corresponding to practical surface texturing operations becomes more challenging and computationally expensive for the finite-element methods discussed. Analytical models with simplified assumptions have been proposed that are more suitable for integration with machine control and path planning software. Natsu et al. used the experimentally obtained profile of single straight groove to calculate the tool path and the required feed rate for a superimposed surface geometry [32]. The same approach was applied to the prediction of curved tool-paths [38].

2.3.4. Material influence in simulations

The microstructure and the electrochemical properties of the work piece material significantly affect the performance of EJM, but the detailed consideration of this influence in material removal simulations is challenging. Simulations based on Ohm's law do not consider in detail the reacting species for the specific material-electrolyte combination. The material influence on removal is considered through the electrochemical equivalent and prior knowledge of the current efficiency of dissolution as function of current density to substitute in Eq. (2.11) [11, 39, 93]. This approach is adequate for the overall cut shape of homogenous single-phase material, but for a multi-phase material the electrochemical kinetics of individual reacting phases may become important dependent on the grain size [18]. The presence of passive oxide films in certain materials also significantly affect the process. Liu et al. [94] considered the electrolyte jet machining of the titanium of alloys by including a correction for the passivating oxide break down. The proposed simplified approach tracks the local charge transferred per unit area of the surface (charge density) as the jet is traversed. Removal only occurs where the charge density exceeds a specific threshold value, corresponding to the breakdown of the passivating film prior to anodic dissolution. This methodology resulted in a more accurate prediction of depth of cut compared to directly applying Faraday's laws.

2.4. Applications of EJM

2.4.1. Surface functionalisation and finishing

Although the adoption of EJM for industrial applications is currently limited, the potential for surface functionalisation through topographic and chemical

modification through EJM has been demonstrated in literature. Both the large-scale surface features of the order of nozzle size and the microscale surface texture due to the electrochemical interaction with the work piece produced through EJM have been utilised. Yang et al. [95] used EJM to modify surface wettability by patterning an electrochemically etched Al surface with hydrophilic micro-dimples. Subsequent coating with stearic acid, to lower surface energy, resulted in water droplet pinning. The authors demonstrated control over droplet sliding angle depending on EJM processing parameters of applied potential and dwell (100 – 600 V, 1 – 20 s). Certain mechanical properties of surfaces can be enhanced using EJM approaches. Walker et al. applied EJM to texture Al-Si cylinder liner material with arrays of micro-pits [81]. The authors showed that when pits were aligned parallel to the sliding direction, the coefficient of friction, tribo-film formation, and wear coefficients were reduced. This effect was attributed to a lubricant reservoir effect [96], exploiting the fine features achievable with EJM.

Complex microscale surface textures created with EJM have recently been shown to have antimicrobial properties. For example, Lutey et al. indicated a reduction larger than 90% in the surface retention of bacteria when compared with a rolled stainless steel surface [97]. This effect is likely to hold where the surface topography feature sizes approach the length scale of the microbe [98].

EJM has been investigated as a possible finishing tool for laser powder-bed fusion (LPBF) parts fabricated from Ti [99]. The authors reduced the surface roughness from $18.6 \pm 2.0 \mu\text{m Sa}$ (as built) to $2.4 \pm 0.2 \mu\text{m Sa}$ during finishing and micro-machined pit arrays into the surface. Debris analysis indicated erosive removal mechanisms, where individual unfused powder particles were

detached and washed away in addition to corrosive electrochemical removal. Wang and Qu applied EJM as an anodic levelling process to finish Inconel 718 surfaces, produced by direct energy deposition, which often exhibit relatively large scale waviness [100]. In this study, a small IEG and high potential were determined to benefit levelling. Cheng et al. applied a combined machining and deposition approach to machine and electrodeposit material as part of a finishing operation for powder bed fusion parts [101].

2.4.2. Metallographic etching and analysis

Information regarding material properties obtained from polycrystalline materials are highly sought after as they define suitability to end application [102]. Obtaining this information using conventional methods like transmission electron microscopy (TEM), electron backscatter diffraction (EBSD), or X-ray diffraction is often costly and time consuming due to the nature of the technology and sample preparation time. An EJM approach to prepare and thin samples to electron transparency for TEM inspection has existed for over half a century, for example Chase et al. jet polished GaP semiconductor samples in a chlorine-methanol mixture [103]. This jet approach is rapid (i.e., taking seconds), although it does not allow control over site-specific analysis like focused ion beam milling.

Additionally, EJM offers the opportunity to selectively etch and reveal the microstructures of various metals and alloys without hazardous chemical etchants. For example, Speidel et al. [35] exposed the surface topographies of Al-6082 and Ni samples by etching with a translating jet from which the crystallographic texture can be inferred (Fig. 2.24a). Etching sample preparation time can be increased by using a slot jet to etch the surface and

prepare large surface areas (e.g., 160 mm²) [104], where appropriate parameter selection allows surface measurement with EBSD with no further preparation (Fig. 2.24b).

The microstructure of the workpiece can significantly affect the resulting surface after EJM. Lu et al. [105] investigated the resulting surface topography, removal characteristics, surface chemistry, and texture pattern of rolled stainless steel. The results showed how rolling direction and surface orientation of stainless-steel impacts the anisotropic behaviour of texture produced (Fig. 2.24c). Further applications of electrochemical jet surface processing include defect finding, unveiling of crystal microstructure and fusion mechanisms of process induced volume defects with parts additively manufactured using LPBF [106]. The authors showed very effective localised microstructural unveiling which allows for rapid identification of internal defects. This method simultaneously characterises defects in the micrometre range and crystallographic microstructure in the nanometre range, a schematic setup of the experiment is shown in Fig. 2.24d.

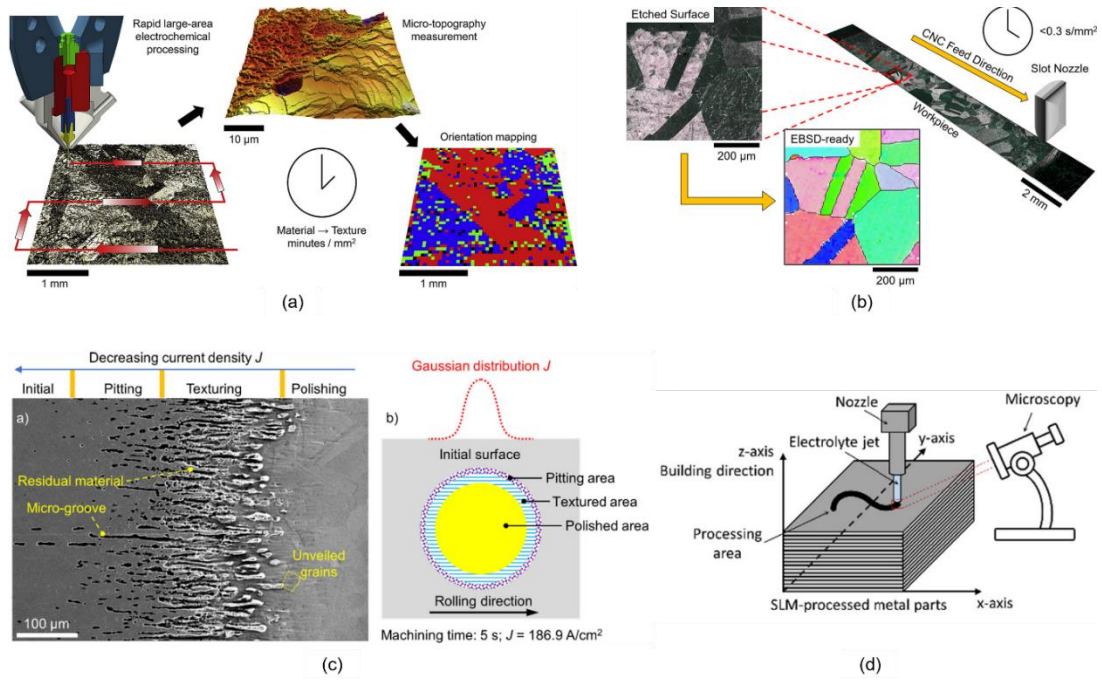


Fig. 2.24. Etching applications in EJM. (a) EJM etching and subsequent topography measurements and the analysis to map crystallographic texture. (b) Resultant surface after etching using slot jet allows faster surface preparation and subsequent EBSD readiness without additional sample preparation. (c) Transitional surface topography at the boundary of machined area and schematic illustration of different dissolution behaviours owing to the varying current density. (d) Schematic of the principle for inspecting LPBF parts. (a) taken from [35], (b) taken from [104], (c) taken from [105], (d) taken from [106].

2.5. On-machine measurement for machining processes

The large number of parameters that affect EJM and the complex interaction between the chemistries of the electrolyte and the work piece surface as reviewed in Section 2.2 necessitates experimental studies to characterise the response of a given work piece material. Measurement of the machined geometry is an indispensable part of this and is often performed ex-situ in laboratories designed for metrology. On-machine measurement, as defined in review by Gao et al. [107] refers to the measurement of the work piece surface within the same machine set up, where the work piece is manufactured. This approach has a number of benefits, including avoidance of moving and refixturing in a different machine for measurement, detection of problems earlier

on in the manufacturing process of a product close to the point of failure and reduction of human intervention for machine setting and tool path programming. In-process measurement is the ideal case for on-machine measurement where the measurement is taking place simultaneously with the manufacturing process, thus enabling the largest productivity improvements.

Available technologies suitable for on-machine surface measurement can be classified into full-field systems, where topographic data is acquired simultaneously from the whole field of view and scanning probe methods, which involve the translation of some type of displacement probe over the area of interest [107]. Full-field measurement systems are predominantly optical and include methods based on interferometry, structured light projection techniques and photogrammetry. This family also includes three-dimensional microscopy techniques for microscale surface texture measurement such as laser confocal microscopy, focus variation microscopy and coherence scanning interferometry. An advantage of full-field systems is that measurement is fast and independent of the kinematics of the machine tool, but the drawback is that instrument size is proportional to the required measurement area [107]. Scanning probe methods are generally slower than full-field systems, but have lower equipment cost and can achieve high resolution. Methods such as stylus profilometry [108] and atomic force microscopy [109] are known as contact methods and rely on the mechanical displacement of a tip tracing the measured surface. Non-contact optical approaches utilising laser triangulation [110] or chromatic confocal probes [111, 112] have been a common choice for on-machine applications in milling and electrical discharge machining.

2.5.1. On-machine measurement for ECM and EJM

One of the main challenges for on-machine metrology is the fact that the measurement sensor must often operate in a harsh and uncontrolled environment. This is particularly relevant to electrochemical machining, where the corrosive environment can compromise the health of an additional measurement system.

In traditional die-sinking ECM, where the tool electrode is fed into the work piece in order to replicate the shape of the tool, monitoring of the IEG width is important to prevent collision and ensure optimum machining conditions. Machine vision systems have recently been employed to monitor this in real time and control the feed [113]. Clifton et al. [114] used an ultrasonic transducer bonded to the work piece to measure time-of-flight of ultrasonic pulses reflected from the work piece – electrolyte and electrolyte – tool interfaces. The study found that the latter reflection was attenuated when the electrochemical dissolution process was running, so effectively the thickness of the workpiece was measured through the first reflection. Han et al. [115] used the measured gap voltage during machining in pulse ECM as a measure of the distance between the electrodes and successfully implemented a closed-loop tool feeding system. With regards to EJM, limited work has been reported on process monitoring and on-machine measurement. Yahyavi Zanjani et al. [116] operated the process in controlled voltage mode and demonstrated that the jet resistance and respectively the machining current is affected by pre-existing features on the work piece surface and can be correlated to the depth of these features, however fundamental understanding of the response is still lacking and acquisition of detailed topography data has not been achieved.

2.6. Electrochemical surface characterisation techniques

In electrochemical research, a number of scanning probe techniques have been developed that can measure surface topography and local electrochemical properties for substrates. The most established ones – scanning electrochemical microscopy (SECM) and scanning ion conductance microscopy (SICM) are briefly described here. SECM (Fig. 2.25a), introduced by Bard et al. [117], utilises a microelectrode tip to obtain topographic and chemical information from the substrate surface immersed in an electrolyte solution. Different operating modes of SECM have been developed suited for conductive and non-conductive substrates [118], but in most cases the measurement is based on the detection of diffusion-controlled currents at the tip due to redox reactions involving electroactive species present in the electrolyte solution. The distance between the tip and the surface affects diffusion rates and the measured current. An AC bias can be applied to tip to measure the solution resistance and correlate this to tip-to-substrate distance without relying on a redox reaction [119]. The scanning electrochemical microscope has also been used as a tool for micromachining [120].

SICM (Fig. 2.25b) was first proposed by Hansma et al. [121] for topography imaging of non-conductive substrates immersed in an electrolyte bath. A micropipette filled with electrolyte is scanned over the surface while the current flowing between an electrode in the pipette and a reference electrode in the bath is monitored. The proximity of the substrate surface blocks the flow of ionic current, hence allowing to infer surface topography. Adaptations to the technique have been reported that allow topographic and surface capacitance measurements of conductive samples by applying an AC bias to the electrode

inside the pipette with respect to the sample instead of an electrode in the bath and performing spatially-resolved impedance measurements [122]. Although these techniques are widely applied for electrochemical studies, they are not relevant to manufacturing applications. Electrochemical measurements using an electrochemical jet may prove applicable to industrial settings for characterisation of engineering components.

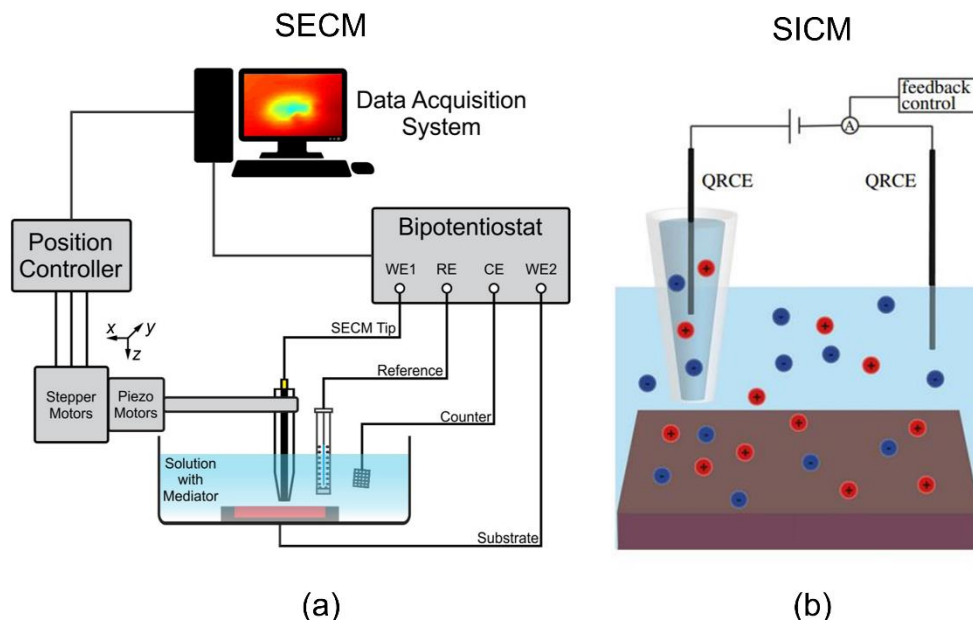


Fig. 2.25. Overview of the apparatus for scanning electrochemical characterisation techniques. (a) Scanning electrochemical microscopy, taken from [118]. (b) Scanning ion conductance microscopy, taken from [123].

2.7. Summary of literature review and key research gaps

In this chapter, the state-of-the-art in electrochemical jet machining was reviewed and critically assessed. This included the fundamental electrochemical processes governing surface formation, the developments in machine tool and process design, modelling of the process and the applications reported in literature. The research gaps identified in each of these areas are summarised in Fig. 2.26.

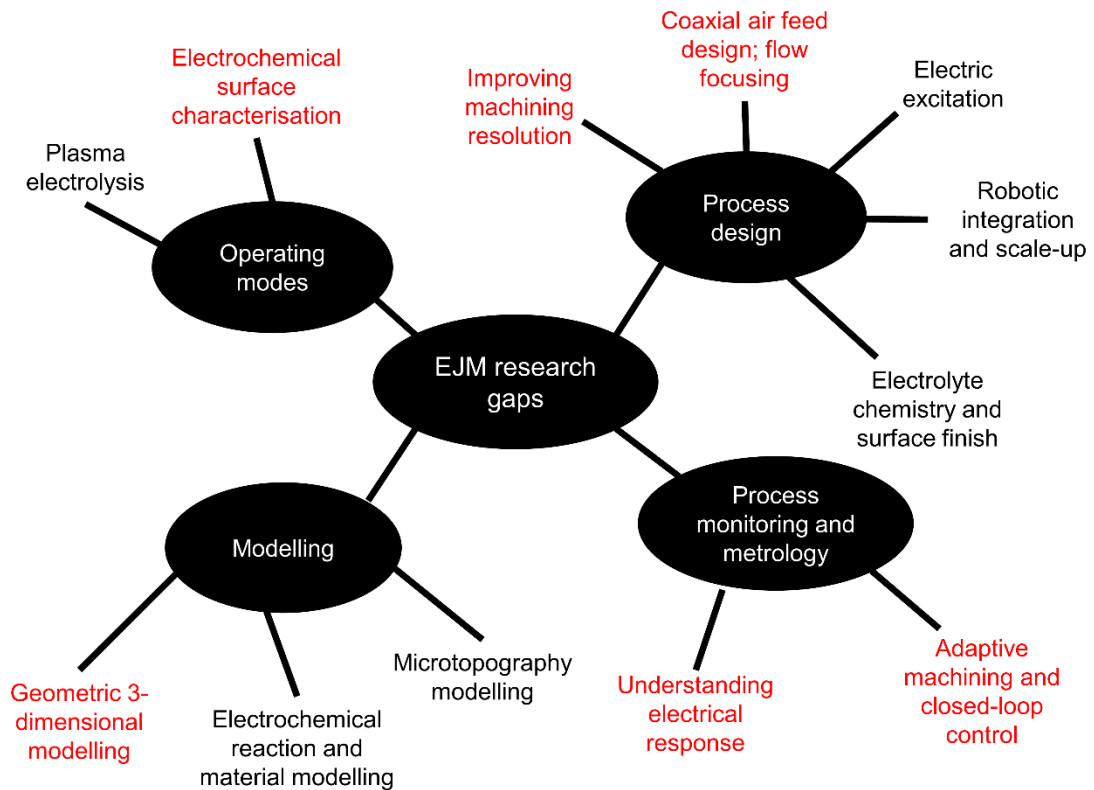


Fig. 2.26. Knowledge gaps relating to EJM identified through the literature review. Gaps addressed by this thesis in red.

As outlined in Section 2.1.4, the electrochemical jet apparatus has been used to adapt a number electrochemical surface treatment technologies for localised processing. Among these, electrochemical machining, anodizing and electrodeposition for localised processing have been widely researched. Methods based on plasma electrolysis - jet-based plasma electrolytic oxidation and plasma electrolytic polishing, are relatively less understood in terms of mechanisms and potential applications. Aside from surface modification, the possibility to perform electrochemical measurements within the apparatus and exploit the jet as a scanning probe for surface metrology and characterisation has not been explored. Such capability would represent significant augmentation to the electrochemical jet methods as it will allow on-machine and

in-process measurement without the need for additional contact-based or optical systems.

In the area of process design, significant efforts have been devoted to the improvement of machining resolution in EJM through the modification of the power source signal, the electrolyte chemistry, nozzle electrode design and the application of masks, but this remains fundamentally limited by the size of the nozzle electrode. The design of coaxial air flow has not been studied so extensively in comparison to other machine systems. In particular, a hydrodynamic method for jet constriction by a co-flowing gaseous stream known as 'flow focusing' is capable to produce jets smaller than 10 μm , but its application to EJM has not been investigated. Another important area of research is the scaling-up of the process for industrial applications through integration with robots and multiple end effectors, which would enable faster processing rates of larger areas compared to the widely used Cartesian set-ups. Although pulsed power supplies have been applied for the finishing of stainless steels and dissolution of passivating oxide films (see Section 2.2.4) fundamental understanding of excitation waveform design, its influence on transient phenomena at the interface and the corresponding effect on the work piece surface is still lacking. Similarly, the optimisation and understanding of the influence of electrolyte composition on resulting surface finish in EJM and development of electrolyte chemistries for passivating materials, such as titanium alloys, are promising areas for research.

Numerical modelling has been extensively applied to understand the physics of material removal and work piece shape formation in EJM. However, limited attention has been paid to the use of more simplified analytical models that can

be used to simulate surface geometry resulting from more complex machining operations in 3-dimensions. These would be beneficial for integration with tool path planning software to enable optimisation of parameters for surface texturing of real-world components and are investigated in this thesis. In terms of physics-based models, a gap remains in the more detailed consideration of the different electrochemical processes at the surface, in contrast to models based on Ohm's law. This can lead to better understanding of current efficiency in different material-electrolyte systems. No mathematical modelling has been reported for EJM on the microscale, considering the work piece composition and microstructure in order to simulate the resulting microtopography after processing.

Although on-machine and in-process measurement could bring clear benefits to EJM by increasing productivity and product quality, limited consideration has been paid to their implementation. Initial investigations have been conducted on the resistance of the IEG and its variation in response to nozzle and work piece geometry, but detailed understanding of the electrical response and the parameters that affect it is still lacking. Limited consideration has been paid to the utilisation of electrical signals monitored in-process to provide real-time information for the machining performance and enhance process control.

The key applications of EJM include the texturing of surfaces to enhance functional performance - machining of lubricant retaining features, modifying hydrophobicity and imparting antibacterial properties have been reported in literature. EJM has also been proven as a capable polishing and surface finishing process. A promising application area is the use of EJM as an

environmentally-friendly etching process to reveal material microstructure for further characterisation by optical or electron microscopy.

A critical factor limiting wider applications of EJM is the spatial resolution – advancements in this area can make the process more competitive compared to established processes such as laser texturing and micromachining and open new possibilities. For example, an improved resolution in combination with the high-quality surface finish could make the process suitable for cutting of medical stents. The integration of on-machine metrology with EJM would also be beneficial for such high precision applications and would also enable faster process parameter optimisation. In the area of materials characterisation, the combination of a surface preparation tool and measurement within the same set up would allow for fast defect detection. The following research gaps were consequently chosen as the focus of this thesis:

- Investigation of the hydrodynamic constriction of the electrolyte jet by ‘flow focusing’ with the objective of improving spatial resolution of EJM.
- Development of geometric models of material removal that can be used to simulate more complex machining operations in 3 dimensions and inform tool path planning.
- Understanding of the electrical response of system and the parameters that affect it. Utilisation of electrical signals monitored in-process to provide real-time information for the machining performance and enhance process control.
- Exploring the possibility to exploit the jet as a scanning probe for surface metrology and characterisation based on electrochemical

measurements to allow on-machine and in-process measurement without additional sensors.

3. Methodology

This chapter details the experimental techniques, equipment and parameters used in the reported investigations. The data analysis employed is also described.

3.1. EJP apparatus

The experimental work reported in this thesis was performed using the EJP research platform at the University of Nottingham. A schematic of the apparatus is shown in Fig. 3.1. The nozzle is mounted in servo-driven 3-axis positioning system with a positional repeatability of each axis of $\pm 20 \mu\text{m}$. Compressed air was supplied around the electrolyte jet through an annular passage to clear excess electrolyte from the specimen surface. For machining operations, a Keysight N5751A DC power supply rated to 300 V and 2.5A is connected across the inter-electrode gap. This also provides internal monitoring functions of voltage and current which are used to investigate IEG response during machining. For fundamental investigations of the electrical behaviour of the IEG the electric excitation is provided by an Ivium Vertex One potentiostat/galvanostat (maximum voltage $\pm 10 \text{ V}$, maximum current 100 mA) with frequency response analyser capability instead of the power supply. This was necessary for conducting experiments with low applied currents, so the effect on the surface is minimised. The instrument also allows to investigate the

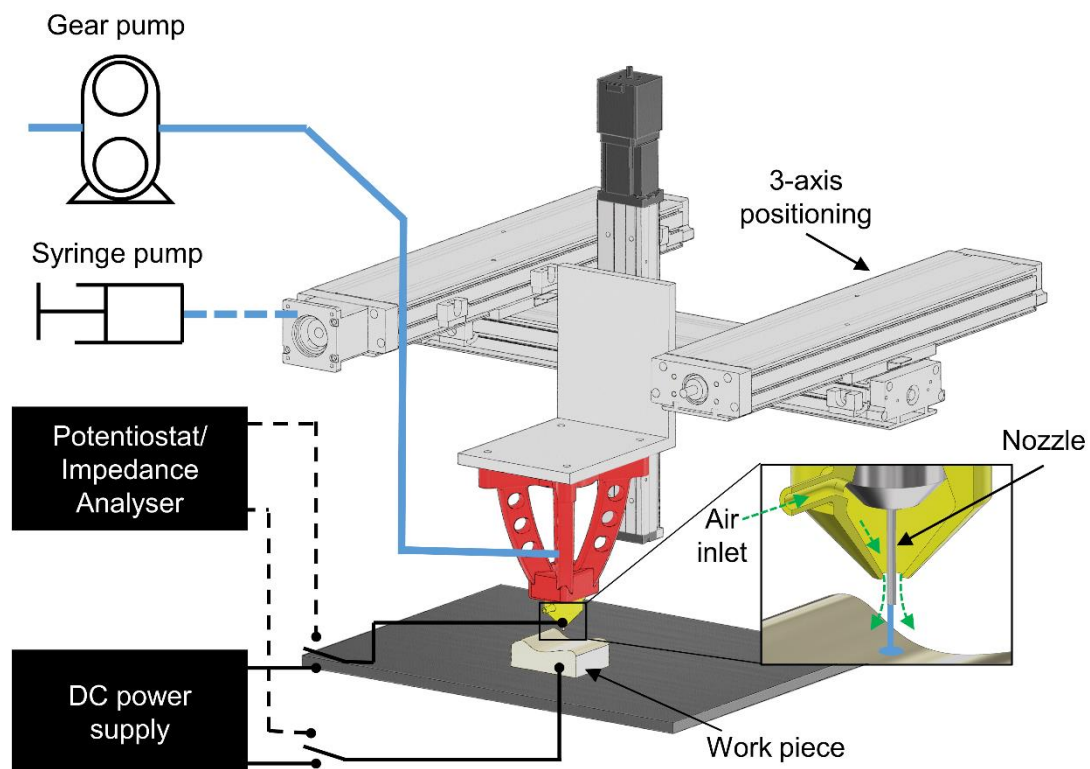


Fig. 3.1. Schematic of the EJP apparatus and its different configurations used for the experimental work in this thesis.

IEG under alternating current (AC) conditions. The electrical measurements performed are explained in detail in Section 3.3. Electrolyte was supplied to the nozzle by a gear pump with a maximum specified flow rate of 470 ml/min. The set flow rate was measured by weighing the amount a fluid passed through the nozzle for a timed interval with the average jet speed calculated from the nozzle electrode cross-sectional area. The nozzle motion, pump and power supply are controlled as part of the machine programme, allowing the execution of surface texturing operations. The set up allows the setting of initial stand-off distance between the work piece and the nozzle through automated detection of electrical contact between the nozzle electrode and the work piece. In the reported experiments, this was performed by touching off the work piece at a

single point at the start of the tool path and moving the nozzle vertically at the desired distance.

3.1.1. Modifications for flow focusing

To investigate the application of the flow focusing principle [2] to EJM in Chapter 6, modifications to the standard apparatus were required. A novel end effector was developed, with the design rationale presented in Chapter 6. Precise flow rates < 10 ml/min were necessary for its operation, so a KDS 200 syringe pump with a programmable flow rate with specified accuracy of $\pm 1\%$ was used to supply the electrolyte instead of the gear pump (Fig. 3.1). In addition, an air flow of controlled pressure had to be supplied and this was realised by means of a Festo VEAB pneumatic regulator with a range of 5 – 1000 mbar and accuracy of ± 7.5 mbar. Since the developed end effector was made from a non-conductive material, the short circuit detection routine for stand-off setting could not be applied, so the initial position above the workpiece was set with estimated accuracy ± 0.05 mm by using a feeler gauge.

3.2. Materials and electrolytes

Stainless steel adhesive dispensing nozzles with internal diameter accuracy of ± 0.01 mm were used for tool electrodes in this work. For initial investigations of the electrical response of the electrolyte jet commercially pure nickel 201 (composition in Table A.1 of the Appendix) was used as the work piece material and this was hand polished to P1200 grade. Different electrolytes were tested which were aqueous solutions of sodium chloride (NaCl) at different concentrations. For the experiments in this thesis the electrolyte conductivity was a critical parameter, so the solution concentration was adjusted during the experimental work through the addition of salt or deionised water to

compensate for variation of temperature and achieve of a conductivity within $\pm 2\%$ of values stated throughout the thesis. The conductivity was measured using a Mettler Toledo S3 conductivity meter ($\pm 0.5\%$ specified accuracy). The electrolytes used are hence specified by their conductivity for each experiment described in this chapter and the results reported in the following chapters.

To assess the effects of different materials and electrolyte chemistry on the electrical response, experiments were also performed with a Ti-6Al-4V titanium alloy (composition in Table A.2) and an aqueous sodium nitrate (NaNO_3) solution.

All machining experiments in this thesis, relating to the development of in-process monitoring and novel tooling were performed using NaNO_3 electrolyte and stainless steel grade 304 (composition in Table A.3) as the work piece, since this a widely researched system.

The possible cathodic reactions occurring at the tool nozzle in the investigated electrolyte solutions are hydrogen evolution and nitrate ion reduction (for NaNO_3) [124], thus no electrodeposition on the tool occurs during machining.

3.3. Electrochemical measurements

The behaviour of the IEG was investigated through AC and DC electrical measurements performed in galvanostatic mode, meaning that the current flowing through the jet was controlled while the potential difference between the work piece electrode and nozzle electrode was measured. Since the impinging electrolyte jet is a 2-electrode cell, the work piece is the working electrode, while the nozzle is both a reference and counter electrode. The measurements of total cell potential hence include contributions from both the nozzle and the

work piece, and the Ohmic drop across the gap as outlined in Section 2.1.3. A simplified galvanostat circuit diagram and the connection to the electrolyte jet

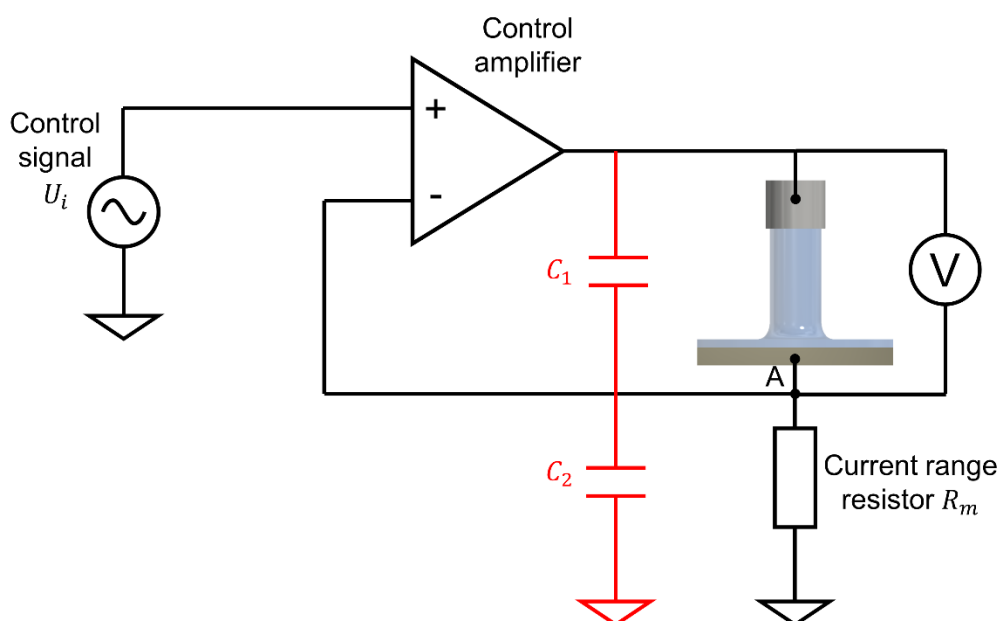


Fig. 3.2. Simplified schematic of a galvanostat connected to the electrolyte jet with stray capacitances.

cell is shown on Fig. 3.2. A control signal (AC or DC) U_i is applied to the non-inverting input of the control amplifier, which adjusts its output, so that both inputs are at the same potential. This means that point A in Fig. 3.2 is also at potential U_i and the current flowing through the electrolyte jet is independent of the impedance of the cell and is given by $\frac{U_i}{R_m}$ where R_m is the current range selection resistor. The instrument can switch between a set of resistors R_m , thus allowing different ranges of applied current.

3.3.1. Considerations for AC measurements

The non-idealities of the galvanostat become important when high frequency measurements are performed. Stray capacitances C_1 and C_2 are present at the instrument inputs, between the reference electrode and the working electrode and between the working electrode and ground respectively (Fig. 3.2). At high

frequencies and high jet impedances, some current will leak through the stray capacitances reducing the accuracy of the measurement. This is also affected by the value of the current range resistor with a higher resistance (lower current range) degrading performance at high frequency. This can be appraised through the impedance spectrum acquired from a fixed resistor with nominal resistance value $22\text{ k}\Omega$ ($\pm 5\%$ tolerance) for frequencies $1\text{ kHz} - 250\text{ kHz}$ (Fig. 3.3a), which exhibits high frequency artefacts leading to error in measured impedance magnitude and a phase shift from 0° . The frequency for which the error in magnitude $< 1\%$ and phase shift $< 1^\circ$ could be considered as the maximum acceptable frequency for measurement of impedance of a certain magnitude at the applied current range. Performing this measurement for several fixed resistance values and finding the corresponding frequency where the measurement error reaches 1% (Fig. 3.3b) allows to characterise the instrument performance as a function of frequency. A compromise must be made between capabilities of the instrumentation and the optimal conditions

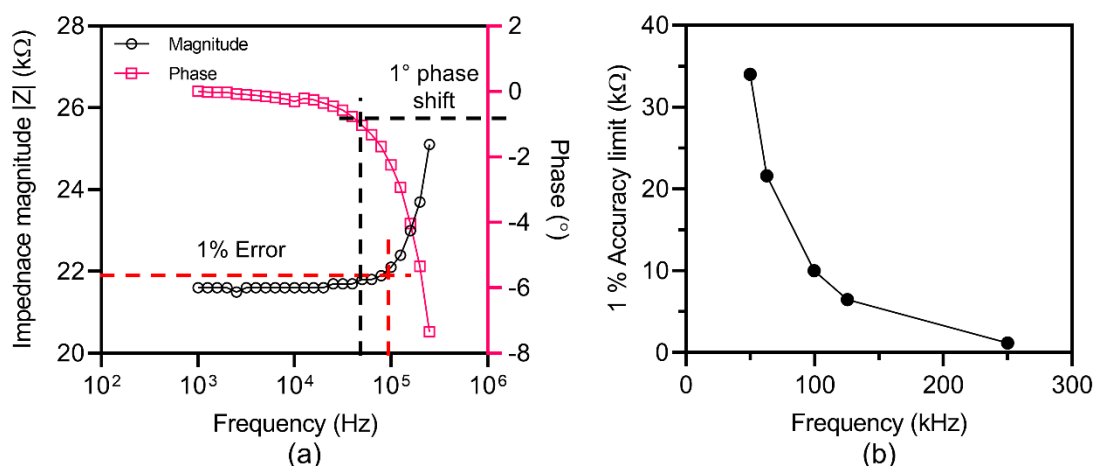


Fig. 3.3. Measurement range characterization for the instrument used in this thesis for the 1mA current range. (a) Impedance spectrum acquired from a fixed resistor showing instrumental artefacts at high excitation frequencies. The accuracy limit plot against frequency is constructed by finding the maximum frequency resulting <1% magnitude deviation and <1° phase shift. (b) Accuracy limit plot for the instrument acquired with fixed resistors.

required for the electrochemical measurement. This is achieved by maintaining maximum excitation frequency such that electrochemical cell impedance is within the 1% accuracy limit as per Fig. 3.3b. This ensures meaningful measurements.

3.3.2. Linear current sweeps and electrochemical impedance spectroscopy

Initial appraisal of the electrical behaviour of the electrolyte jet cell was performed with a 0.23 mm nozzle and 60.6 mS/cm NaCl electrolyte solution at a jet speed of 10.9 m/s. A plate of nickel 201 was employed as the work piece. A linear current sweep from 0 – 400 μ A at a scan rate of 5 μ A/s was applied with the jet over a fixed location on the work piece surface to investigate the voltage of the cell under DC excitation. The average current density in the jet was estimated by dividing the supplied current value over the nozzle cross-sectional area. Although this is not representative of the current density distribution on the anode surface, which is highly non-uniform (see Fig. 2.1c), it

is an established quantity to compare the condition of the IEG across nozzle sizes [36].

Electrochemical impedance spectroscopy under sinusoidal excitation in the 1 Hz – 250 kHz frequency range with a programmed current amplitude of 40 μ A was performed to establish the optimum excitation frequency for resistance measurement. The current amplitude was chosen to avoid excessive potential swings at low frequencies and maintain system linearity over the chosen range [125]. Both experiments were performed with the nozzle positioned at 0.5 mm. Additional impedance spectra in the range of 1 kHz – 250 kHz were acquired for the different combinations of the nickel 201 and Ti-6Al-4V work piece materials with the NaCl and NaNO₃ electrolyte solutions to appraise the influence of material and electrolyte chemistry on the high frequency electrical response.

The impact the measurement has on the integrity of the work piece surface under DC and AC excitation frequencies of 1kHz, 10 kHz and 100 kHz was also assessed by setting the jet to dwell at fixed location. Under DC, the applied current was 400 μ A and the excitation was applied for 30 s resulting in 12 mC of charge transfer. The same amplitude was applied under AC, but the dwell time was scaled to 94.25 s to maintain equivalent charge transfer during the anodic half-cycle of the waveform (charge transfer during the half-cycle $q = It/\pi$, where I is the current amplitude and t is dwell time).

3.3.3. Acquisition of resistance response curves

The resistance response of the jet to changing stand-off distance was evaluated by traversing the nozzle vertically in the Z-direction and recording the measured

resistance as a function of the Z-axis position read from the drive motor encoder, obtaining a resistance response curve for a selected parameter set. The traverse speed in Z was set at 0.1 mm/s and both resistance and position were sampled every 0.1 s resulting in 10 μm sampling distance. Response curves were acquired in this manner at varying electrical excitation (DC or AC), electrolyte conductivity (13.2 mS/cm – 100.3 mS/cm), nozzle size (0.1mm – 0.31 mm) and average flow speed (7.8 – 17.4 m/s) to establish the influence of system parameters. When different nozzle sizes were compared, the volumetric flow rate of the electrolyte was adjusted to maintain an average jet speed of 10.4 m/s (± 0.6 m/s). Under DC, the resistance is calculated as the ratio between the measured voltage and applied current, while under AC the resistance is taken as the real component of the complex impedance. Further measurements to acquire the resistance response to translation of the Z axis were conducted using a NaNO_3 solution of 60.6 mS/cm electrolytic conductivity and a Ti-alloy (Ti-6Al-4V). This was done to appraise the influence of work piece material and electrolyte chemistry on the resistance response.

Resistance response curves were also acquired for the flow focusing end effector presented in Chapter 6 for supplied air pressures 100 mbar – 300 mbar and electrolyte flow rates in the range 0.25 ml/min – 2 ml/min. The electrolyte used was NaNO_3 with a conductivity of 120.1 mS/cm (S.D. = 0.7 mS/cm). Due to the expected higher IEG resistances relative to the standard configuration, sinusoidal current waveform of amplitude 100 μA and frequency 10 kHz was applied while the nozzle was translated in the Z-direction between 0.2 mm and 2 mm stand-off distance and the resistance was sampled every 10 μm .

3.3.4. Scanning resistance measurements

The resistance response to surface features was assessed by performing measurements while the jet was traversed across an electroformed nickel step height reference specimen (Rubert 513) consisting of three grooves with depths 495 μm (S.D. = 1 μm), 196 μm (S.D. = 0.4 μm) and 30 μm (S.D. = 0.07 μm) and corresponding widths of 2 mm, 2 mm and 0.5 mm measured between the top edges (Fig. 3.4). The quoted dimensions were obtained via stylus profilometry and were used as a reference to evaluate the electrolyte jet measurements. The jet resistance measurement was carried out at a fixed excitation frequency of 60 kHz at the 1 mA current range of the galvanostat while varying internal nozzle diameter (0.1 mm – 0.31 mm), electrolyte conductivity (21.3 mS/cm – 100.3 mS/cm), average jet speed (7.8 m/s – 17.4 m/s), feed rate (0.25 mm/s – 2 mm/s) and initial stand-off distance to the top surface (0.25 mm – 1mm). The resistance was sampled with a sampling distance of 10 μm . A full experimental matrix is given in Table B.1 of Appendix B.

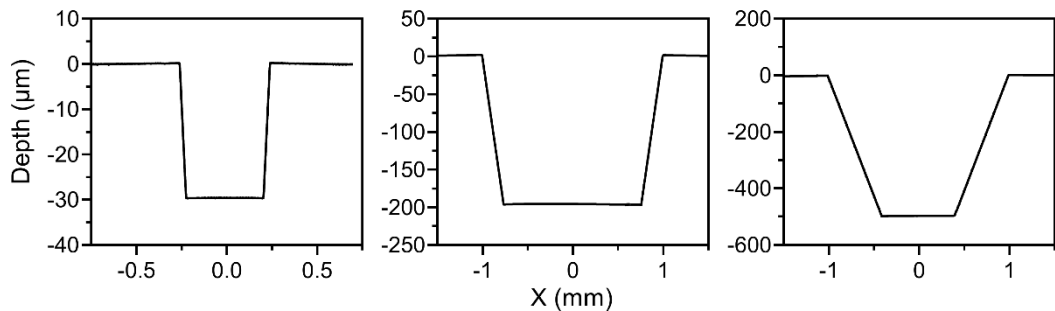


Fig. 3.4 Cross-sectional profiles of the grooves in the Rubert 513 step height reference specimen measured with stylus profilometry.

Scanning measurement was also performed with the flow focusing end effector presented in Chapter 6 with a 120.1 mS/cm NaNO_3 electrolyte delivered at 1 ml/min and a supplied pressure of 150 mbar. The feed rate was set to 0.5 mm/s

and the sampling distance to 10 μm . The electric excitation was of magnitude 100 μA and frequency of 10 kHz.

Areal resistance imaging was investigated on the surface of a British pound coin (year 2017), by traversing a 0.1 mm internal diameter nozzle along a zig-zag path at a scanning speed of 0.5 mm/s, with NaCl electrolyte with conductivity of 60.6 mS/cm an average jet speed of 9.8 m/s. The sampling interval of the galvanostat was set to 0.04 s resulting in 20 μm sampling distance along the X-axis and the step over distance between parallel passes was 100 μm . To synchronise the scanning motion with signal acquisition, the galvanostat was triggered by the motion control system upon motion start via an analogue signal. This allowed the assignment of resistance data to the corresponding spatial coordinate.

3.3.5. In-process resistance measurement

Measurement of resistance during machining as described in Section 3.4.1 was performed by using internal monitoring functions of the EJM power supply which provided analogue feedback signals to the machine controller. The sampling period of the signals was 10 ms which is the shortest time allowed by the machine controller. The acquired signals were filtered to remove high-frequency noise with a Gaussian filter with a cut-off of 0.5s (corresponding to a frequency of 2 Hz).

3.4. EJM parameters

All machining experiments discussed in this thesis were conducted using grade 304 stainless steel as the work piece and an aqueous NaNO_3 solution as the electrolyte.

3.4.1. Machining for in-process monitoring experiments

The electrical resistance response during machining was investigated (Chapter 5, Section 5.3) for different machining operations, namely static pit machining, straight channel machining and curved channel machining. All experiments were performed with a fixed supplied current of 150 mA, nozzle diameter of 0.25 mm, average jet speed of 14 m/s and stand-off distance of 0.5 mm. The electrolyte conductivity was maintained at 115.8 mS/cm (S.D. = 1.1 mS/cm).

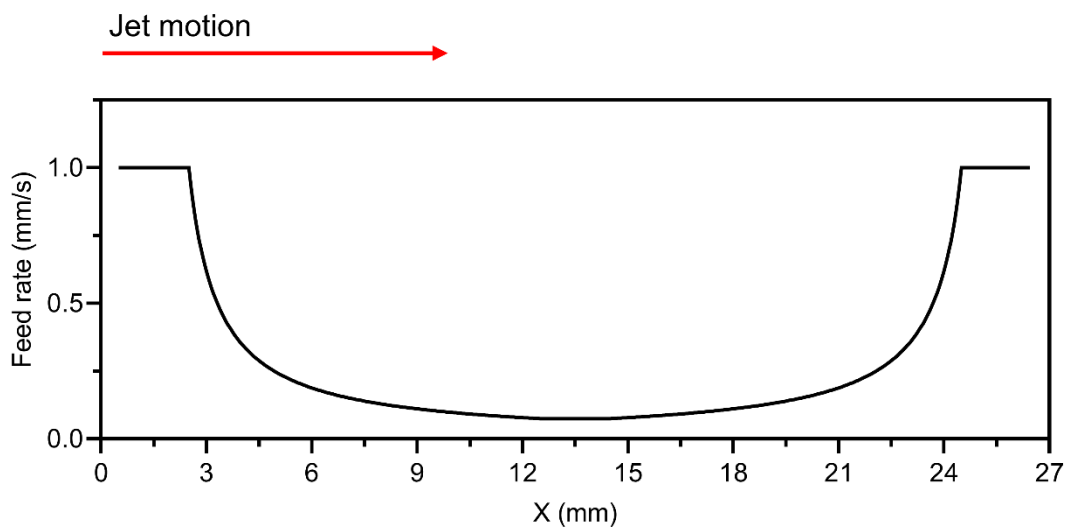


Fig. 3.5. Programmed feed rate as a function of distance of travel for machining of straight channels.

In the static pit machining experiment the dwell time was varied from 1 s to 11 s to appraise the resistance response as the depth of the pit increases. For the straight channels, a 26 mm long tool path was programmed with the feed rate v_f programmed to vary between 1 mm/s and 0.075 mm/s along the axis of motion to result in changing depth of the channel (Fig. 3.5). The feed rate variation function was chosen to result in a linear variation of exposure time to the jet along the tool path. The constant feed rate sections at 1 mm/s at the two ends of the tool path were planned to provide a reference level for the acquired signal against which changes can be measured.

The electrical resistance along curved tool paths was investigated by planning jet trajectories containing 90° fillet corners of radii 0.125 mm, 0.25 mm and 0.5 mm. The feed rate along the tool path was set to 0.2 mm/s.

3.4.2. Machining with automated tracking of the component surface

In Chapter 5, the closed-loop control of stand-off distance during machining based on resistance monitoring is investigated. This was performed with a nozzle diameter of 0.25 mm, supplied current of 120 mA, an average jet speed of 14 m/s, horizontal feed rate of 0.5 mm/s and 136.6 mS/cm NaNO₃ electrolyte solution.

3.4.3. Machining with flow focusing end effector

The machining performance of a novel end effector for EJM based on the principle of flow focusing is investigated in Section 6.5 of Chapter 6 with the objective of improving the machining resolution. Machining parameter selection for these experiments was based on results from the characterisation of the design reported in Chapter 6, so they are described in detail within that chapter.

3.5. Conventional surface metrology

Established surface metrology techniques were used for comparison against the resistance-based measurements investigated in this thesis and for geometric measurement of EJM generated features.

3.5.1. Stylus profilometry

Stylus profilometry was employed in this thesis to acquire surface profiles from the Rubert 513 groove reference specimen for comparison with the resistance-based electrolyte jet measurements. This is a contact surface topography measurement method, which is based on laterally scanning a diamond stylus

tip across the surface. The stylus is attached to an arm whose vertical motion is detected for example by means of a linear variable differential transformer. The instrument used for this work was Taylor Hobson Form Talysurf Intra stylus profiler with 2 μm stylus tip radius, 0.5 μm data sampling interval in the scanning direction and specified vertical resolution of 4 nm (specifications summarised in Table 3.1). Measurements were performed by Dr Adam Thompson of the Manufacturing Metrology Team at the University of Nottingham.

Table 3.1. Specifications of Taylor Hobson Form Talysurf Intra stylus profilometer.

Stylus tip radius	2 μm
Vertical range	1 mm
Vertical resolution	4 nm
Straightness error	0.2 μm over 20 mm
Data point spacing	0.5 μm

3.5.2. Focus variation microscopy

Areal measurements of surface topography were conducted with an Alicona G4 focus variation microscope. Focus variation microscopy (FVM) utilises an optical system with a narrow depth of field to determine the surface height by finding the position at which the image is in focus (see Fig. 3.6). The focus search is performed by moving the sample in the axial direction relative to an objective lens, with some focus measure $F_z(x, y)$ defined over a region around each pixel. The surface height corresponds the z-position where the focus measure is maximised. One possible definition of the focus measure is as the standard deviation of the grey values of pixels within the region, with higher standard deviations corresponding to an increased sharpness, however more

advanced algorithms are also in use [126]. The calculation is performed for every position in the field of view of the images sensor resulting in an areal topography map of the sample. Large areas are imaged by translating the objective parallel to the sample and stitching multiple fields of view. The quality and resolution of the acquired dataset is dependent on the magnification of the objective lens with higher magnifications allowing to resolve finer features. This thesis is mainly concerned with large-scale surface topography so objectives with x5 and x20 magnification and corresponding numerical apertures (NA) of 0.15 and 0.4 were used. The overall system specifications for each objective are listed in Table 3.2. The applied objectives are stated throughout the thesis.

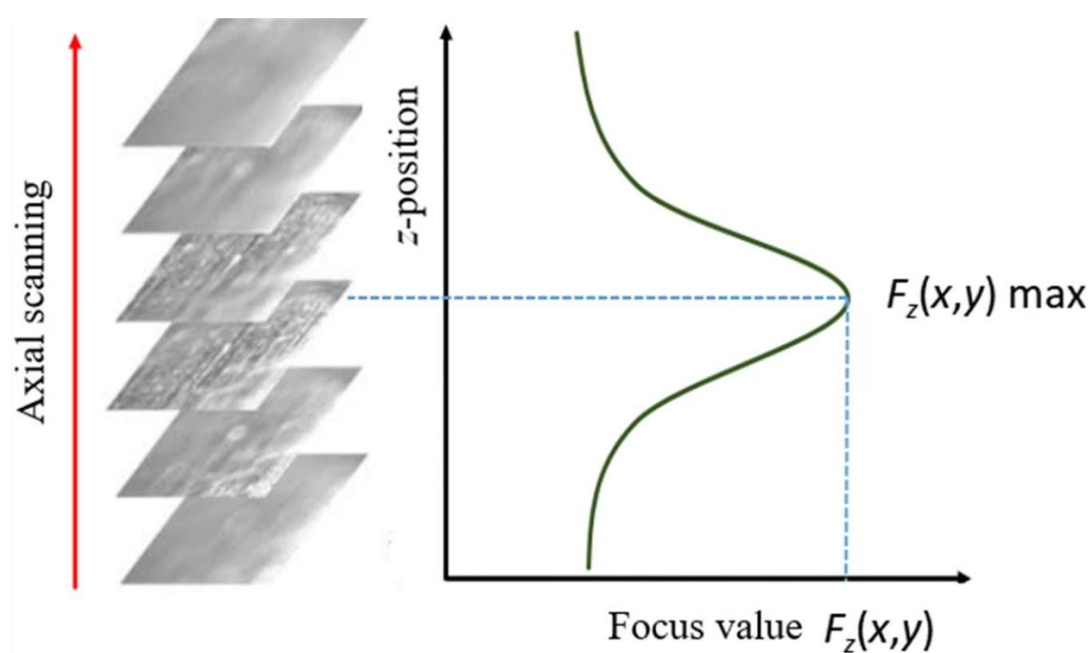


Fig. 3.6. Principle of operation of the FVM method. Surface height is inferred from the z-position where the image is the sharpest. Diagram taken from Santoso et al. [127]

Table 3.2. Specifications of the Alicona G4 focus variation microscope.

Objective magnification	x5	x20
NA	0.15	0.4

Step height accuracy (1 mm)	0.05%	0.05%
Minimum vertical resolution	410 nm	80 nm

3.6. Optical imaging of the jet

To characterise the jets generated through the flow focusing end effector, an optical microscopy configuration was set up in the EJP apparatus. The jet and the surrounding field were illuminated uniformly using a collimated incoherent light source (Thorlabs M625F2 LED, wavelength: 625 nm). The transmitted light was used to image the jet on CMOS camera (Thorlabs DCC1545M, pixel size: 5 μ m). The microscope consisted of a 10x objective lens (Mitutoyo, numerical aperture NA = 0.28, focal length: 20mm) and a tube lens (focal distance of 160 mm) configured as shown in Fig. 3.7 to provide 8x magnification where each pixel of the acquired images is equivalent to 0.63 μ m. The jet was positioned in focus using the motion axes of the EJP apparatus. The optical system was designed and built by Dr Sidahmed Abayzeed of the Optics and Photonics Research Group at the University of Nottingham.

Jet diameter was measured near the exit of the nozzle directly from the acquired images using ImageJ image analysis software. Uncertainty was assigned by defining maximum and minimum limits within which the measured diameter lies for each image. The reported measured value is the average.

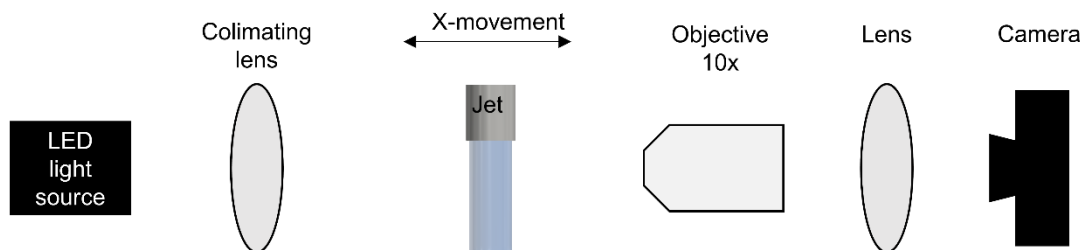


Fig. 3.7. Optical set used for jet imaging designed by Dr Sidahmed Abayzeed.

3.7. Data processing and analysis

3.7.1. Determination of sensitivity

To convert the measured electrical resistance of the electrolyte jet to surface height, the sensitivity S of the measurement system was defined as:

$$S = \frac{\partial R_{jet}}{\partial L} \quad (3.1)$$

From Eq. (2.10), the theoretical sensitivity for an ideal cylindrical conductor is $S = \frac{1}{\sigma A_{jet}}$. Experimentally, the sensitivity is obtained by least squares linear regression fit in the range of interest to the resistance response curve at the relevant parameter set obtained as described in Section 3.3.3. This is used as a parameter-dependent calibration factor for resistance-based surface topography measurements.

3.7.2. Scanning resistance measurement analysis

The signals acquired from scanning the jet across the groove reference specimen as described in Section 3.3.4 were levelled by subtracting the line of best fit to the top surface and filtered with a Gaussian filter (cut-off length 0.08 mm) to reduce noise. The depth of the grooves and the corresponding resistance change was characterised according to ISO 5436-1 by fitting two parallel lines to the bottom and top surfaces and measuring the distance between them (see Fig. 3.8). The measured resistance was converted to surface height through division by the experimentally calibrated sensitivity from the resistance response curve for the corresponding parameter set.

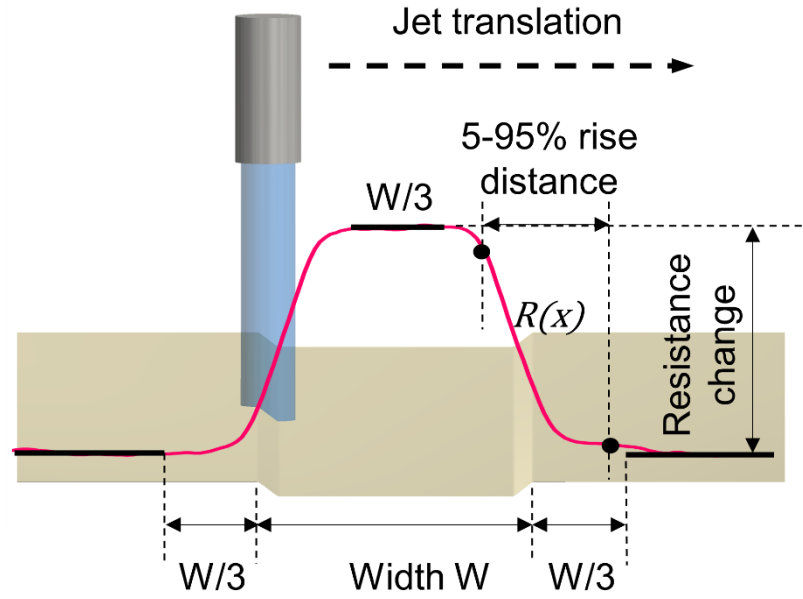


Fig. 3.8. Analysis of resistance response acquired as the jet is traversed across a reference groove.

The uncertainty in the measured profiles was calculated by considering repeatability of the measurement v_{rep} taken as standard deviation from five repeated measurements, the uncertainty in calibration due to electrolyte conductivity variation v_{cond} , obtained considering the measured variation of conductivity during experiments, and the uncertainty due to the calibrated sensitivity v_s , obtained considering the standard deviation of the sensitivity obtained from 3 replicates of the response curve. The combined uncertainty is

$$\sqrt{v_{rep}^2 + v_{cond}^2 + v_s^2}.$$

To perform quantitative comparison of spatial resolution of the resistance measurement at different conditions, the response as the jet traversed across the side walls of the grooves was analysed by evaluating the 5%-95% rise distance, defined as the horizontal distance in X over which the resistance signal changes from 5% to 95% of the peak corresponding to the full depth of the groove (Fig. 3.8). An equivalent approach is applied to the data from the

stylus profilometer. The reported rise distances were averaged from both sides of each groove.

The time-domain signal acquired from the areal scan of the British pound was filtered using a 1-dimensional median filter with window size of 5 data points to remove speckles before further processing. The acquired data was compared against FVM. The data sets were processed in MountainsMap surface analysis software and were aligned to minimise the root mean square error between them. An error map as a function of spatial coordinates was generated by subtracting the electrolyte jet data set from the FVM data.

3.7.3. Machined surface geometry analysis

All analysis of machined surface geometry measured through FVM in this thesis was performed using the MountainsMap surface analysis software by Digital Surf. This allows visualisation of topography maps, profile extraction, filtering and measurements of depth, kerf width, and cross-sectional area as shown in Fig. 3.9. Gaussian filtering was applied to the acquired surface profiles in this thesis to remove short-wavelength surface roughness since the overall geometry is of interest. The filter specifications in terms of cut-off length are given when the data is presented in the results chapters.

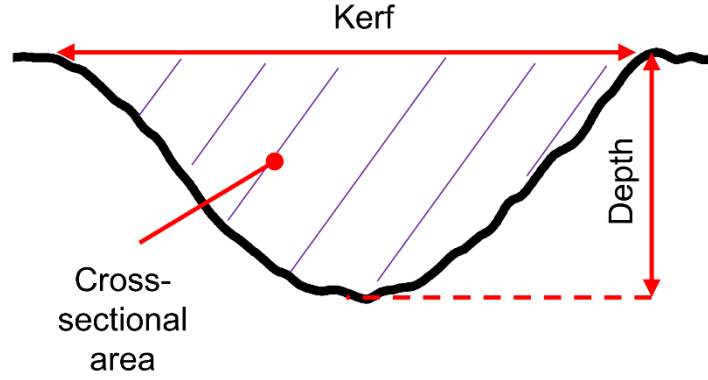


Fig. 3.9. Metrics quantified from the cross-sectional profiles of machined channels in this thesis.

3.7.4. Calculated quantities

In Chapter 6 of this thesis, several quantities are calculated from the observed experimental results to characterise machining performance. The specific material volume removed V_q per unit charge passed expresses the dissolution efficiency of the machining process and was calculated from the cross-sectional area of machined grooves and the current measured from the power supply during machining according to:

$$V_q = \frac{A_{cross} v_f}{I} \quad (3.2)$$

where A_{cross} is the cross-sectional area of the channel, v_f is the applied feed rate and I is the measured current. The standard uncertainty in the specific removal v_{V_q} is calculated according to the rules for propagation of uncertainty

and was given by $v_{V_q} = V_q \sqrt{\left(\frac{v_I}{I}\right)^2 + \left(\frac{v_{A_{cross}}}{A_{cross}}\right)^2}$ where $v_{A_{cross}}$ is the uncertainty in area taken as the standard deviation of the performed measurements and v_I is the standard uncertainty of the current measurement estimated to be 2.0 mA from the power supply specifications.

The experimentally observed average current density in the was calculated from the optically measured jet diameter d_{jet} (as per Section 3.6) and the measured current I

$$J = \frac{4I}{\pi d_{jet}^2} \quad (3.3)$$

with uncertainty $v_J = \sqrt{\left(\frac{\partial J}{\partial I}\right)^2 v_I^2 + \left(\frac{\partial J}{\partial d_{jet}}\right)^2 v_{d_{jet}}^2} = \frac{4}{\pi d_{jet}^2} \sqrt{v_I^2 + \frac{4I^2}{d_{jet}^2} v_{d_{jet}}^2}$

The theoretical temperature increase ΔT of the electrolyte due to Joule heating as it flows through the gap was estimated by neglecting all heat losses according to

$$\Delta T = \frac{IU}{\rho_l Q c_p} \quad (3.4)$$

where U is the voltage across the IEG, ρ_l is the density of the electrolyte, Q is the flow rate and $c_p \approx 4182 \text{ J/(kg.K)}$ is the specific heat capacity of the electrolyte assumed to be the same as for water. The standard uncertainty is estimated as $v_{\Delta T} = \Delta T \sqrt{\left(\frac{v_I}{I}\right)^2 + \left(\frac{v_U}{U}\right)^2 + \left(\frac{v_Q}{Q}\right)^2}$ where $v_U = 0.04 \text{ V}$ from the power supply specifications and $\frac{v_Q}{Q} = \frac{0.01}{\sqrt{3}} \approx 0.006$ from the syringe pump specifications.

4. On-machine resistance measurement

4.1. Introduction

The literature review identified that on-machine metrology could add significant value to electrochemical jet manufacturing processes by enabling rapid inspection and providing opportunities for automation of set up. Although this can be performed via optical and laser-based methods, it would involve placing an additional sensor or illumination source in the proximity of the interaction zone where the environment is corrosive and potentially damaging for the measurement device. It is understood that the electrical resistance of the electrochemical jet cell is dependent on the distance between the nozzle and work piece surface [20] and the jet has been suggested to approximate a cylindrical conductor (see Eq. (2.10) in Section 2.1.3). It is proposed here that this behaviour can provide a route to a reliable low-cost on-machine metrology for EJM and EJP. Following an appropriate calibration of the resistance response to changes in stand-off distance, measurement of work piece surface topography could be achieved by scanning the nozzle at a constant height across the surface and mapping resistance as a function of spatial coordinates. To realise this approach, the capacitive properties of the electric double layer forming at the electrode-electrolyte interface are exploited by applying a high-frequency excitation current between the nozzle and the work piece to suppress the electrochemical reactions at the electrodes and minimise the contribution from surface overpotentials to the acquired signal. This principle has been applied previously for realising micrometric precision electrochemical micromachining with ultrashort pulses [128] by preventing anodic dissolution at large distances from the cathode-tool. Here, it is key for ensuring that only the

ohmic contribution due to variation of surface geometry is measured and, in contrast to EJM, the anodic dissolution of the work piece is minimised. This approach can be applied towards geometry verification pre- and post-machining, and it can also serve as a low-cost inspection tool that can be integrated with other manufacturing processes. In comparison to other electrochemical methods, such as scanning electrochemical microscopy and scanning ion conductance microscopy reviewed in Section 2.6, surface metrology with an electrolyte jet would not require the immersion of the work piece in a bath and would be suited to measurements of larger areas in manufacturing settings.

This chapter investigates the fundamentals of the electrical response of the electrolyte jet to work piece surface geometry and identifies the key parameters for the design of the proposed measurement system. The measurement method performance in terms of sensitivity and spatial resolution is evaluated and the capability for on-machine areal surface geometry measurement is demonstrated.

4.2. IEG electrical excitation

As discussed in the literature review (Section 2.1.3), the inter-electrode gap in ECM and in electrochemical systems in general exhibit complex non-linear electrical behaviour. This necessitates careful consideration of the type of electric signal to be applied for the successful implementation of the proposed spatial resistance mapping approach. One possible approach is to apply a direct current excitation as is conventionally done in EJM, but at reduced current densities ($<1 \text{ A/cm}^2$, minimum used for machining usually $> 10 \text{ A/cm}^2$) to reduce local anodic dissolution and corrosion of the work piece surface.

When direct current flows through the IEG (Fig. 4.1a-b), the potential difference between the nozzle and work piece is comprised of the equilibrium potential of the electrochemical cell (potential at zero current), the electrode overpotentials, required to drive the electrochemical reactions at the electrode-electrolyte interface, and the ohmic drop due to the resistance of the jet. The presence of overpotentials causes a non-linear response of total voltage upon application of a current sweep at a fixed stand-off (Fig. 4.1a). For measurement of stand-off distance, only the ohmic drop is of interest, but the electrode overpotentials manifest themselves as an additional interfacial impedance Z_F associated with the Faradaic process at the electrode surface. Resistance response curves acquired according to Section 3.3.3 of Methodology under DC conditions (see Fig. 4.1b) exhibit an offset from the origin demonstrating that the interfacial impedance appears in series with the resistance of the jet R_{jet} . The offset is likely to display sensitivity to heterogeneities in work piece surface reactivity and could occupy a significant portion of the available measurement range of the signal acquisition device, hence for accurate appraisal of work piece geometry, this must be delineated. To appraise the contributions of the different components of the overall signal, the linear ohmic drop is estimated at 0.5 mm stand-off by considering the slope of the resistance-stand-off response curve at an average current density of 0.96 A/cm^2 (Fig. 4.1b) and is subtracted from the total recorded voltage to obtain the overpotentials (Fig. 4.1a). The overpotentials dominate the signal in the low current density range making the ohmic contribution difficult to detect at current densities $J < 0.1 \text{ A/cm}^2$ but are expected to obey a logarithmic Tafel relationship $E - E_{eq} = a + b \log J$ (see Section 2.1.2) with increasing current density resulting in a slower increase

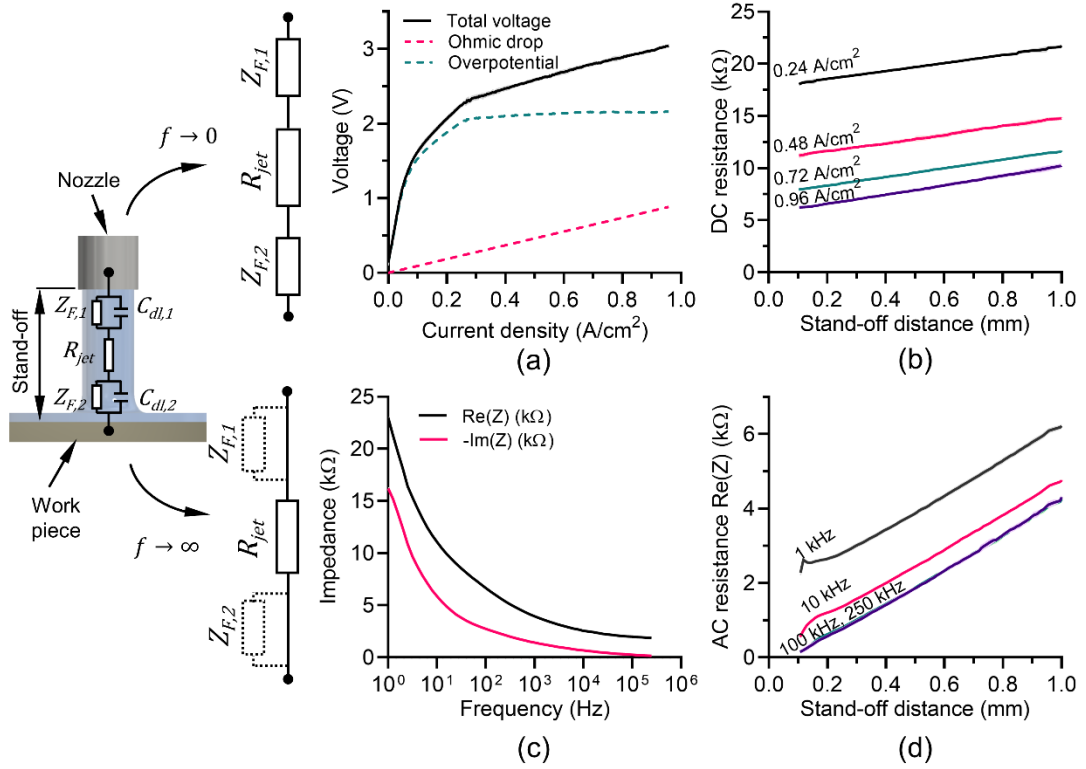


Fig. 4.1. The influence of electric excitation on the resistance response of the electrolyte jet. (a) Total voltage between the work piece and the nozzle as a function of average current density under DC, revealing the contributions of the ohmic drop and electrode overpotential. (b) Resistance as a function of stand-off distance under DC conditions at different current densities acquired during Z-translation of the nozzle. (c) Frequency response of the electrochemical jet the impedance demonstrating decay of Faradaic contributions with increasing AC frequency. (d) Resistance as a function of stand-off distance under AC conditions. The curves obtained for 100 kHz and 250 kHz overlap.

compared to the linear ohmic potential drop. This leads to an effective decay of Faradaic impedance with increasing current density as evidenced by the reduction in offset in the acquired resistance calibration curves (Fig. 4.1b) allowing for more accurate detection of jet resistance. At sufficiently high current densities, such as those typical for electrolyte jet machining, ($J > 10 \text{ A/cm}^2$ [36]) the ohmic drop is expected to be the dominating component of the inter-electrode gap voltage. However, significant anodic dissolution takes place at the work piece making this mode of operation unsuitable for non-destructive surface measurement.

The dissolution of the work piece can be mitigated by exploiting the properties of the electrical double layer at the electrode-electrolyte interface, which behaves as a capacitor C_{dl} in parallel with the Faradaic impedance (Fig. 4.1). Under DC conditions, the double-layer capacitor is open-circuit and has no contribution to the acquired resistance signal. However, when alternating current is applied, it contributes with an imaginary impedance component $Z_{C_{dl}} = -\frac{j}{2\pi f C_{dl}}$, where j is the imaginary unit, which results in overall decay of the interfacial impedance with increasing excitation frequency f . At high frequencies, the low-impedance path provided by the double layer capacitor allows the by-passing of the faradaic impedance, resulting in the ohmic resistance being the dominating component of the signal and mitigating work piece material dependent contributions. This behaviour is confirmed by the recorded impedance spectrum of the jet at a stand-off of 0.5 mm (Fig. 4.1c), where the imaginary component of the impedance approaches 0 with increasing frequency, along with a corresponding reduction in the real component, which decays to the value of the jet resistance as frequency approaches infinity. As demonstrated by the response curves obtained for the real impedance component (taken as a measure of resistance under AC conditions) and stand-off distance obtained at different frequencies (Fig. 4.1d), the non-ohmic contributions to the signal are reduced when increasing the excitation frequency allowing measurement of jet resistance at frequencies > 10 kHz in this configuration. The resistance under these conditions is expected to be only a function of the geometry of the electrochemical cell and the current flowing is predominantly charging and discharging current, thus minimising effects on the work piece surface due to Faradaic reactions like anodic

dissolution. This is verified by the optical images (Fig. 4.2) obtained from the work piece surface after the jet was set to dwell at fixed locations under different excitation conditions while maintaining a constant total anodic charge transfer of 12 mC. Significant material dissolution and pitting was observed under DC, while increasing AC frequencies >1 kHz resulted in reduced surface dissolution. No effects on the surface were visible optically at an excitation frequency of 100 kHz. The frequency at which negligible Faradaic current (corresponding to



Fig. 4.2. Effect on the workpiece surface at different excitation frequencies for a consistent total of 12 mC anodic charge transfer.

material removal) flows would be dependent on the values of the Faradaic impedance and the interfacial capacitance, which are affected by the surface chemistry. Reactions with higher impedance (less reactive work piece material) and high capacitance of the interface would lead to reduction of the required frequency for non-destructive measurement. However, the desired frequency for measurement can be optimised by acquiring the impedance spectrum for the material of interest (Fig. 4.1c) and selecting a frequency in the region where the real component of impedance reaches a plateau. It should also be noted that the AC experiments on Fig. 4.2 were conducted with the jet dwelling statically for 94.25 s. During scanning operations, the interaction time of the surface with the jet would be significantly reduced (for example ≈ 0.5 s for a scanning speed of 0.5 s and jet diameter of 0.25 mm) leading to insignificant effects on the surface. This demonstrates that non-destructive resistance measurements can be achieved within the electrolyte jet. Although a high

excitation frequency for measurement is beneficial for reduction of interfacial impedance, the impedance that can be measured accurately by the potentiostat/galvanostat reduces with increasing frequency due to the presence of stray circuit elements and in particular stray capacitance in the electronic circuitry of the device as discussed in Section 3.3.1 of Methodology. The excitation frequency for further experiments was hence fixed at 60 kHz, corresponding to a 1% accuracy limit of the instrument of approximately 20 k Ω (Fig. 3.3b) allowing a wide range of measurable resistance while maintaining an acceptably low interfacial impedance.

4.3. Understanding system response

4.3.1. Influence of work piece material and electrolyte

As discussed in Section 4.2, a key reason for choosing a high excitation frequency (>10 kHz) is to ensure that the ohmic resistance is dominating in the measured signal and the proposed method is made robust to the work piece material surface chemistry. To appraise this, resistance response curves were acquired for commercially pure nickel 201 and Ti-6Al-4V alloy in combination with two aqueous solutions of sodium chloride (NaCl) and sodium nitrate (NaNO₃) prepared with a similar electrical conductivity of 60.6 mS/cm ($\pm 2\%$), nozzle size, jet speed and excitation frequency were fixed. The average sensitivities obtained from least-squares linear fitting to the response curves (Fig. 4.3a) are summarised in Table 4.1. The observed variation of these values across the tested conditions is within 1.8% of the overall average of 4.31 k Ω /mm. Due to the uncertainty in conductivity of the electrolyte, it is not possible to attribute this variation to the specific material and electrolyte tested. Additionally, impedance spectra were acquired for frequencies 1 kHz – 250 kHz

at a fixed stand-off of 0.5 mm (Fig. 4.3b). The maximum difference in the real component of impedance, which is taken as a measure of resistance, was observed between the Ti-6Al-4V – NaCl system and the Ni - NaCl which was on average 273 Ω and appeared nearly uniform across the frequency range. This is consistent with the difference in intercepts of the lines of best fit reported in Table 4.1. This suggests that some influence of surface properties on the measured impedance may be present, due to different reactivity or different surface capacitance. However, it should be noted the specified positional repeatability of ± 20 μm of the Z-axis could result in an initial positioning error the nozzle above the surface when setting up a different work piece corresponding to 172 Ω at the observed sensitivity and on its own could account for 63% of the difference. Such an offset in resistance however, irrespective of its origin, would not affect the measurement accuracy for a homogenous

material as the proposed method relies on detection of relative resistance change corresponding to relative height, which is defined by the sensitivity.

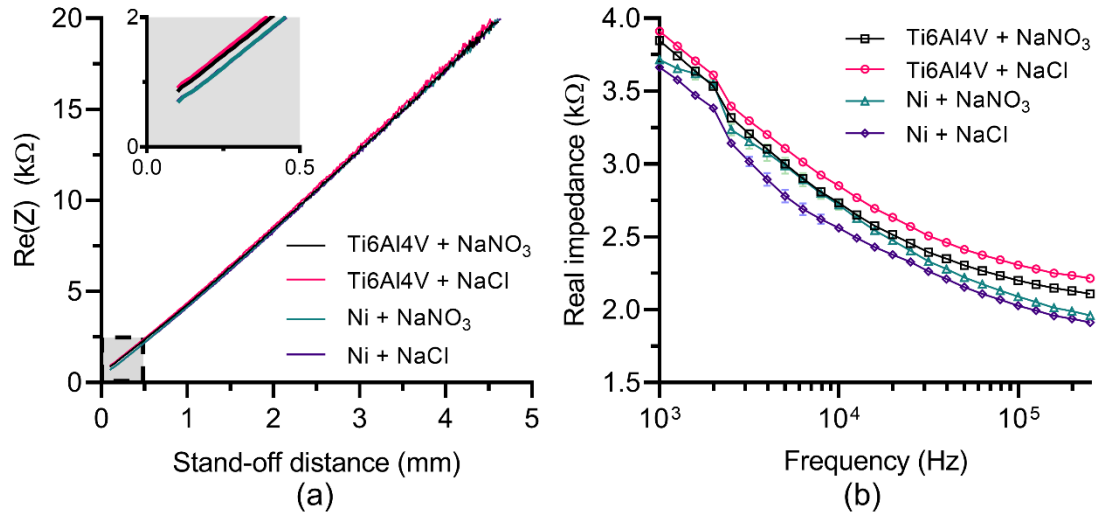


Fig. 4.3. Influence of the work piece material and electrolyte on the jet impedance response. (a) Exemplar resistance response curves acquired for four material-electrolyte combinations at fixed nozzle size and electrical conductivity for a frequency of 60 kHz. (b) The real component of the complex impedance of the electrochemical jet for frequency 1 kHz – 250 kHz at a fixed stand-off.

Table 4.1. Average sensitivity and intercept of resistance response for the tested material-electrolyte systems.

Material-electrolyte combination	Average sensitivity from resistance response ($k\Omega/mm$)	Intercept ($k\Omega$)
Ti-6Al-4V + NaNO ₃	4.26 (S.D. = 0.01, n = 3)	0.05 (S.D. = 0.02, n = 3)
Ti6-Al-4V + NaCl	4.29 (S.D. = 0.002, n = 3)	0.09 (S.D. = 0.03, n = 3)
Ni + NaNO ₃	4.39 (S.D. = 0.09, n = 3)	-0.17 (S.D. = 0.08, n = 3)
Ni + NaCl	4.30 (S.D = 0.001, n = 3)	-0.18 (S.D. = 0.04, n = 3)

4.3.2. Influence of system parameters

The effect of varying electrolyte conductivity, nozzle diameter and jet speed on the measurement system was appraised by acquiring resistance response

curves at a fixed frequency for a stand-off distance range of 0.1 – 5.0 mm with the resistance limited to 20 k Ω to remain within the defined measurement range for the galvanostat. As expected from Eq. (2.10), the resistance corresponding to a fixed stand-off distance increases with reduction of electrolyte conductivity and nozzle cross-sectional area (Fig. 4.4a-d). Average sensitivity values from 18.40 k Ω /mm (S.D. = 0.1 k Ω /mm) to 2.82 k Ω /mm (S.D. = 0.02 k Ω /mm) were observed for the investigated range of conductivity (13 mS/cm – 100 mS/cm) for a 0.23 mm nozzle and the variation of nozzle internal diameter d_{noz} from 0.1 mm to 0.31 mm at a conductivity of 60.6 mS/cm resulted in decreasing average sensitivity from 18.87 k Ω /mm (S.D. = 0.08 k Ω /mm) to 1.80 k Ω /mm (S.D. = 0.01 k Ω /mm). This demonstrates that the selection of electrolyte conductivity and nozzle size can be optimised for a particular required working range and vertical

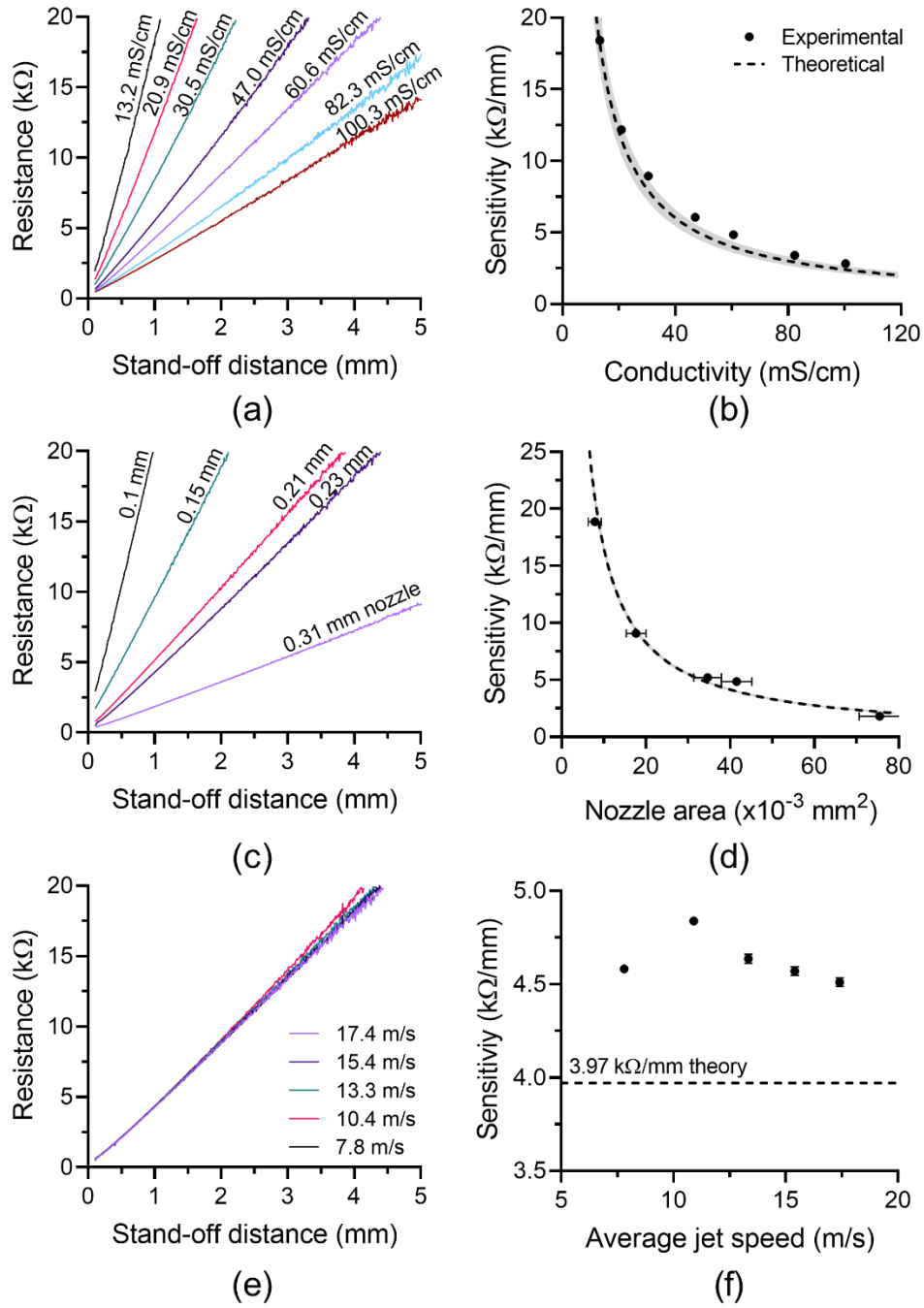


Fig. 4.4. Electrolyte jet resistance response. Exemplar resistance response curves and average sensitivities obtained at varying (a) – (b) electrolyte conductivities, (c) – (d) nozzle sizes (e) – (f) flow speeds. Vertical error bars on b, d, f correspond to standard deviation of the sensitivity obtained from 3 replicates of the response curves. The theoretical curves on b, d, are calculated from (2.10) , assuming the cross-sectional area of the jet is identical to the area of the nozzle, with the error regions representing the effect of uncertainty in nozzle area and conductivity, respectively.

resolution. This is analogous to changing probes of different size in

conventional coordinate measurement apparatus to suit a particular measurement challenge.

The resistance response and sensitivity were also affected by the average flow velocity of the jet (Fig. 4.4e-f). For a 0.23 mm internal diameter nozzle and conductivity of 60.6 mS/cm, a maximum sensitivity of 4.84 kΩ/mm (S.D. = 0.01 kΩ/mm) was obtained at flow speed of 10.4 m/s and this reduced to 4.51 kΩ/mm (S.D. = 0.02 kΩ/mm) upon increase in speed up to 17.4 m/s. This variation of sensitivity with jet speed is likely caused by a change in jet shape dependent on the flow condition. It is known that the shape of a falling round liquid jet is not cylindrical, but exhibits contraction downstream of the nozzle [129], resulting in a reduction of cross-sectional area and a corresponding increase in resistance. This contraction is expected to become less significant with increasing of jet speed which is consistent with the observed reduction in sensitivity on Fig. 4.4f. The presence of a maximum sensitivity obtained at 10.4 m/s could be indicative of a change in flow regime. Considering the Reynolds number of the flow in the nozzle $Re = v_{jet} d_{noz}/\nu$ where v_{jet} is the average flow speed, d_{noz} is the nozzle diameter and $\nu \approx 1 \text{ mm}^2/\text{s}$ is the kinematic viscosity of water at room temperature, the change of flow velocity from 7.8 m/s to 10.4 m/s corresponds to a change in Re from 1794 to 2392. This range of Re is likely to include the critical point for the onset of turbulence in the nozzle [130], affecting the jet shape and the corresponding resistance response. This investigation demonstrates that electrolyte conductivity, nozzle diameter and flow speed are the key physical and geometric parameters affecting the proposed measurement method and are critical for the calibration of the system.

Examining further the level of agreement between the acquired response curves and the simple linear model based on (2.10), a significant non-linearity was observed in the variation of resistance with stand-off distance. An exemplar residual plot for the 0.23 mm nozzle, 60.6 mS/cm electrolyte conductivity and 10.4 m/s jet speed, is shown on Fig. 4.5a. The difference between the measured stand-off distance for a certain resistance and the value predicted by the linear regression fit from the acquired response curve is non-uniform across the measurement range and can reach values as high as 0.1 mm for a predicted stand-off of 0.23 mm. The local gradient of the corresponding resistance response curve evaluated over a moving 0.23 mm interval increases from 4.13 k Ω /mm at 0.1 mm-off stand distance to 5.34 k Ω /mm at 4.2 mm stand-off (Fig. 4.5b) revealing a significant difference from the behaviour of a cylindrical conductor and demonstrating that the sensitivity S in Eq. (3.1) is function of the stand-off distance. This increase in local gradient is consistent with the hypothesised contraction of the jet at a distance from the nozzle exit and corresponds to 12.3% reduction in jet diameter. The obtained gradient values significantly overestimate the calculated from Eq. (2.10) sensitivity of 3.97 k Ω /mm based on the internal nozzle diameter. These results suggest that the application of the ideal cylinder model is inadequate for accurate prediction of distance from the measured resistance and the obtained sensitivity values from global linear regression fitting are only valid for a limited portion of the measurement range. Higher-accuracy calibration can be achieved by directly interpolating the calibration curve or applying a piecewise linear approximation with local sensitivity as a function of measured absolute resistance. For further experimentation reported here, the linear approximation was performed only

within the measurement range of features to minimise errors originating from this non-linearity.

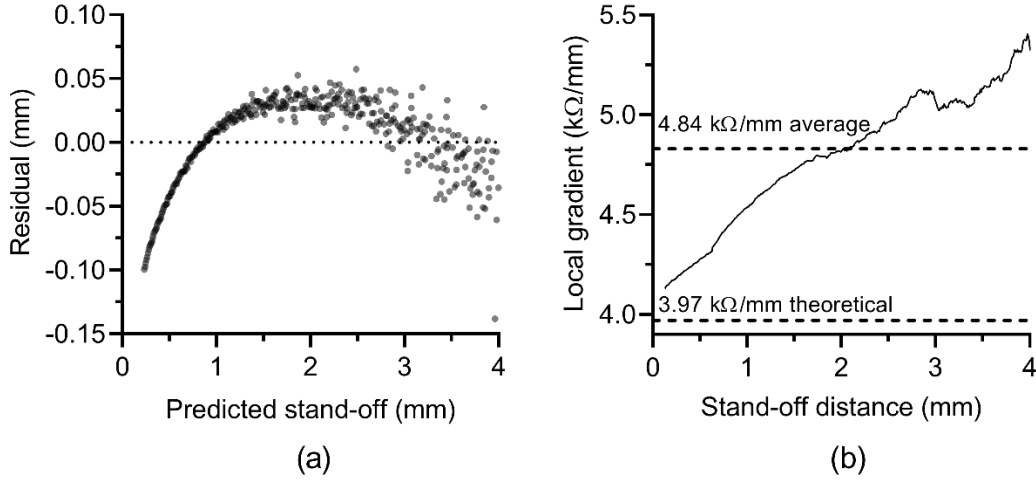


Fig. 4.5. Non-linearity in resistance response. (a) Exemplar residual plot against predicted stand-off distance obtained for $d_{noz} = 0.23$ mm, $\sigma = 60.6$ mS/cm, $v_{jet} = 10.4$ m/s from a linear regression fit to the response curve. (b) Local gradient evaluated from the response curve ($d_{jet} = 0.23$ mm, $\sigma = 60.6$ mS/cm, $v_{jet} = 10.4$ m/s) by linear regression over a moving 0.23 mm interval.

4.4. Measurement of surface features

The performed resistance response calibration was applied to measure reference grooves in a step height specimen at different parameters including nozzle diameter, flow velocity, scanning speed, electrolyte conductivity and stand-off distance. Considering the non-linearity of the response curves discussed in the previous section, a linear approximation was performed only in the expected range of absolute stand-off distance for the measured features to obtain the required sensitivities for each parameter set.

4.4.1. Parameter influence on spatial resolution

Distortion of the measured profiles was observed across all applied parameters (Fig. 4.6a). This is caused by the limited spatial resolution resulting from the

finite size of the jet leading to a 'filtering' effect. To quantify this, for each measured profile the 5-95% rise distance (defined in Section 3.7.2 of Methodology) was calculated and presented in Fig. 4.6b-f. The size of the nozzle was found to be the dominating factor in determining the spatial resolution of the system. The 5% - 95% rise distance increased with increasing nozzle size (Fig. 4.6b). The minimum rise distances obtained with a 0.1 mm diameter nozzle were 0.17 mm (S.D. 0.04 mm), 0.3 mm (S.D. 0.04 mm), 0.52 mm (S.D. 0.01 mm) for the 30 μm , 196 μm and 495 μm groove respectively compared to 0.03 mm, 0.21 mm and 0.54 mm rise distances from the stylus measurement. The rise distance increased to 0.3 mm (S.D. 0.03 mm), 0.56 mm (S.D. 0.03 mm), 0.68 mm (S.D. 0.02 mm) for the 30 μm , 196 μm and 495 μm groove respectively when using the 0.31 mm internal diameter nozzle with other investigated parameters (Fig. 4.6c-f) showed negligible influence on the 5-95% rise distance for a fixed nozzle internal diameter of 0.23 mm. This demonstrates that these parameters can be selected based on the desired measurement range and the available capabilities of the signal acquisition electronics without compromising the spatial resolution of the measurement.

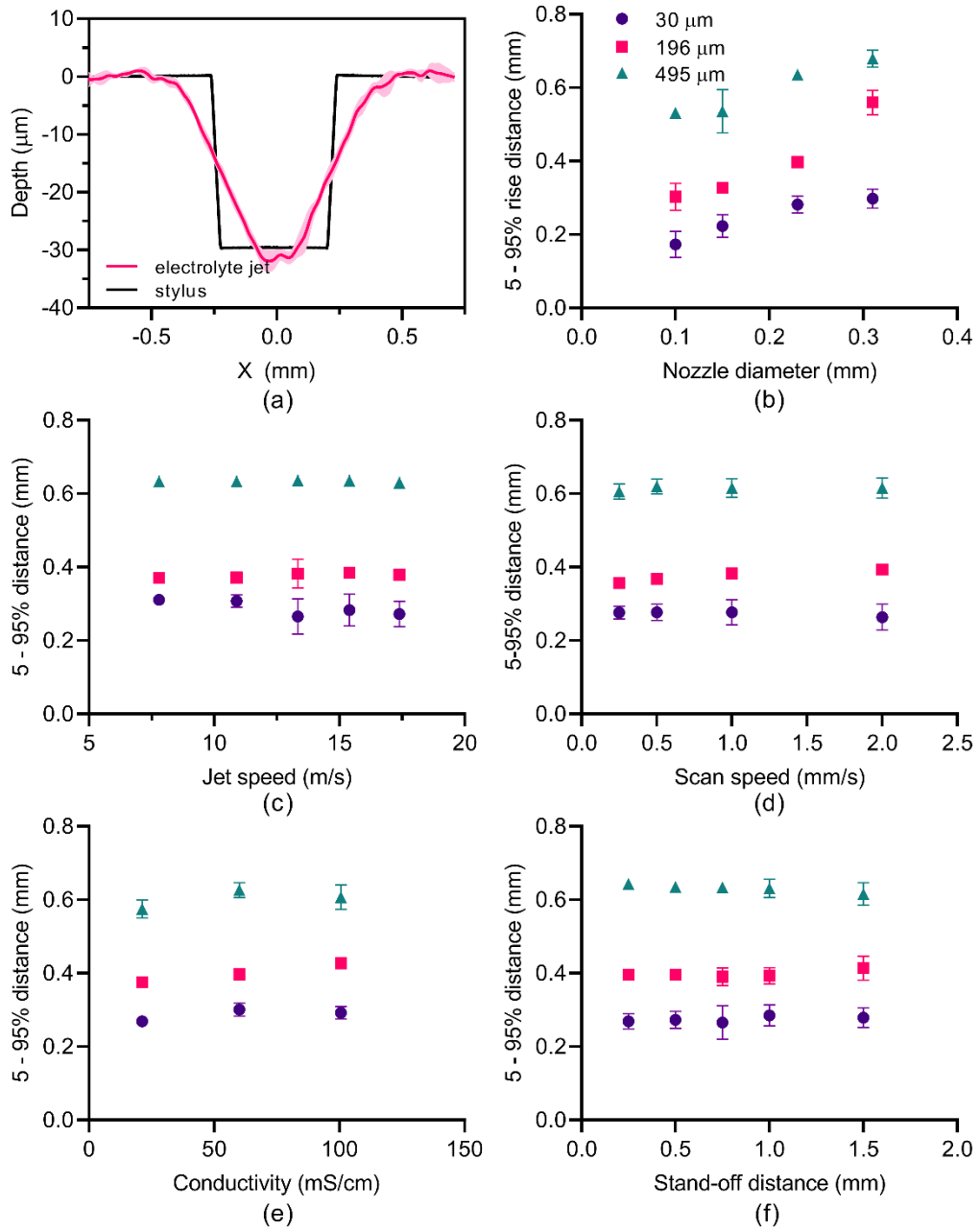


Fig. 4.6 Influence of measurement system parameters on spatial resolution. (a) Profile acquired with $d_{jet} = 0.23$ mm, $\sigma = 60.6$ mS/cm, $v_{jet} = 10.4$ m/s demonstrating the distortion of sharp edges. The profile represents an average from 5 replicates with error regions corresponding to combined uncertainty due to repeatability and uncertainty in conductivity. 5% - 95% rise distance as a function of (b) nozzle size, (c) jet speed, (d) scanning speed, (e) conductivity, (f) stand-off distance. Error bars represent standard deviation of 10 replicates (5 repeated measurements, both sides of the groove). Measured profiles for the calculation of rise distances are presented in Fig. B.1. of the Appendix.

4.4.2. Mathematical modelling of resistance response

To utilise the proposed measurement system in practice it is desirable to obtain a transfer function that could predict the output for an arbitrary surface and hence define the limitations in terms of spatial resolution evident in Fig. 4.6. This would require the computation of the fluid dynamic interaction of the traversing jet with the surface and then numerical solution of Laplace's equation within the domain defined by the electrolyte at every time instant with an approach identical to the one applied in [11]. This can be extremely computationally expensive for a realistic surface topography, so instead a simplified analytical approximation of the transfer function was proposed. The results presented in Section 4.4.1 demonstrated that the influence of fluid dynamics and other parameters is relatively insignificant compared to the influence of nozzle geometry, hence justifying an approximate approach.

The electrolyte jet is assumed to be an ideal cylinder, neglecting the shape of the impingement region, and the nozzle is assumed to be sufficiently far from the surface, so that the electric potential is uniform at the nozzle exit plane. It is also assumed that potential gradients are only significant in the Z-direction. The current density on the anode surface is approximated as $J(x, y) = \frac{U\sigma}{l}$ where U is the potential difference applied between the nozzle and the work piece and $l(x, y)$ is the local vertical distance between the nozzle exit plane and the work piece surface. The overall measured resistance is obtained from calculating the total current by integrating the current density over the impingement area on the work piece surface:

$$R_{jet} = \frac{U}{\iint J dA} = \frac{1}{\sigma \iint \frac{1}{l} dA} \quad (4.1)$$

The equivalent distance output by the measurement system corresponding to the resistance of the cylindrical jet according to Eq. (2.10) is:

$$L_{eq} = \frac{A}{\iint \frac{1}{l} dA} \quad (4.2)$$

This model is equivalent to considering the overall resistance of the jet as resulting from a network of discrete, parallel resistors connecting the work piece surface and the nozzle plane with resistance values proportional to the local vertical distance (Fig. 4.7a). However, performing the integration above with $A = A_{noz}$ predicts less distortion and better representation of the groove shape than observed experimentally (Fig. 4.7b). This is due to the fact that the jet expands and deviates from the cylindrical geometry upon impingement and the current density is non-uniformly distributed in the impingement significant current flow outside the area defined by the nozzle bore [11]. Taking the double integral over an area $A = 4A_{noz}$ (corresponding to twice the nozzle diameter) resulted in a closer agreement with the experimental results (Fig. 4.7b). This is consistent with the observations in EJM research that the affected region is larger than the area of the nozzle [38]. A more general form of Eq. (4.2) is proposed by applying a weight function $w(x,y)$ to the local distances to correct for the widening of the jet and the non-uniformity of the current. This results in the following expression:

$$L_{eq} = \frac{\iint w dA}{\iint \frac{w}{l} dA} \quad (4.3)$$

The corresponding general resistance change measured at any point on the surface relative to the initial value is then:

$$\Delta R = S \Delta L_{eq} \quad (4.4)$$

where S is the experimentally calibrated sensitivity as described in Section 3.7.1 in Methodology. Although more refined models can be sought in the future with optimised expressions for $w(x, y)$, for the purposes of this work it is assumed that $w(x, y) = 1$ within the region defined by a circle with diameter $2d_{noz}$ and $w(x, y) = 0$ otherwise.

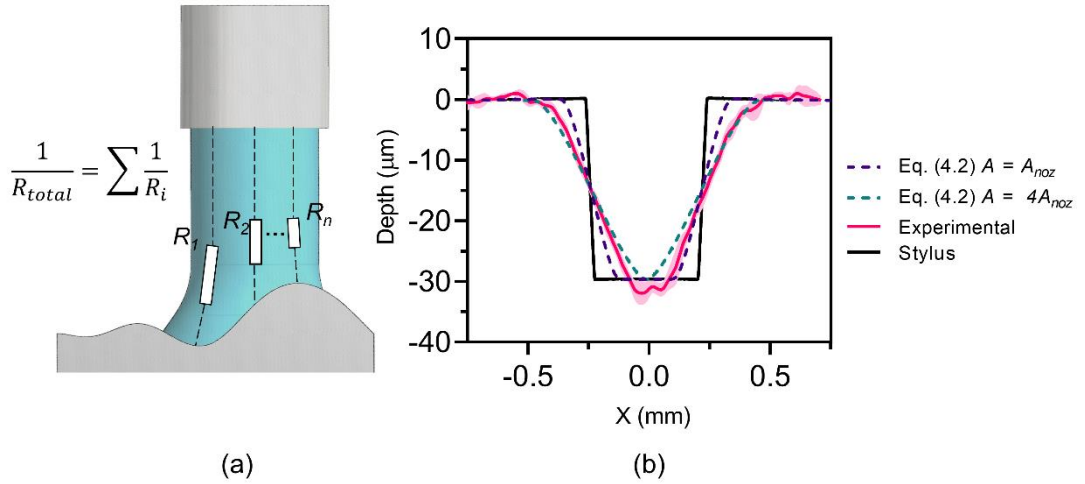


Fig. 4.7. (a) Representation of the inter-electrode gap as a network of parallel resistors to approximate the resistance as the jet traverses across surface features. (b) Calculated equivalent length according to Eq. (4.2) with different integration limits.

4.4.3. Step height measurement results

Acquired profiles from the step height reference specimen with different nozzle sizes along with the expected measurement output from Eq. (4.3) are presented in Fig. 4.8. The applied sensitivities were 19.12 k Ω /mm (S.D. = 0.12 k Ω /mm), 8.92 k Ω /mm (S.D. = 0.06 k Ω /mm), 4.39 k Ω /mm (S.D. = 0.01 k Ω /mm) and 1.70 k Ω /mm (SD =0.01 k Ω /mm) for the 0.1 mm, 0.15 mm, 0.23 mm, and

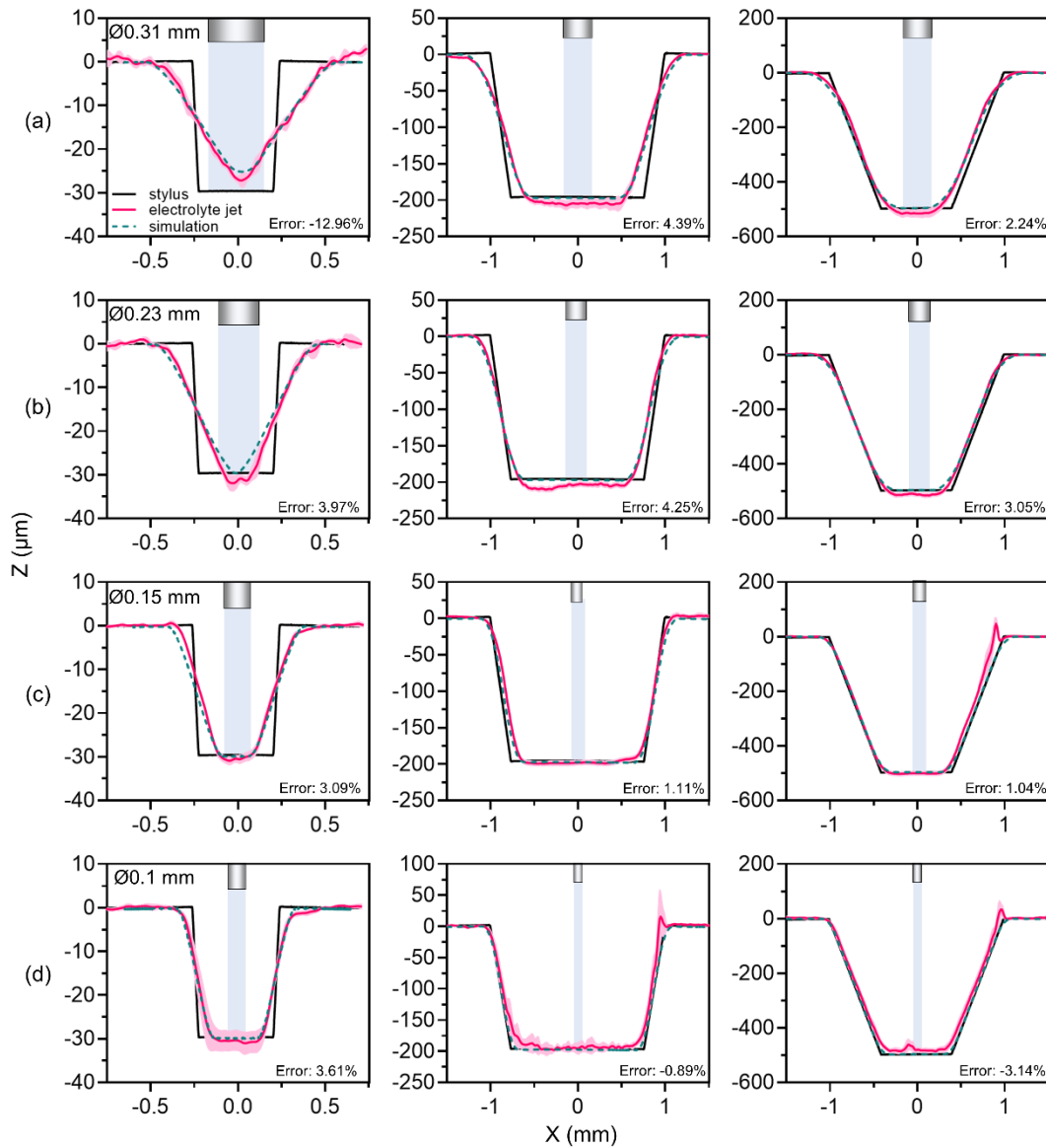


Fig. 4.8. Acquired and simulated measurements of reference grooves for (a) 0.31 mm, (b) 0.23 mm, (c) 0.15 mm and (d) 0.1 mm nozzles respectively. Experimental profiles represent average from 5 replicates with error regions corresponding to combined uncertainty due to repeatability, uncertainty in conductivity and uncertainty in the calibrated sensitivity.

0.31 mm nozzles respectively and were obtained by linear approximation in the expected range of absolute stand-off distance of 0.5 – 1.0 mm. Further measured profiles across all system parameters and the applied sensitivities can be found in Appendix B. As seen in Fig. 4.8, the proposed transfer function captures the distortion of the measured profile. This is also evident when comparing the 5-95% rise distance predicted by the model with experiment (hollow markers on Fig. 4.9) – for the 0.1 mm nozzle rise distances of 0.16 mm, 0.27 mm and 0.56 mm for the 30 μ m, 196 μ m and 495 μ m grooves are predicted compared to the corresponding experimental values of 0.17 mm (S.D. 0.04 mm), 0.3 mm (S.D. 0.04 mm), 0.52 mm (S.D. 0.01 mm). The largest discrepancy in 5%-95% rise distance between the model and experiment was observed for the 196 μ m groove measured with 0.23 mm nozzle where the simulation predicts a rise distance of 0.33 mm compared to an experimental value of 0.28 mm (S.D. 0.03 mm), thus overpredicting by 18%. Both the model and the experimental data show that the 0.5 mm wide (30 μ m depth) is at the limit for the spatial resolution of the 0.31 mm internal diameter nozzle and the full depth of the groove is not transmitted by the measurement system. The model transfer function predicts 21.3% depth reduction compared to the depth measured by the stylus, while a 12.96% reduction was observed experimentally. The obtained results however demonstrate that the investigated geometries are reliably measurable without detailed consideration of the fluid dynamics. It can be expected that concave surface features, which prevent effective liquid removal from the impingement region such as high-aspect ratio grooves are likely to challenge accurate measurement. However, these are generally not achievable by EJM, so the proposed method proves to

be suited to considering the application of on-machine pre- and/or post-machining measurement for EJM.

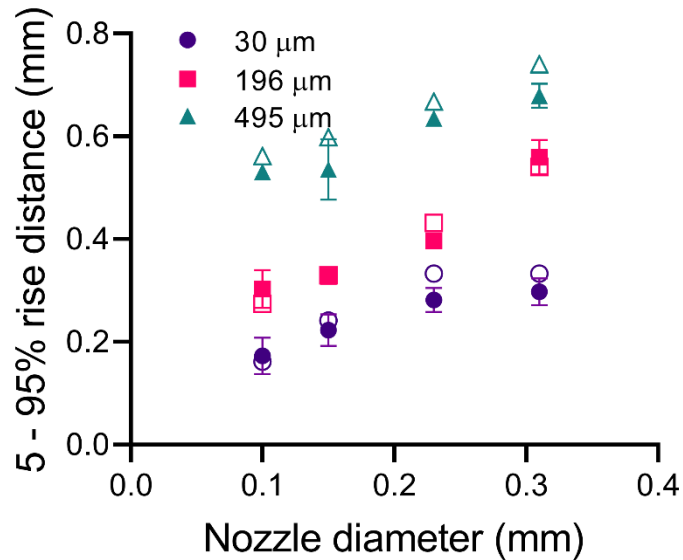


Fig. 4.9. 5-95% rise distance as a function of nozzle size for the three grooves. Hollow markers represent values obtained from analysing the simulated measured profiles for each nozzle, Error bars represent standard deviation of 10 replicates (5 repeated measurements, both sides of the groove)

Fig. 4.10 summarises the results of the depth measurements of the reference grooves in terms of the relative error compared to the stylus profilometer measurement. The average error remained within 4.5% of the depth of the groove with the exception of the measurement of the 30 μm deep 0.5 mm wide groove with the 0.31 mm nozzle whose depth was reduced due to the limited spatial resolution.

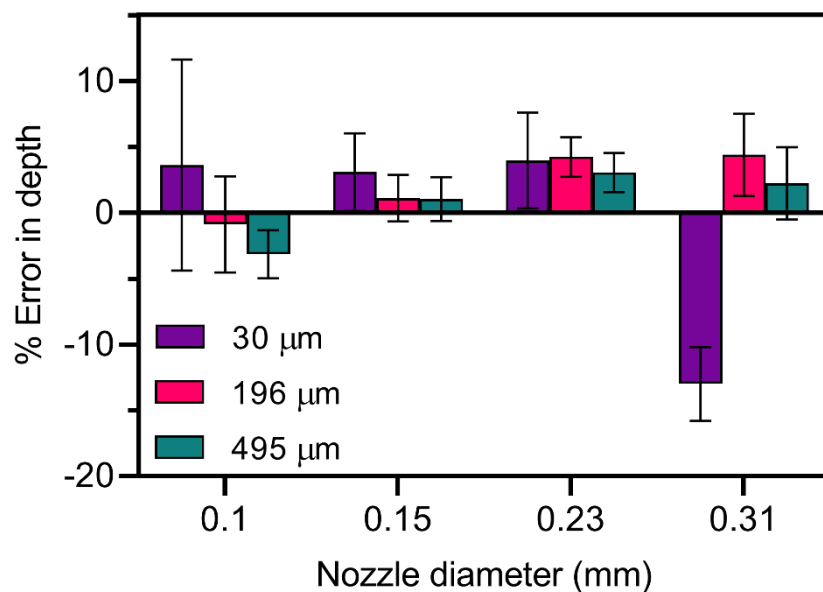


Fig. 4.10. Measurement error for the depth of reference grooves across nozzle sizes. Error bars represent the combined uncertainty due to repeatability, uncertainty in conductivity and uncertainty in calibrated sensitivity relative to the depth of the respective groove measured with a stylus profilometer.

4.5. Electrochemical jet areal surface evaluation

Demonstration of areal surface topography measurement was performed by scanning the electrolyte jet over a 2017 British pound coin (Fig. 4.11a) using an 0.1 mm nozzle and 60.6 mS/cm electrolyte. The raw resistance data was converted to surface height (Fig. 4.11b) by applying a linear regression to the corresponding response curve (Fig. 4.4c) within the range of measured absolute resistance 3.5 – 9 k Ω resulting in a sensitivity of 18.46 k Ω /mm (S.D. = 0.02 k Ω /mm). The overall surface height variation of the coin obtained from focus variation microscopy was 331 μm . The spatial error map calculated following a comparison with a focus-variation microscopy data set (Fig. 4.12a) indicates the error is most significant around edges and sharp features.

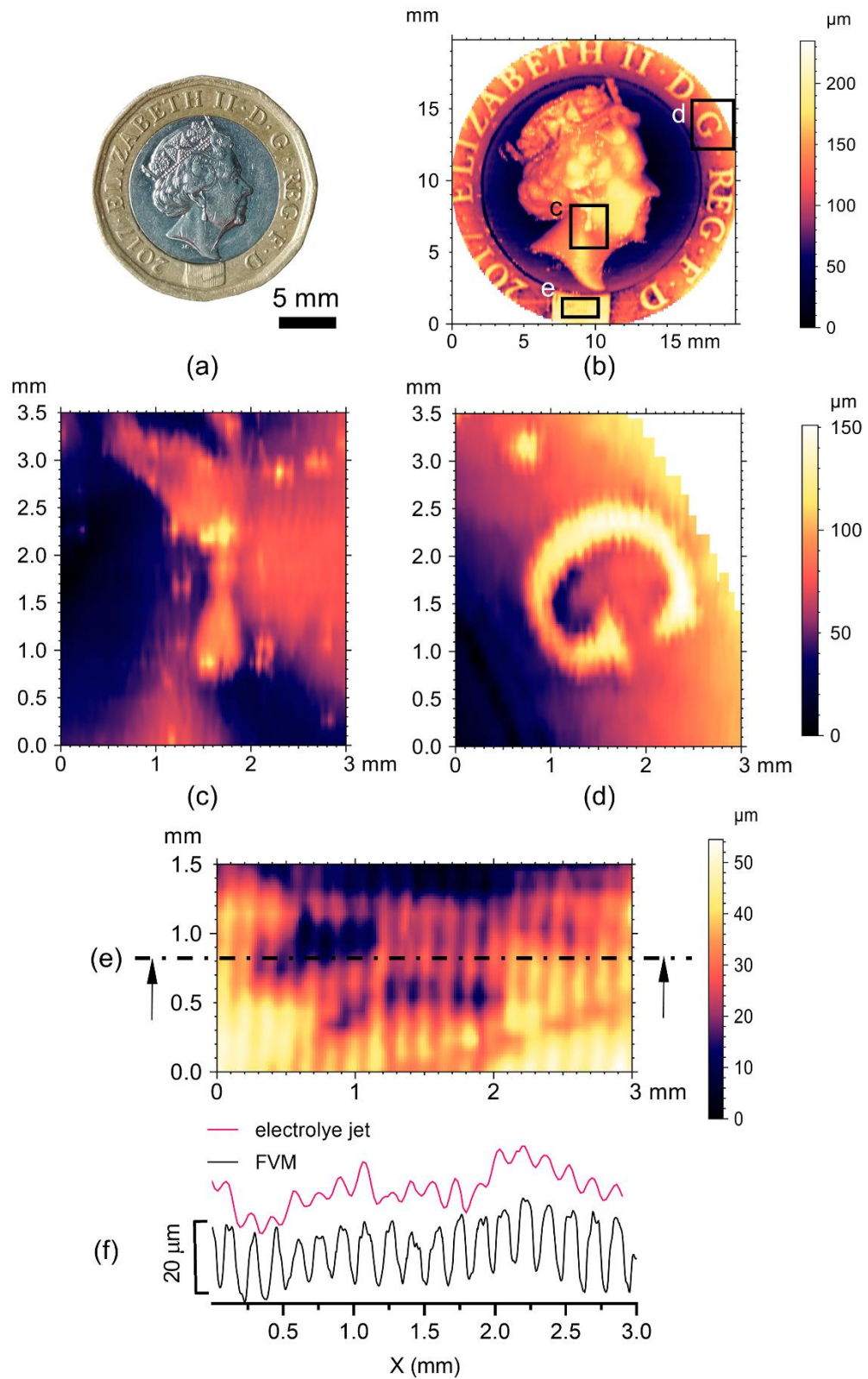


Fig. 4.11. Areal surface imaging with an electrolyte jet. (a) The British pound coin. Scale bar corresponds to 5 mm. (b) Surface height data acquired through the electrolyte jet measurement method. (c) – (e) Details of the measured surface – ‘earring’, ‘letter ‘G’ and ‘grating feature’ respectively. (f) Extracted profiles from the ‘grating feature’ acquired with electrolyte jet and focus variation microscopy.

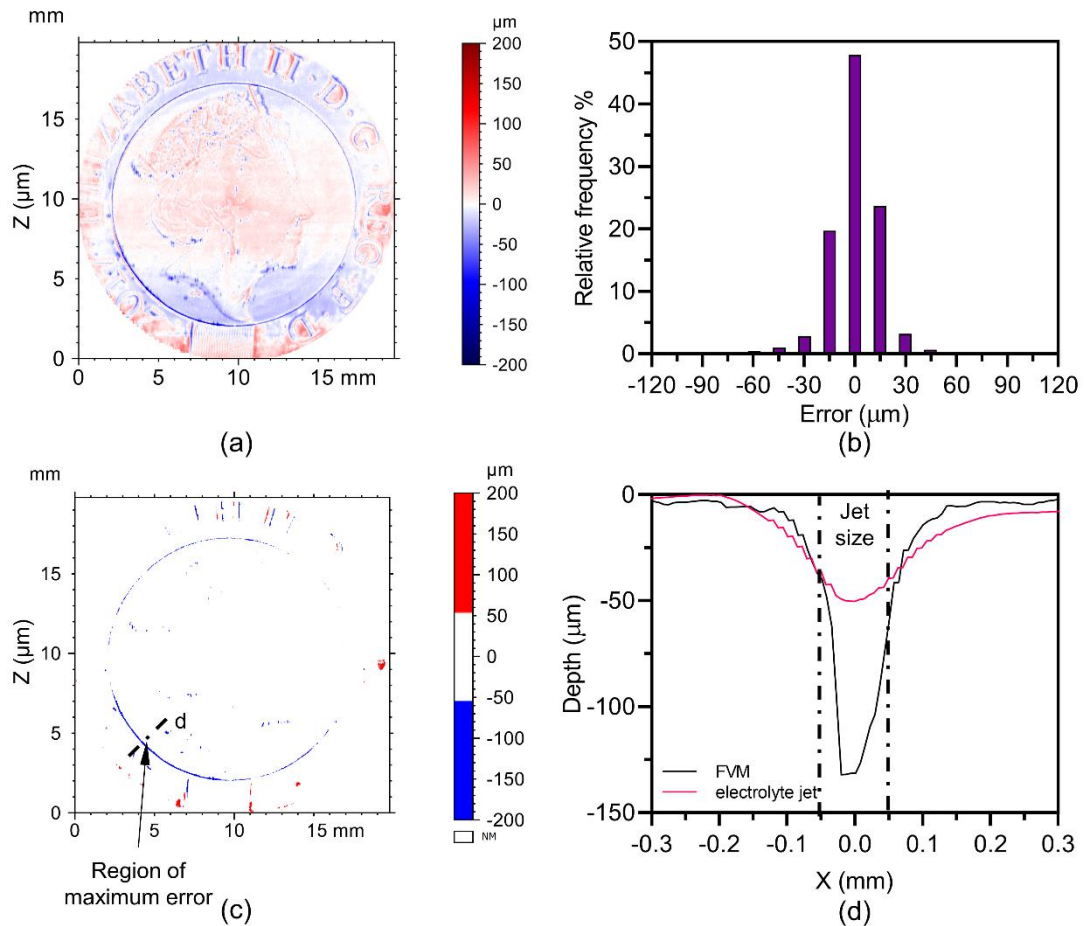


Fig. 4.12. Error in the electrolyte jet areal measurement compared to focus variation microscopy. (a) Spatial error map compared to FVM measurement with x5 objective. (b) Error distribution. (c) Error map against focus variation microscopy highlighting errors larger than 52 μm . (d) Profile extracted from the gap between the two parts of the coin, showing a high-aspect feature not measurable by the electrolyte jet.

Although errors as large as 193 μm in height were observed, these errors are localised and occur for features of the coin which are below the spatial resolution of the jet such as the gap where the inner disc meets the outer ring (Fig. 4.12c). Considering the error frequency distribution (Fig. 4.12b) data points with errors larger than $\pm 52 \mu\text{m}$ only account for 1.03% of the error surface while for approximately 91.2% of the data points the error is within $\pm 22.5 \mu\text{m}$. The error map of is presented with an exaggerated colour scale in Fig. 4.12c to highlight the locations of errors larger than 52 μm . The gap between the disc and the ring represents a high-aspect feature on the scale of jet size

(Fig. 4.12d) and is not measurable accurately through the proposed approach. The root mean square error of the whole topographic data set is 14.6 μm , validating the method proposed in this work as a capable but facile technology for measurement of large-scale surface topography. The capability of this technique is evident in resolving the grating-like surface features at the bottom of the coin (Fig. 4.11e) that have a wavelength of approximately 160 μm , however the amplitude has been attenuated to approximately 30% of the value measured by FVM (Fig. 4.11f) demonstrating that the scale of these features is at the limit for the currently achievable lateral resolution with the electrolyte jet. The British pound coin also presents an example of measurement of a non-homogenous material. The composition of the coin material is specified as 70% Cu, 24.5% Zn, 5.5% Ni, but the inner disc is Ni-plated. As demonstrated in Section 4.3.1. the sensitivity of system to change in height is only defined by the conductivity of the electrolyte and nozzle geometry and is independent of the work piece material. However, as shown in Table 4.1 and Fig. 4.3a, a different material may offset the calibration curve and hence add a bias to the measurement. Consequently, in the measurement of the coin, any influence of the differential compositions of the inner disc and the outer ring would be expected to manifest itself as an offset in measurement error between the two regions on Fig. 4.12a. Such variation in measurement performance between the two regions of the coin however was not observed, suggesting that electrolyte jet measurement approach is robust to material variation. Further quantitative studies are required to evaluate the influence of surface composition on the measurement independent of surface topography.

4.6. Summary

This chapter demonstrated that the measurement of electrical resistance of the electrolyte jet provides a low-cost method for on-machine surface form and large-scale surface topography measurement in EJM. The application of alternating current excitation (> 10 kHz) between the nozzle and the work piece reduced electrode overpotentials and inhibited the corrosion of the electrodes, allowing non-destructive surface geometry appraisal.

Electrolyte conductivity, nozzle cross sectional area and the jet flow speed were shown to be the dominant factors determining the resistance response to change in stand-off distance and the measurement system requires calibration for a set of these factors. The sensitivity of the system proved to be robust to material composition without significant effects on calibration caused by different surface chemistries of commercially pure nickel and Ti-6Al-4V alloy. The measurement of heterogenous surfaces however requires further research since variation in material chemistry may alter the offset of the resistance calibration curve as a function of distance.

Significant non-linearity was found in the resistance response, which can be attributed to the shape of the impinging jet, but a linear approximation over the expected measurement range resulted in an accuracy of $\pm 4.5\%$ for depth measurements of reference grooves within the spatial resolution of the jet.

Analysis of the rise distance of the measurement response when traversing the electrolyte jet across the reference grooves demonstrated that jet diameter determines the spatial resolution of the electrolyte jet measurement, while this remains stable with respect to other system parameters. A simple analytical

expression was proposed to approximate the transfer function of the measurement system, which provided an adequate agreement with the experimental response. The developed on-machine measurement capability was successfully demonstrated by performing areal measurement of a British pound coin with a root-mean-square error of 14.6 μm compared against FVM measurement.

The proposed approach has inferior performance in terms of resolution and accuracy compared to conventional optical or contact metrology, however it is valuable for on-machine metrology in EJM since it has the benefits of low cost and not requiring a separate measurement sensor. SECM, which is an established electrochemical surface characterisation method has achieved sub-micrometre spatial resolution through the fabrication of miniature electrodes, but it is a bath-based process which is suited to laboratory studies of small areas and has not been used for manufacturing metrology of engineering components. In contrast, the proposed jet-based system has a spatial resolution limited by jet size, but the “probe” can be easily manipulated around more complex geometries. The operating principle also does not require the use of redox species in the electrolyte, relying on the measurement of jet length through impedance. In the proposed application to on-machine metrology for EJM, the overall geometry of the work piece located in the machine tool can be measured before machining to inform path planning and the quality of large-scale features can be assured through post-machining inspection.

5. In-process monitoring for EJM

5.1. Introduction

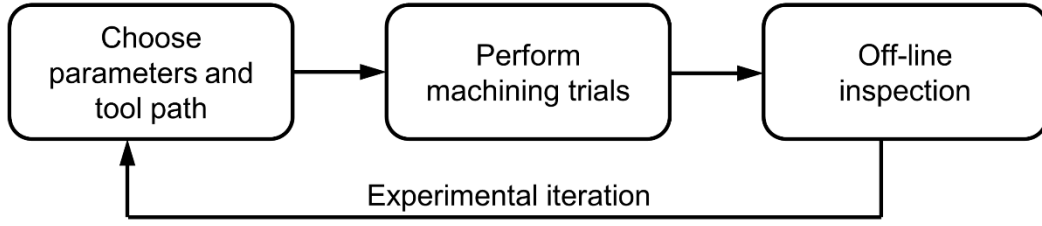
The literature review highlighted that the large number of parameters affecting material removal and resulting surface geometry in EJM often necessitates an iterative experimental optimisation including a series of cutting trials with the material of interest followed by inspection of the produced geometry to arrive at a parameter set that delivers the required design intent (Fig. 5.1a). The capability to perform on-machine surface measurement presented in Chapter 4 illustrates the potential to accelerate this process by eliminating the off-line inspection step. However, further improvement can be delivered through an accurate simulation of machining operations based on a minimal amount of experimental calibration to enable model-based tool path planning and parameter selection instead of the commonly used “trial-and-error” approach.

Section 2.3 of the literature review discussed the current state of the art of process models for EJM. While the multi-physics approaches provide a valuable insight to the fundamentals of the process, their application to more complex machining operations and integration with machine control and path planning software is challenging due to the computational expense. Analytical models have been developed for predictive planning of laser cutting [131] and abrasive water jet machining [132], while more recently a generic energy beam process model [133] has been applied for the generation of free-form surfaces with both processes following an initial model calibration. The application of this type of models to EJM has received limited attention.

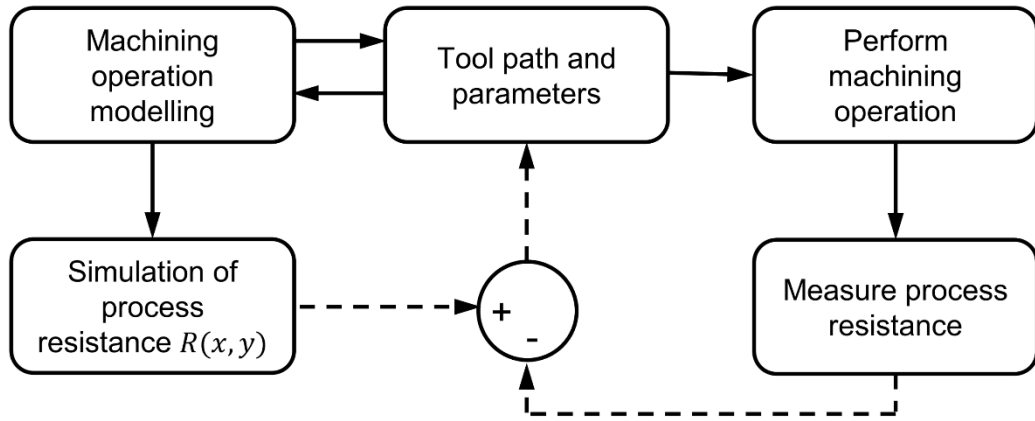
Further to the development of process models, the capability to monitor the performance of the live machining process and make adaptations could further improve the fidelity of machined features while reducing optimisation trials. During machining, the resistance of the IEG in EJM is affected by the evolving shape of the work piece since the distance between the nozzle and work piece increases with increasing depth of cut. It could be expected that within a certain feature aspect ratio (shown in this chapter to be < 0.5), fluid dynamic effects have a negligible influence, and each surface texturing operation would have a specific and predictable electrical resistance response, which could be monitored for quality control purposes. Deviations from the expected response could indicate dimensional errors compared to the design or disturbances to the process such as inert phases in the work piece material or fluid dynamic instabilities. Understanding and predicting process resistance would enable live in-process measurement and closed-loop control of machining parameters to compensate for disturbances. Such an approach is presented in Fig. 5.1b – prior to machining the process model is used to select the parameters and tool path to achieve the required surface geometry. In addition, the expected resistance response for the machining operation is simulated. The measured resistance in-process is compared to the prediction and on-the-fly adjustments to machining parameters can be made to reduce any detected error.

In this chapter, advances are made towards the approach in Fig. 5.1b by analytical modelling of the work piece surface evolution in EJM and the corresponding resistance response. The model is validated experimentally for a number of machining operations and its limitations are investigated. This lays the foundation for development of closed-loop control. A number of applications

for the developed path planning and in-process monitoring strategies are presented.



(a)



(b)

Fig. 5.1. (a) Conventional “trial and error” approach for surface texturing through EJM. (b) Advanced EJM enabled by predictive modelling and in-process resistance monitoring.

5.2. Geometric modelling of surface evolution in EJM

The evolution of the surface $z(x, y, t)$ of the anodic work piece in electrochemical machining as a function of spatial coordinates (x, y) and time t is governed by [134] :

$$\frac{\partial z}{\partial t} = -\eta \frac{k}{\rho} J \sqrt{1 + (\nabla z)^2} \quad (5.1)$$

where $J(x, y, t)$ is the current density distribution at the surface, η is the current efficiency, k and ρ are the electrochemical equivalent and the density of the

work piece material respectively and ∇z is the gradient of the surface. The physics-based approaches to solving the above equation for EJM involve the numerical computation of potential and current density distributions in the domain defined by the shape of the impinging jet. Further experimental calibration may be required to obtain values for η for the specific material of interest [39]. Instead of simulating the current density distribution and calibrating for the constants in Eq. (5.1) , the energy beam process model proposed by Axinte et al. in [133] is adapted to EJP, so that surface evolution is expressed by

$$\frac{\partial z}{\partial t} = -P(\mathbf{x}', t)\sqrt{1 + (\nabla z)^2} \quad (5.2)$$

where $P(\mathbf{x}', t)$ defines removal rate as function of the local coordinates $\mathbf{x}' = (x', y')$ in the coordinate system attached to the nozzle. $P(\mathbf{x}', t)$ is dependent on the shape of the evolving work piece surface because jet hydrodynamics and current density distribution are altered as depth of cut progresses. However, under constant current conditions the volumetric material removal rate (MRR) during the machining process remains constant at any instant in time:

$$MRR = \iint \frac{\partial z}{\partial t} dA = \iint P(\mathbf{x}', t)\sqrt{1 + (\nabla z)^2} dA = const \quad (5.3)$$

The above equation can be used to provide an approximation for the change of $P(\mathbf{x}', t)$ with work piece geometry. If $P_0(\mathbf{x}')$ is the initial removal rate function at the start of the machining process for a flat surface ($\nabla z \approx 0$), it is proposed here that $P(\mathbf{x}', t)$ is estimated from:

$$P(\mathbf{x}', t) = \frac{MRR}{\iint P_0(\mathbf{x}') \sqrt{1 + (\nabla z)^2} dA} P_0(\mathbf{x}') \quad (5.4)$$

This enforces that Eq. (5.3) remains balanced. For the modelling of machining involving the translation of the jet along a tool path, Eq. (5.2) is parametrised with respect to arc length s instead of time as proposed in [133] resulting in

$$\frac{\partial z}{\partial s} = \frac{-P(\mathbf{x}', s) \sqrt{1 + (\nabla z)^2}}{v_f} \quad (5.5)$$

where v_f is the magnitude of the feed rate, since $\frac{ds}{dt} = v_f$. For the simulation of EJM using this model, an initial estimate for the material removal rate function $P_0(\mathbf{x}')$ is required and this can be obtained experimentally as described in Section 5.2.1. The proposed process model is implemented in the Python programming language with the work piece surface is discretised over a regular rectangular grid with grid spacing of 0.005 mm. The machine tool path is defined as a set of control points in the x-y plane and corresponding feed rate defining the jet translation speed for the motions between points. The jet nozzle position interpolates linearly between these points with the surface evolution and the resistance along the tool path calculated at every arc length step $ds = 0.005$ mm. The simulation is completed when the full length of the tool path is traversed. A flow chart summarising the implementation is shown in Fig. 5.2.

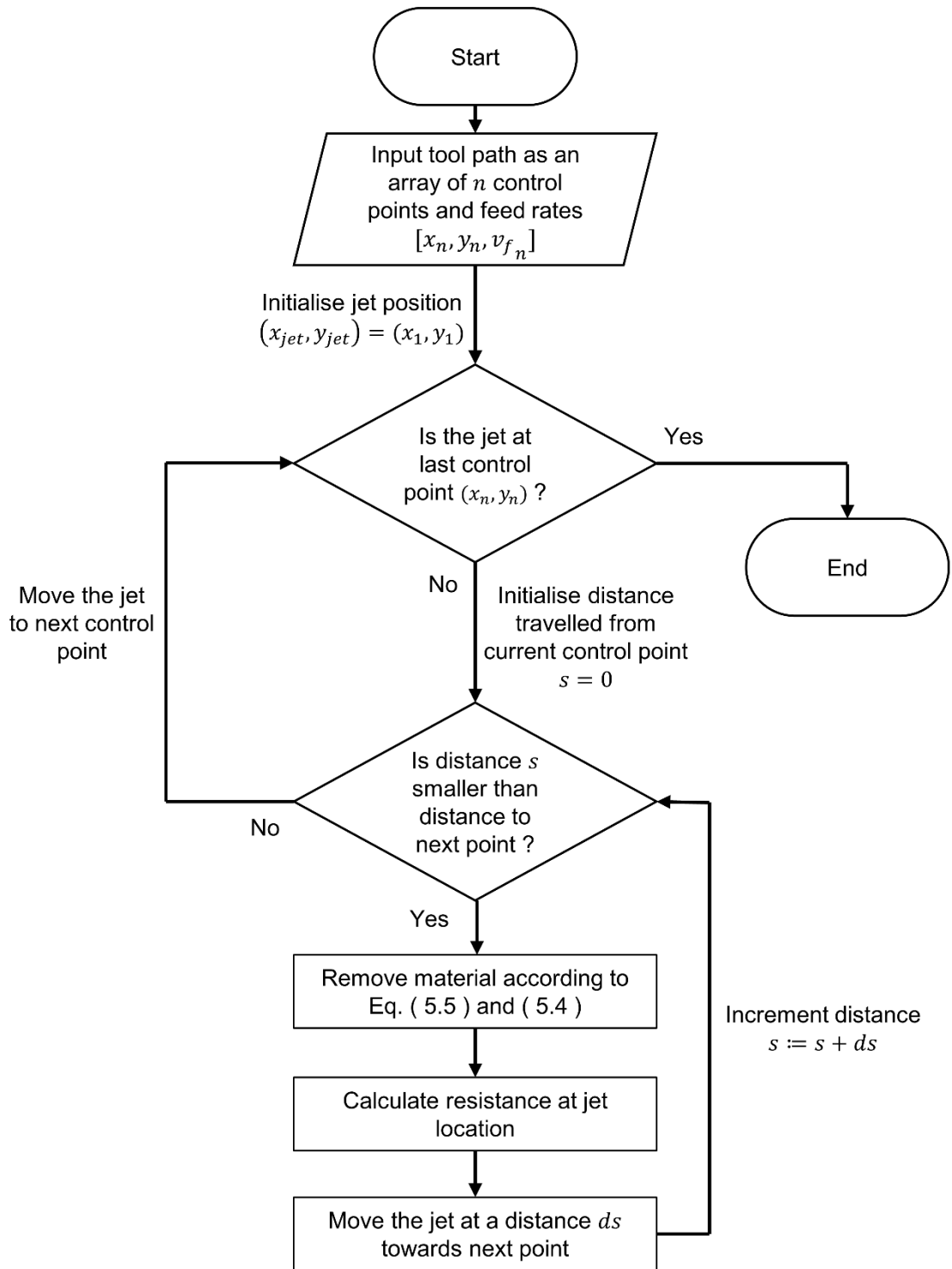


Fig. 5.2. A flow chart visualising the implementation of the developed process model.

5.2.1. Calibration of material removal

To perform the simulation as described in the previous section numerical values are required for the volumetric material removal rate (MRR) and the initial removal rate function $P_0(\mathbf{x})$. The MRR is found experimentally by measuring the volume of pits machined with dwell times 1 s – 11 s at the process conditions specified in Section 3.4.1 of Methodology. The slope of the line of best fit gives the value of 0.0029 mm³/s (see Fig. 5.3) The initial removal rate function $P_0(\mathbf{x})$

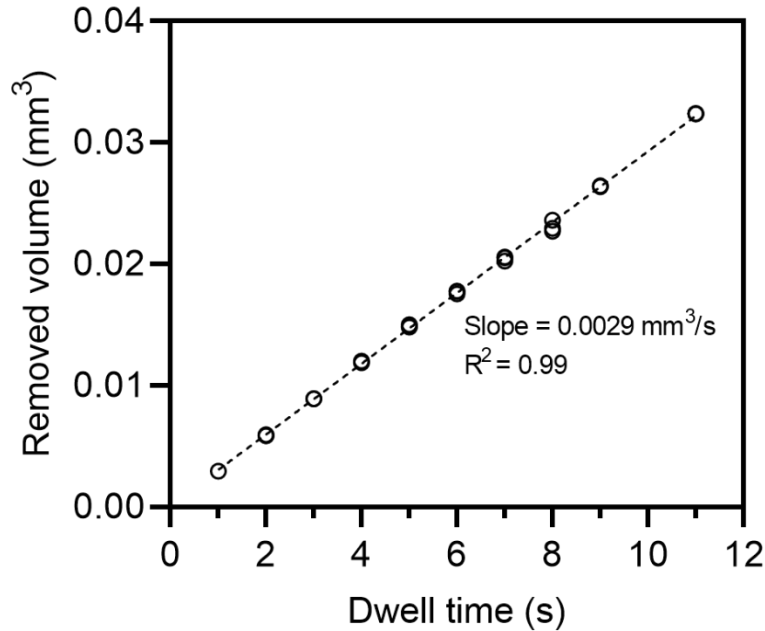


Fig. 5.3. Material removal volume as a function of time in static pit machining for fixed electrical conditions.

can be estimated from the cross-sectional profiles of pits machined at short dwell times. Here, pits machined at 1 s and 2 s were used. In the case of a conventional cylindrical nozzle, the pit shape is approximately axisymmetric, so over the time interval of $\Delta t = 1$ s, from Eq. (5.2) it can be assumed that

$$P_0(r) = - \frac{\Delta z}{\Delta t \sqrt{1 + \left(\frac{\partial z}{\partial r}\right)^2}} \quad (5.6)$$

where r is the radial distance from nozzle centre, Δz is the difference between the two experimental profiles at the two different dwell times (Fig. 5.4b) and the surface gradient $\frac{\partial z}{\partial r}$ is taken as the average of the surface gradients for the two profiles (Fig. 5.4c). A Gaussian filter with a cut-off length of 0.05 mm was applied to the obtained result from the calculation to smooth out the noise caused by the calculation of surface gradient. The result is presented in Fig. 5.4d.

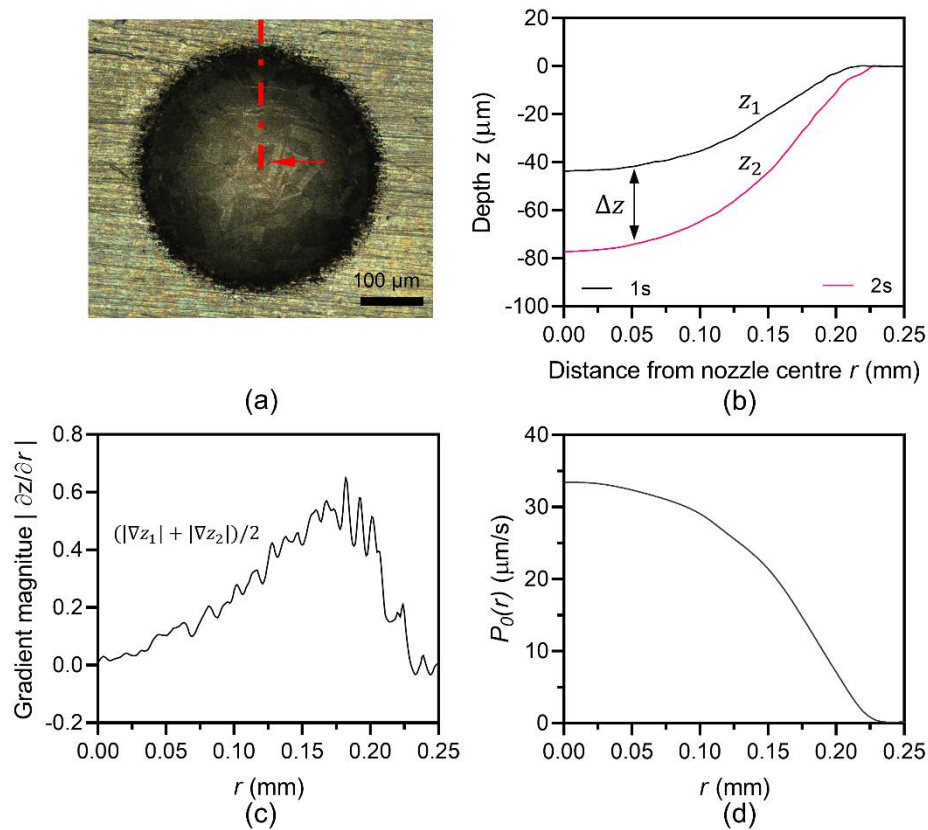


Fig. 5.4. Experimental calibration of the removal rate function $P_0(\mathbf{x}')$. Machining of pits (a) at two different dwell times allows to extract averaged surface profiles (b) and calculation of surface gradient (c) as a function of distance to nozzle centre. The removal rate function (d) is obtained from (5.6).

5.2.2. Resistance and PSU dynamics

To calculate the resistance response from the machining process, Eq. (4.2) was applied to calculate the equivalent distance L_{eq} to the surface at any point of tool path. The local distance to the surface is given as $l(x,y) = L_0 - z(x,y)$, where $L_0 = 0.5$ mm is initial stand-off distance set between the nozzle and the work piece. The resistance at any point of the jet trajectory is then obtained according to Eq. (4.4) by multiplication with the experimentally calibrated sensitivity as described in Section 3.7.1 in Methodology. The value used here was 2.3 k Ω /mm (S.D.= 0.01 k Ω /mm) obtained by linear fitting to the resistance response curve in the range of 0.4 mm – 0.8 mm stand-off distance.

The IEG resistance during machining is inferred from the measured current and voltage. These signals however are influenced by the dynamic characteristics of the power supply and its ability to maintain the set conditions when the load varies. To characterise the device used in this work, the transient behaviour of voltage was investigated when a current step of 50 mA was programmed through a fixed 1 k Ω ($\pm 1\%$) resistor connected across the PSU terminals.

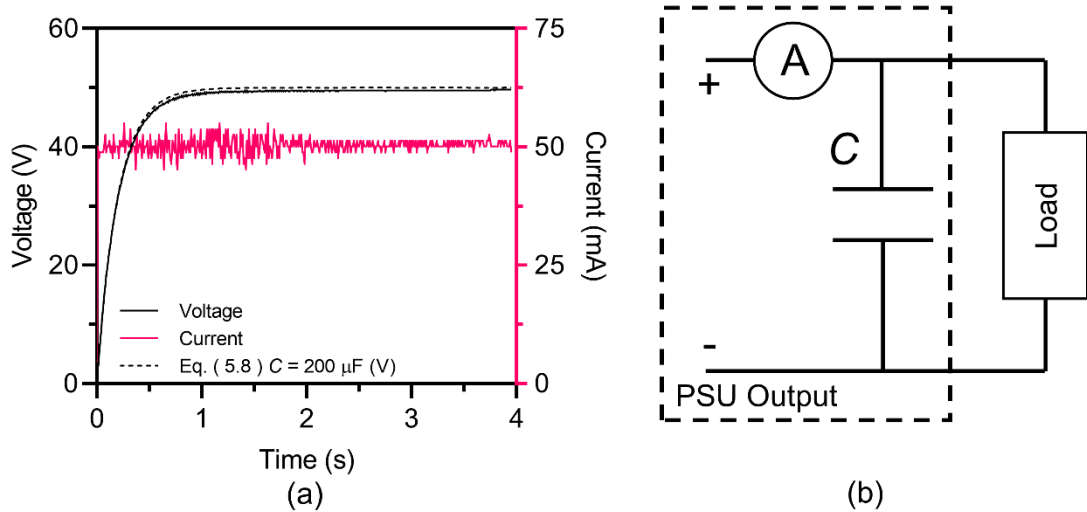


Fig. 5.5. Dynamic behaviour of the PSU used for machining. (a) Measured voltage response to an applied current step through a 1k Ω fixed resistor. (b) Circuit diagram of the PSU output consistent with the observed response.

Voltage and current measured directly by the internal monitoring provided by the PSU are shown in Fig. 5.5a. The observed transient response in voltage is likely caused by the charging of decoupling capacitors at the power supply output, whose purpose is to reduce voltage fluctuations across the load (Fig. 5.5b) The measured current is the total current flowing through the capacitor and the load. During the transient phase, the actual current through the load is lower than the set value. If a total current I is flowing through a capacitance C and a resistive load R in parallel as shown in Fig. 5.5b, the voltage as a function of time $U(t)$ can be obtained by solving

$$C \frac{dU}{dt} + \frac{1}{R} U = I \quad (5.7)$$

where I and R are also in general time-dependent. This equation was solved numerically using the SciPy open-source computing library [135] with an initial condition $U = 0$ V at $t = 0$ s and $R = 1$ k Ω , $C = 200$ μ F under a current step of 50 mA and the result is plotted as the dashed line in Fig. 5.5a demonstrating agreement with the experimentally observed response. These dynamic effects are specific to the experimental configuration used in this work but need to be taken into account to enable comparison between the modelling and the experimentally acquired signals. Thus, after obtaining resistance as a function of time from the process simulation, this is used to solve (5.7) and calculate the final expected voltage during machining.

5.3. The IEG resistance during machining

To validate the developed models the process resistance was investigated experimentally for several machining scenarios – machining of static pits

without jet translation, machining of straight channels and machining of curved channels. The observed response was compared to the one expected from the simulation.

5.3.1. Static machining

The cross-sectional profiles of pits machined for 1 – 11 s are compared against simulated ones in Fig. 5.6a. The developed model exhibited overall agreement with the experimentally observed evolution of the pit shape with root-mean-square error between the profiles ranging from 4.4 μm for a dwell time of 4 s to 16.5 μm for a dwell time of 11 s. Comparing the depths, the largest deviation was observed for a dwell time of 11 s where the model underpredicted the average experimental depth by 24.7% and the smallest relative error was 0.1% for a dwell time of 5 s. The mean absolute percentage error in depth between the simulation and the experiment was 7.2% across all machined pits. The geometry of the machined pits was repeatable with standard deviation of pit depth from 3 replicates within 1.2 μm across all dwell times. The deviations between the model and the experimental data and the fact the depth is underpredicted for short dwell times but is then overpredicted for larger times are most likely due to the model not considering lateral growth of the machined pit. For example, the pit machined for 1 s has a smaller diameter than the ones at longer dwell times. Since the model enforces a constant volumetric removal rate and a constant “spot size”, a reduction in depth is seen. This is illustrated in Fig. 5.7 by plotting the relative error between the simulated and the experimental depth as function of the observed pit diameter. When the experimental diameter was smaller than the simulated one (0.46 mm) , the simulated depth was lower (negative error), while larger experimental

diameters correspond to a positive relative error. This demonstrates that material removal rate is distributed differently at larger depth of cuts compared to the assumed removal rate function.

An exemplar voltage trace acquired during machining for 11 s is shown in Fig. 5.6b. When the PSU is initially switched on, there is a transient effect until the output capacitors are fully charged and a steady voltage is attained. As the depth of cut progresses with time, the voltage increases linearly corresponding to the increased distance between the nozzle and the bottom of the pit. At dwell times larger than 7 s the voltage deviates from the linear trend with sudden drops observed sporadically.

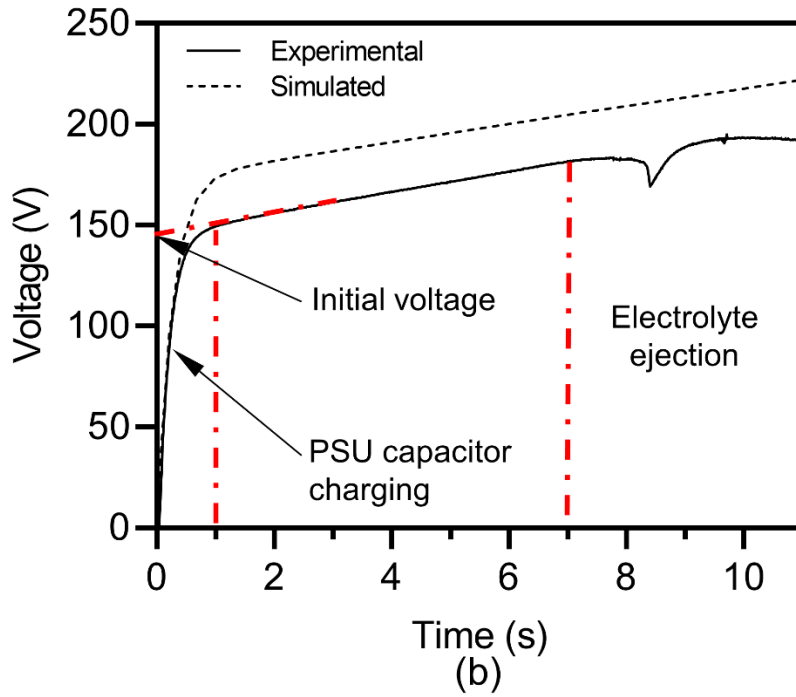
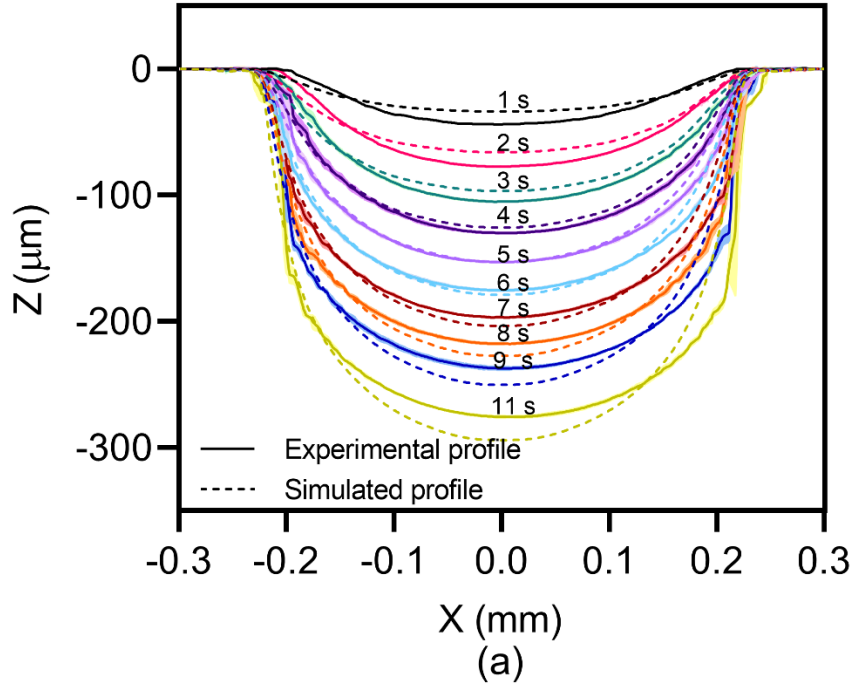


Fig. 5.6. The evolution of pit profile (a) and voltage signal (b) during static machining at a supplied current of 150 mA (average current density 306 A/cm² and for a 0.25 mm nozzle) and a liquid flow rate of 41 ml/min (average jet speed of 14 m/s). Error regions of profiles represent standard deviations from 3 machined pits.

This likely corresponds to the depth of cut at which electrolyte is being ejected from the side walls of the pit as described by Hackert et al. [11] and discussed

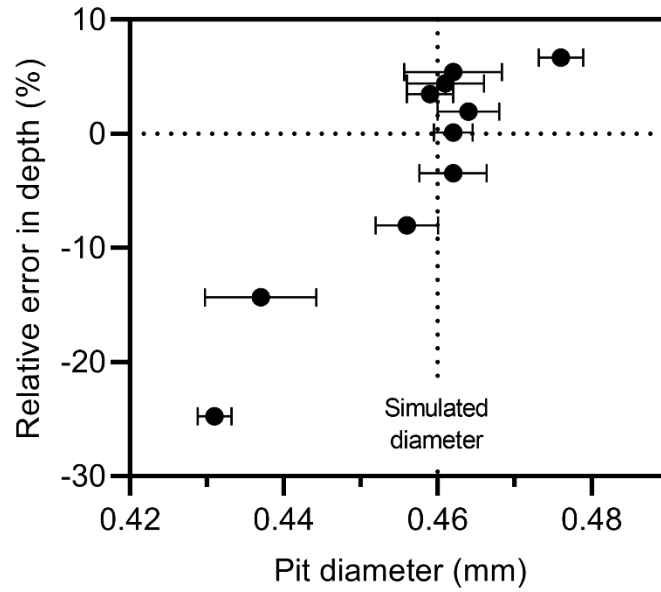


Fig. 5.7. Relative error in depth between the simulated and experimental pits as a function of experimental pit diameter. The simulated pit diameter was constant at 0.46 mm. Larger errors in depth correspond to larger errors in diameter. Error bars correspond to standard deviations across 3 pits machined at identical parameters.

in the literature review. A drop in voltage as seen in Fig. 5.6b could be caused by ejected electrolyte re-contacting the nozzle and thus providing an additional current path resulting in a decrease in resistance. The simulated response exhibited a similar linear behaviour to the experimental one for $t < 7$ s. Some variation in absolute voltage was seen across the preformed pit machining experiments although the overall trace shape remained the same. Due to the capacitive transient, it was not possible to directly measure the “real” voltage corresponding to the resistance of the IEG at the onset of machining. Instead, a least-squares linear fit of voltage against time was performed for $t > 1$ s for each trace. If the machining time was longer than 7 s the upper bound on range was set to $t = 7$ s, to exclude the non-linear region at large depths of cut. The initial voltage was taken as the vertical intercept of the line of best fit. Across all recorded traces this varied in the range 135.5 V – 150.5 V with a mean of 142

V and S.D. = 4.2 V. Considering the calibrated sensitivity of 2.3 k Ω /mm, the range of 15V correlates to a variation in stand-off distance of approximately 43 μ m, so this could be attributed to the flatness of the work piece. The initial voltage predicted by the model is 172.5 V corresponding to a resistance of 1.15 k Ω . The model does not consider that the shape of the jet changes upon impingement resulting in a wider diameter near the work piece surface and consequently a reduced resistance, so it could be expected that the model would always overpredict the voltage in terms of absolute value. Another limitation of the model is that it does not consider the temperature increase of the electrolyte due to Joule heating as it flows through the IEG which according to Eq. (3.4) is maximum 9 °C for the machining parameters chosen. This may result in a maximum 18 % local increase in the electrolyte conductivity (assuming 0.02 1/K temperature coefficient), although this is non uniform along the length of the jet. It is more useful to consider changes in voltage relative to the value at the start of machining. The average change in voltage relative the defined initial value from all machined pits along with the simulated response is presented in Fig. 5.8a. It can be seen than in the linear regime

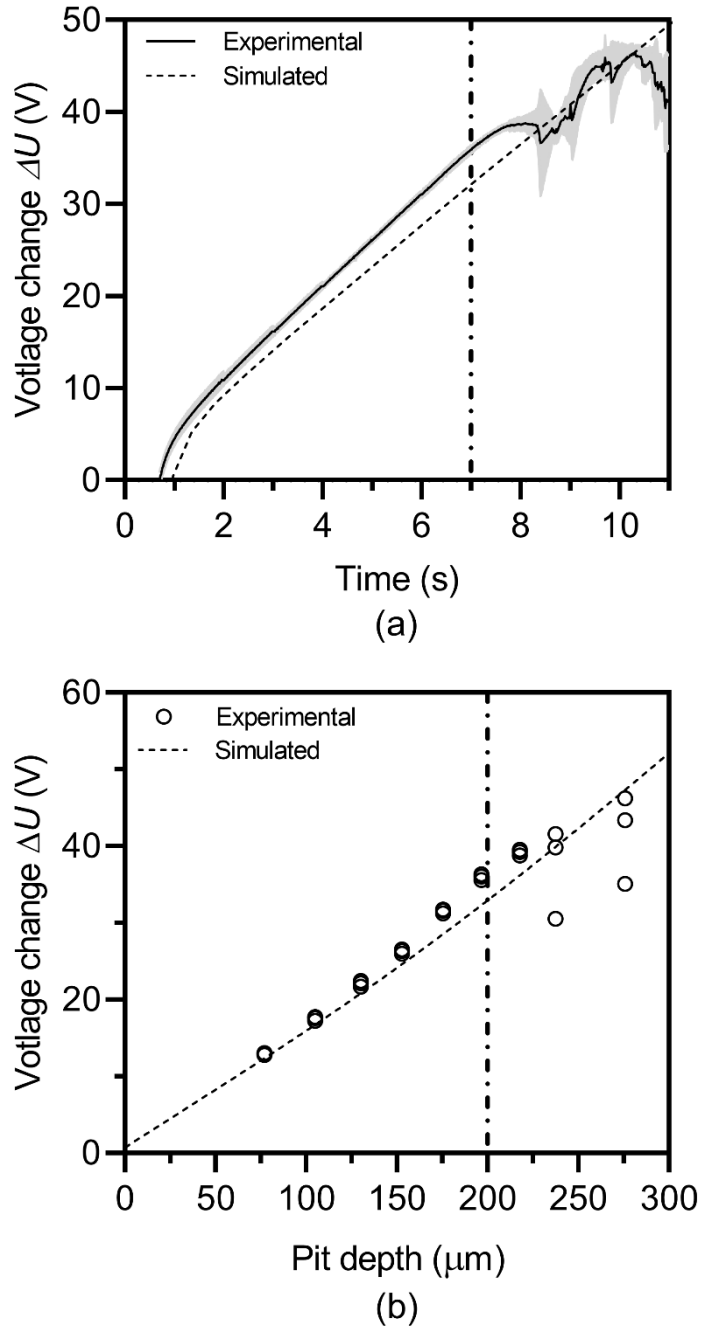


Fig. 5.8. Experimental and simulated voltage change during machining of pits as a function of (a) time, (b) depth of cut. The error regions on a. represent the standard deviation from all acquired voltage traces. The vertical line in both graphs represents the location where electrolyte ejection from the machined pit disturbs the electrical response.

between 1 s and 7 s the voltage change was repeatable with maximum standard deviation across traces of 0.8 V. For in-process monitoring of the depth of cut the observed change in voltage needs to be correlated to the

machined pit depth (Fig. 5.8b). The predicted change in voltage as a function of depth of cut agreed with the experimentally obtained data – considering the pits obtained at dwell times from 2 s to 7 s the experimental average sensitivity to change in depth of cut calculated by least-squares fitting is 192.1 V/mm (corresponding to 1.28 k Ω /mm) and the predicted average sensitivity over the same range of simulated depths is 167.2 V/mm (1.11 k Ω /mm). Both values are significantly lower than the sensitivity obtained through the calibration experiment in Section 5.2.2 (2.30 k Ω /mm) demonstrating that the non-flat bottom of the machined pit has a large influence on the resistance response and confirms the need for process modelling to interpret the acquired signal. The regime transition, where the large depth of cut and the of the machined geometry with the jet disturbs the electrical response, is identifiable in Fig. 5.8. This occurs at pit depth of approximately 200 μ m for the 0.25 mm diameter nozzle used here. This corresponds to a feature aspect ratio (defined as ratio between depth and width) of approximately 0.5. which agrees with the simulations of Hackert et al. [11]. This represents the geometric constraint within which the developed modelling approach can be applied to the machining process. The percentage error in depth between the experiment and the corresponding depth expected from the simulated voltage-depth relationship is shown in Fig. 5.9 for each experimentally observed voltage change within 2 – 7s. The result demonstrates that using the model, the depth of cut of pits can

be measured in-process with an error $<10\%$ (corresponding to $20\text{ }\mu\text{m}$ absolute for a dwell time of 7 s).

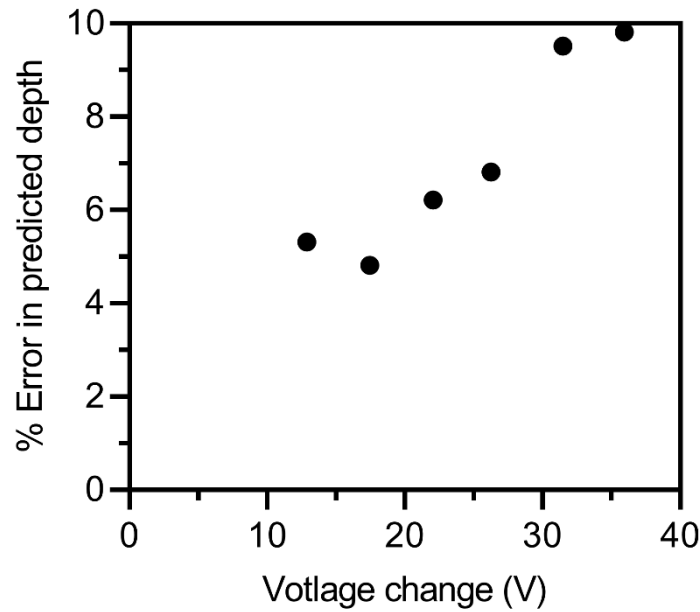


Fig. 5.9. Percentage error between simulated and experimental depth as function of the observed voltage change

5.3.2. Machining of straight channels

Machining of straight channels was undertaken with the feed rate programmed to vary between 1 mm/s and 0.075 mm/s as a function of jet position as described in Section 3.4.1 of Methodology. This resulted in channels with linearly increasing depth towards the centre as shown in Fig. 5.10a-b. The machining experiment was repeated 3 times with a maximum standard deviation of $2.8\text{ }\mu\text{m}$ between the measured depth profiles along the length of the channels justifying averaging of the profiles and the acquired voltage signals. At the shallowest section the average depth was $11.9\text{ }\mu\text{m}$ (S.D. = $0.4\text{ }\mu\text{m}$) and this increased to a maximum of $124\text{ }\mu\text{m}$ (S.D. = $2.8\text{ }\mu\text{m}$) upon reduction of feed rate. The accuracy of depth prediction by the model reduced with

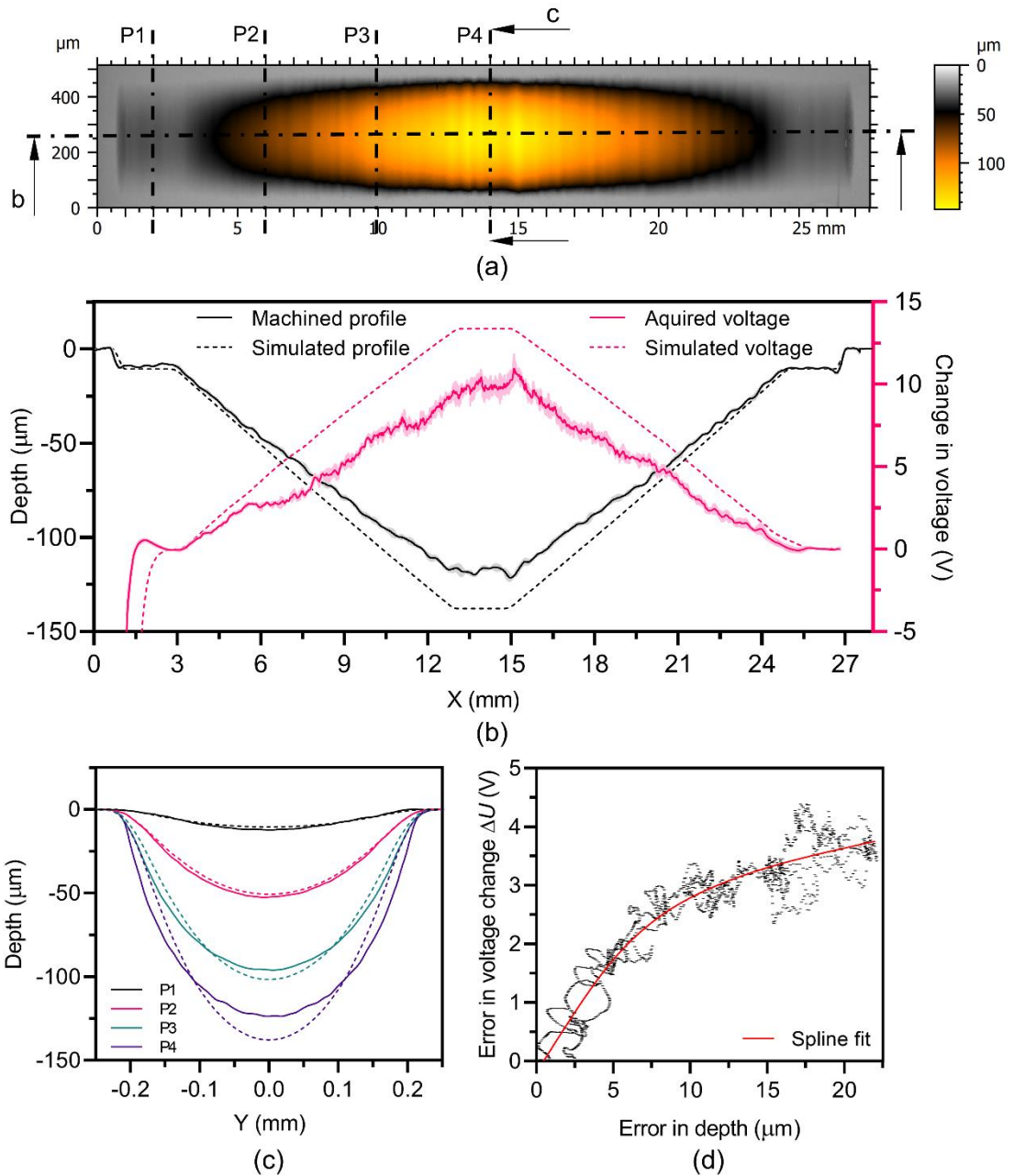


Fig. 5.10. Resistance during machining along straight tool paths. (a) Height map of channel machined with dynamic feed rate variation. (b) Depth profile along the channel length and recorded voltage during machining compared to the simulation. The experimental profiles represent averages from 3 repeated experiments with error regions corresponding to the standard deviation. A Gaussian filter with a cut-off of 0.25 mm was applied to the topography profiles. (c) Exemplar cross-sectional profiles from the machined channels at the locations specified in a. filtered with a Gaussian filter with 0.025 mm cut-off. Dashed lines represent simulated profiles. (d) Errors in voltage change and machine depth compared to the model.

increasing cut depth – the predicted depth at the shallowest section was 10.6 μm and in the deepest section this was 137.8 μm . Similar to the pit machining

case, the model fails to describe the widening of the channel (Fig. 5.10c), which is caused by hydrodynamic effects and alteration of the current density distribution. The mean absolute percentage error between the model profile and the experimental result in Fig. 5.10b was 12.2%. The voltage change during machining is considered relative to the voltage recorded at the constant depth sections at the start and end of each channel. The measured voltage increased on average by 9.8 V (S.D. = 0.5V, taken as the average for $X > 13.0$ mm and $X < 15.0$ in Fig. 5.10b), while the model predicted a voltage change of 13.4 V. The difference between model and measured voltage increase however may have significant contribution from the error in channel geometry. The voltage and geometry profiles in Fig. 5.10b were linearly interpolated at common X sampling points with 10 μ m spacing and the error between experimental and simulated values at each sample point is presented in Fig. 5.10d demonstrating an increasing trend in voltage error with respect to error in depth against the model. Furthermore, machining artefacts such as the depth increase at approximately $X = 15$ mm (likely related to the response of the positioning system to the requested change in acceleration) are reflected in the voltage trace with a corresponding peak in the signal. These results prove the feasibility of using the resistance during machining for live process monitoring, although for the implementation of corrective control loops, refinement of the signal acquisition system and improvement in noise immunity may be required. The conducted experiment provides an example of a possible application for a resistance-based control system where deviations of the machined profile from the designed geometry, in this case represented by the geometric simulation, are inferred from the difference between the measured voltage and an expected

reference signal and corrective action is taken by modification of process parameters such as supplied current or feed rate. It should be noted that simulation of machined geometry still requires a significant amount of time (≈ 10 min for the channel in Fig. 5.10), so the model is not suitable for running in real-time on the machine controller. Rather, it is intended that this can be applied in advance of machining to generate the expected geometry and resistance. Clearly, the application of the described approach to actual engineering components would require accurate knowledge of the surface geometry prior to machining to obtain the predicted reference resistance. This can be realised through pre-process metrology with the method developed in Chapter 4.

5.3.3. Curved tool paths

Machining along curved trajectories distorts the kerf in EJM [38] due to the differential in exposure time to the jet experienced by points on the work piece surface located on the inside and the outside of the curved path. This results in a distinct charge density distribution (charge passed per unit area) compared to machining along straight tool paths, leading to an increase in material removal towards the centre of rotation. This effect also applies to any energy beam process. In the conducted experiments, nozzle trajectories containing a 90° fillet corner were applied with different fillet radii (Fig. 5.11a). The depth increase became more significant at tool path radii of 0.25 mm and 0.125 mm for the 0.25 mm internal diameter nozzle (Fig. 5.11b-c) – the depth measured approximately at the mid-point of the arc was $56.0\text{ }\mu\text{m}$ and $58.7\text{ }\mu\text{m}$ for the 0.25 mm and 0.125 mm compared to an average depth of $52.8\text{ }\mu\text{m}$ (S.D. = $0.9\text{ }\mu\text{m}$) measured from the straight sections and the cross-sectional profiles exhibited evident asymmetry. For the 0.5 mm radius tool path the effects on the cut profile

appeared negligible with a maximum measured depth of 54.6 μm . The process model was shown to exhibit agreement with the experimental profiles (see Fig. 5.11c) with maximum depths at the corner of 53.8 μm , 56.9 μm and 61.9 μm for the 0.5 mm, 0.25 mm and 0.125 mm radius respectively compared to a depth of 52.9 μm predicted for the straight section. The relative errors compared to the experimental results were -1.5%, 1.6%, 3.2% for the 0.5 mm, 0.25 mm and 0.125 mm radius respectively. Considering the change in voltage during machining (Fig. 5.11a), the change in geometry proved to be detectable with the voltage increasing by approximately 1 V for the 0.125 mm radius path relative to the value at the straight section as the jet traversed through the corner. Monitoring the voltage during machining could allow the detection of depth increase in curved tool paths and allow for a partial compensation by adjusting the supplied current or feed rate to reduce the machine depth. Alternatively, the fact that the process model captures the effects associated with curve tool path allows to pre-plan a different parameter set for regions of the tool path where the cut quality is impaired.

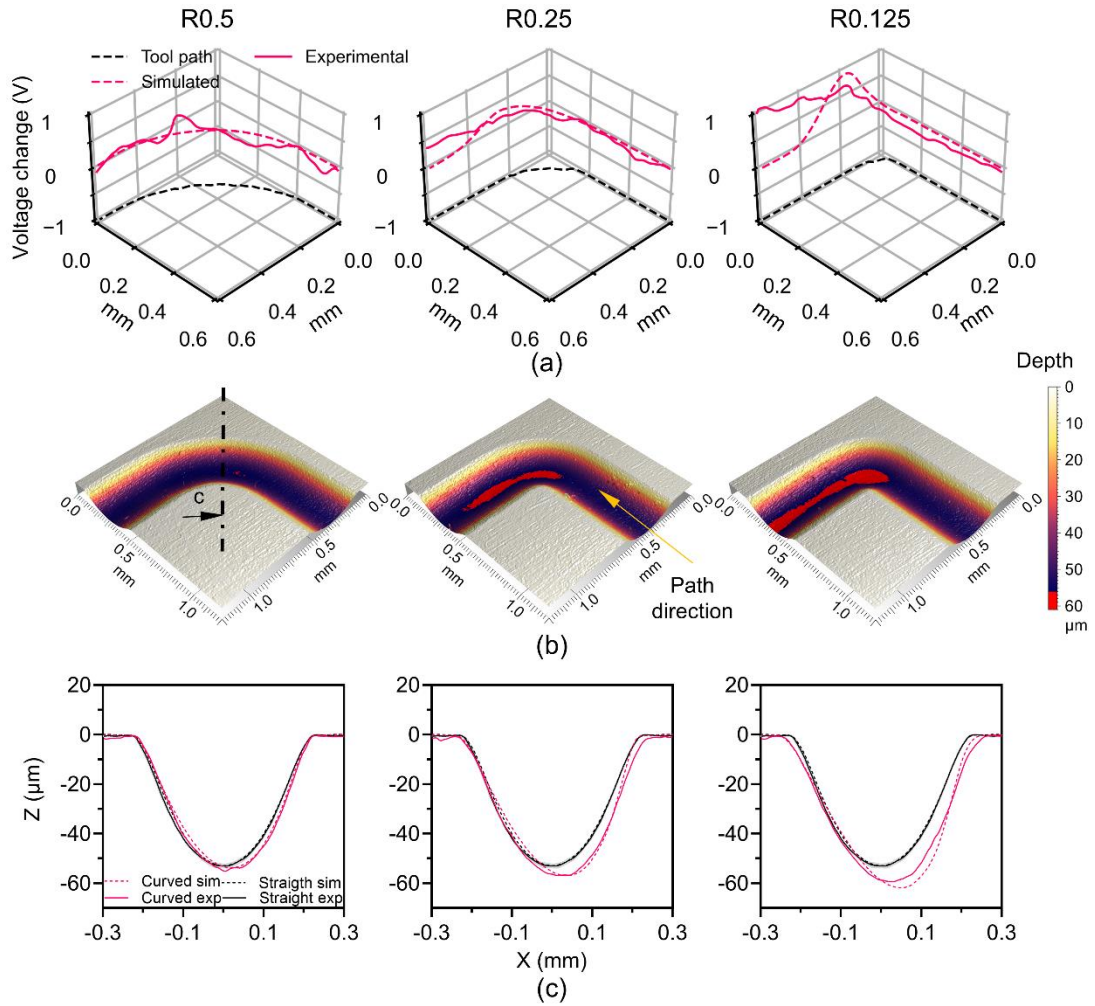


Fig. 5.11. Implications of machining along curved tool paths. (a) Simulated and experimental voltage response to circular arc tool paths with radii of 0.5, 0.25 and 0.125 mm (b) 3-dimensional surface reconstructions of machined curved channels. (c) Cross-sectional profiles extracted along the bisector of the corner compared to an average profile from straight sections. A Gaussian filter of 0.025 mm cut-off was applied to the experimental profiles.

5.4. Automatic stand-off control for 3-dimensional surfaces

A different application for resistance monitoring in EJM is the automated tracking of 3-dimensional component geometry during surface texturing. The operating stand-off distance in EJM is usually sub-millimetre to minimise energy losses, so if the component geometry is not known accurately, automated contour following could prevent collisions and ensure stable conditions in the IEG while dispensing with pre-process measurement. Similar approaches are

in use in laser welding where seam tracking is used to enable beam trajectory compensation [136].

5.4.1. Implementation of closed-loop control

Surface tracking can be achieved by implementing a proportional-integral (PI) controller to adjust the Z-position of the nozzle to maintain a constant resistance and respectively a constant stand-off distance. A block diagram representing the proposed controlled system is presented in Fig. 5.12. The set point resistance R_{set} is detected at the start of the machining process. The actual gap resistance is sampled continuously and controlling action that adjusts the IEG width is executed based on the error $e(t) = R_{set} - R_{actual}$, which is the difference between the setpoint and the measured resistance value. The

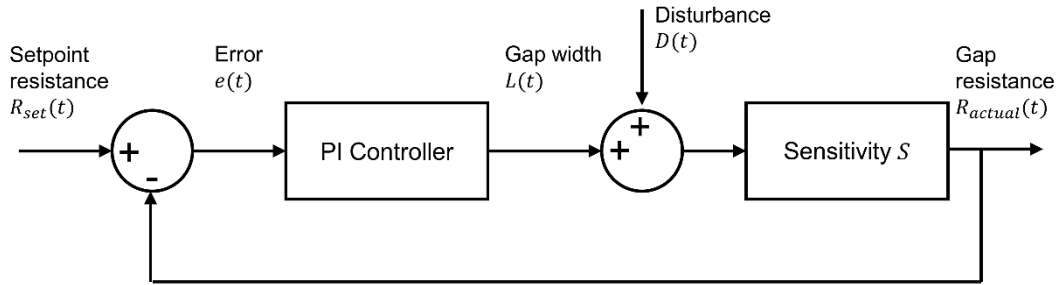


Fig. 5.12. Block diagram of the proposed surface tracking system based on the IEG resistance

disturbance to the process $D(t)$ represents the shape of the work piece surface along the tool path.

A PI controller is implemented in order to maintain the required gap width. The PI controller equation in the time-domain is:

$$L(t) = L(t_0) + K_p e(t) + K_i \int_{t_0}^t e(\tau) d\tau \quad (5.8)$$

In the above $L(t_0)$ is the initial gap width at the starting time t_0 , K_p is the proportional gain and K_i is the integral gain. In order to implement this in a digital system, the above equation is discretised over a sampling interval t_s . At a time instant $t_k = kt_s$, $L(t_k)$ is:

$$L(t_k) = L(t_0) + K_p e(t_k) + K_i t_s \sum_{j=1}^k e(t_j) \quad (5.9)$$

At time $t_{k-1} = (k-1)t_s$:

$$L(t_{k-1}) = L(t_0) + K_p e(t_{k-1}) + K_i t_s \sum_{j=1}^{k-1} e(t_j) \quad (5.10)$$

Subtracting the two equations above results in:

$$\Delta L(t_k) = L(t_k) - L(t_{k-1}) = K_p [e(t_k) - e(t_{k-1})] + K_i t_s e(t_k) \quad (5.11)$$

Eq. (5.11) is the most convenient form for implementation as it directly gives the incremental move on the Z-axis $\Delta z(t_k) = \Delta L(t_k)$ at every control loop cycle. An upper limit was placed on the magnitude of the incremental move so that it is executed with one loop cycle. This minimises latency and prevents the large movements which could cause axis collisions. The maximum speed of the Z-axis is set to $v_z = 50$ mm/s and the sampling time of the control loop was measured to be $t_s = 0.003$ s, so the limit for Δz is $\Delta z_{max} = v_z t_s = 150$ μ m.

For modelling and tuning the control system the resistance change over one cycle was assumed to be $\Delta R(t_k) = S \Delta L(t_k)$, thus neglecting the effects of surface geometry within the jet impingement region. For the sensitivity the theoretical value $S = \frac{1}{\sigma A_{noz}}$ based on electrolyte conductivity and nozzle area

was assumed, to allow scaling of the controller gains K_p and K_i for different conditions according to:

$$\begin{aligned} K_p &= \sigma A_{noz} K'_p \\ K_i &= \sigma A_{noz} K'_i \end{aligned} \quad (5.12)$$

where K'_p and K'_i are the gains tuned for unit conductivity and unit nozzle area. These values are tuned for a 10% - 90% rise time of 0.7 s by simulating the digital control system and in Simulink. Shorter response times resulted in an oscillatory behaviour when implemented in the EJM apparatus likely caused by time delays in the signal acquisition or the dynamics of the positioning system which are not taken into consideration in the system model used for tuning.

5.4.2. Experimental validation

To demonstrate the proposed control system, machining was performed on a three-dimensional free-form surface. The work piece was 30 mm long in the X-direction and the difference between the highest and lowest point is approximately 2.35 mm. Four channels were machined with number of consecutive passes over each channel increasing from 1 to 4. The surface geometry along the machining toolpath acquired through FVM is shown in Fig. 5.13a. The extracted cross-sectional profiles along the machined channels (Fig. 5.13b-e) demonstrate that the depth and kerf width remained consistent

along the tool path. The average depths of the extracted profiles across the 5 locations annotated in Fig. 5.13a were 20.2 μm , 39.7 μm , 55.6 μm and 72.3 μm for the for the tracks machined with 1, 2, 3 and 4 passes respectively, and the

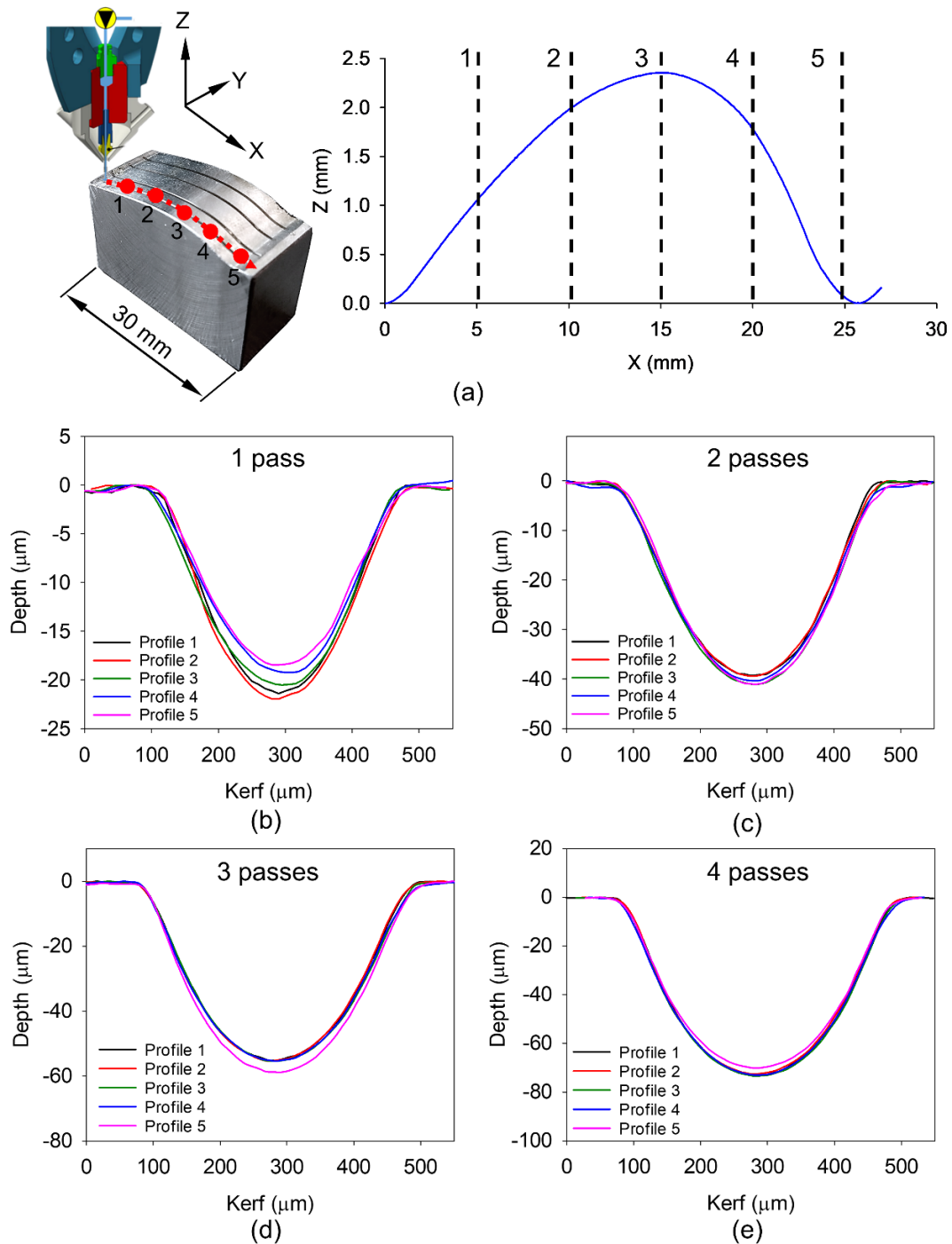


Fig. 5.13. Machining with automated contour tracking. (a) Work piece surface geometry. (b) – (d) cross-sectional profiles extracted from the channels machined with 1 – 4 passes at the locations specified in a. Gaussian filter of 0.025 mm cut-off was applied to all profiles.

standard deviations on these values were 1.3 μm , 0.9 μm , 1.2 μm and 1.1 μm respectively. This suggests that the variance in the profiles is independent of depth of cut and consistent machining performance is maintained with closed-loop stand-off distance control.

The controller response during machining of the single pass channel is compared to the response predicted by the control system simulation in Fig. 5.14. For the Simulink model, the surface profile of the work piece (Fig. 5.13a) is converted to a function of time to obtain the disturbance $D(t)$ to the control system (see Fig. 5.12) If the surface profile is given by a function of the X-coordinate $z(x)$, then the disturbance as the jet traverses over the surface with a horizontal feed rate v_x is $D(t) = -z(v_x t)$

As seen in Fig. 5.14 the behaviour of the control system was similar to the prediction of the simulation. The total range of motion of the Z-axis (see Fig. 5.14a) was 2.35 mm for both the experiment and the simulation and this matches the height of the work piece profile. However, the experimental response appeared to be shifted below the simulated one, indicating that the controller forced the Z-axis to operate closer to the work piece surface than expected with a mean error of -0.07 mm (Fig. 5.14b). The largest discrepancy between the experiment and simulation was -0.19 mm. This difference in response may be due to the non-linearity of jet resistance with stand-off as discussed in Section 4.3.2 of Chapter 4, which is not considered by the model or compensated for in the control system. This may cause the controller to operate with an error relative to the set point value. The difference in Z-axis

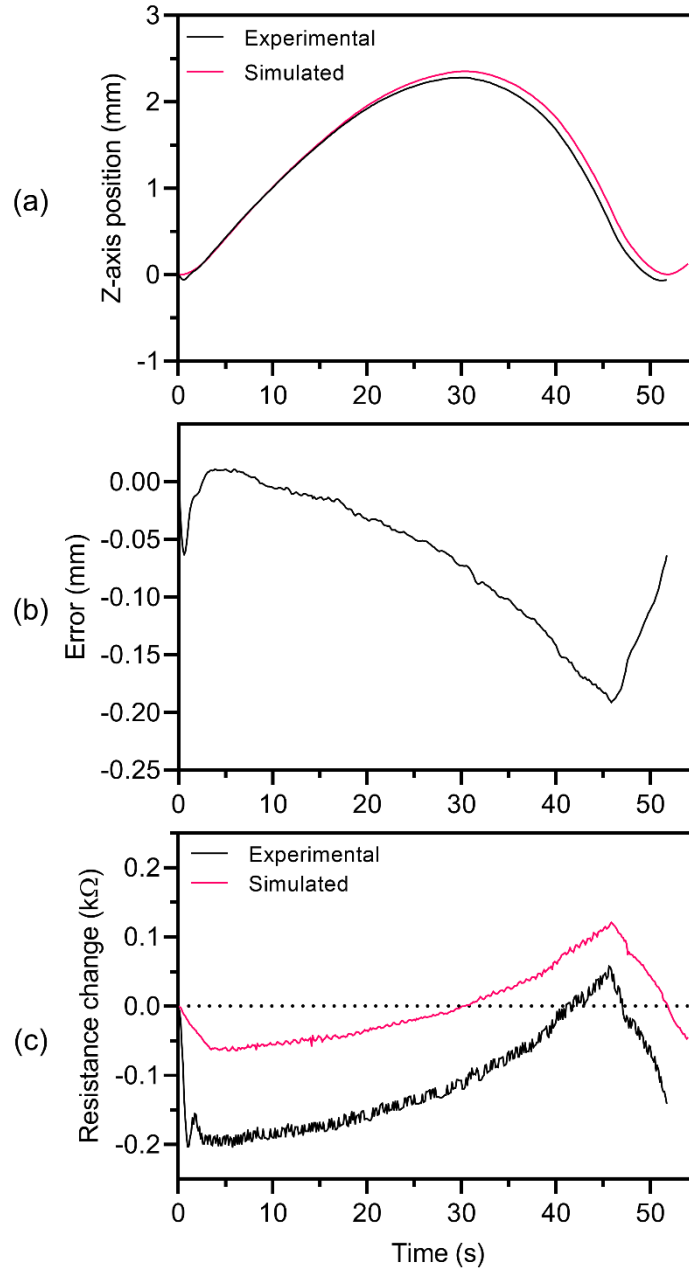


Fig. 5.14. Stand-off controller response to a 3-dimensional surface compared to simulation. (a) Position of the Z-axis. (b) Error between the experimental and simulated Z-axis position. (c) Resistance change during machining over the 3-dimensional surface

response was also reflected in the resistance change during machining (Fig. 5.14c) with the experimental resistance having more negative values than the simulated. The overall variation of resistance during the traverse over the surface was 0.26 kΩ while the simulation predicted 0.19 kΩ. Latencies in the

feedback and uncertainty in the sensitivity of the system could be contributing factors to this difference in response.

A further consideration when using the developed automatic stand-off control for machining on 3-dimensional surfaces is that a constant feed rate v_x is programmed in the X-direction. However, the tangential velocity component v_t parallel to the work piece is what should determine the material removal and depth of cut since it is inversely proportional to the exposure time to the jet. In the case of the conducted form tracking experiment, the Z-position is adjusted at an effective velocity v_z and the tangential velocity to the surface is given by

$$v_t = v_x \cos \theta + v_z \sin \theta \quad (5.13)$$

where θ is the slope of the surface. If the control system succeeded in tracking the surface perfectly i.e. the change in position of the Z –axis exactly matched the surface contour $v_z = \frac{dz}{dt} = \frac{dz}{dx} \frac{dx}{dt} = v_x \tan \theta$ leading to

$$v_t = \frac{v_x}{\cos \theta} \quad (5.14)$$

This suggests that the tangential velocity varies across the surface. This may explain the variation in depth seen in the single-pass machining experiment (Fig. 5.13b). However, the variation in depth is reduced when considered relative to the depth of cut with increasing number of passes. Another factor that is likely to influence the machined profiles is the re-distribution of current density in the jet due to the changing angle of incidence [20]. These effects need to be considered when choosing the appropriate strategy applying EJM to actual engineering components dependent on the required feature fidelity. For large curvatures a multi-axis or a robotic arrangement is required that maintains the nozzle normal to the surface and the feed rate is controlled

parallel to the surface by the motion control. The application of the closed-loop stand-off control in such scenario would be to compensate for errors in path planning and actual part geometry.

The presented closed-loop surface tracking approach can also be applied to the measurement mode of the electrolyte jet apparatus discussed in Chapter 4 for measurement of surfaces with large surface height variation. The surface geometry can be inferred from the recorded Z-axis position and the resistance change, thus allowing for extended measurement range.

The closed-loop stand-off control during machining is applicable to situations where the depth of cut is not expected to vary significantly, since both depth-of-cut monitoring discussed in Section 5.3 and surface form tracking rely on measurement of the same signal, but the compensating controlling actions differ dependent on the origin of the disturbance to the resistance. Hence, variation of depth of cut would cause the proposed PID controller to advance or retract the nozzle. If the depth of cut changes by an amount similar or larger to the stand-off distance to the top surface, this could cause the nozzle to collide with or come in close proximity to the work piece increasing the risk of arcing with side edges of machined features. The simultaneous application of surface form tracking and depth of cut monitoring may be realised by using a separate “measurement jet” and “machining jet” with different electric excitations within the same end effector.

5.5. Summary

In this chapter, the resistance response of the IEG in EJM was investigated during surface processing. To understand the relationship between the

resistance signal and the evolving surface geometry, a 3-dimensional model was developed to simulate EJM machining operations based on initial calibration of material removal rate through simple pit machining experiments. The model allows prediction of the resulting surface geometry and corresponding resistance response for a specified tool path. The simulation was validated against machining experiments including static machining of pits, and machining along straight and curved toolpaths with varying depth of cut. The resistance during the process proved to be predictable when the aspect ratio of machined features was smaller than 0.5 resulting in limited influence of the hydrodynamics of the impinging jet. Through the proposed mathematical models of surface evolution and in-process resistance, depth of cut could be estimated by the monitoring the voltage during machining at constant current. In-process measurement of depth of cut during pit machining based on the model was shown to be within 5% of optical measurements for depth of cut $<130\text{ }\mu\text{m}$ (≈ 0.28 aspect ratio) with the error increasing to 10% for depth of cut of $197\text{ }\mu\text{m}$ (≈ 0.43 aspect ratio). The control of the machined features within these tolerances is sufficient for typical applications of EJM such as the fabrication of lubricant-retaining features. The results also demonstrate that the detection of machining errors relative to the design or disturbances to the expected removal rate, for example caused by a different material composition or microstructure, is feasible through in-process resistance monitoring. This is expected to lead to further development of advanced machining strategies where machining performance can be evaluated online and process parameters can be adapted dependent on the acquired electric signals.

In addition, the application of resistance monitoring to the implementation of an automated surface tracking method was demonstrated. This enables surface texturing of engineering components with complex surface shapes when the exact geometry or orientation after fixturing is unknown. This reduces the requirements for pre-process metrology and accurate path planning for industrial applications.

6. Adapting resolution through flow focusing

6.1. Introduction

The literature review identified that the spatial resolution achievable for localised material processing is one of the key limitations of electrochemical jet methods. Generally, this is defined by the nozzle size and the resulting jet diameter and constrains both the machining mode and the measurement mode of the apparatus as discussed in Chapter 4. Although the use of nozzles smaller than 50 μm internal diameter has been reported [44], this could pose challenges to the design of the electrolyte filtration system to prevent clogging and maintain reliable operation. Improvement of machining resolution for a fixed nozzle size has been shown through the modification of nozzle profile and electrolyte chemistry [83], resulting in machined kerf of the order of the nozzle size.

A method capable of producing micrometric liquid jets and droplets without the need for small nozzles was proposed by Gañán-Calvo [2] and explored in detail in fundamental bench top fluid dynamics experiments. Known as ‘flow focusing’, this approach entails positioning the tip of the nozzle generating the jet upstream of an orifice and applying a co-flowing gas stream, so that both the liquid jet and the gas flow through the orifice. At sufficiently low liquid flow rates, the pressure of the gas stream stabilises a liquid meniscus at the nozzle tip from which a jet much smaller than the orifice size is emitted (Fig. 6.1). For liquids with sufficiently low viscosity (as high as 0.1 Pa.s for typical applied gas pressure differentials across the orifice $\Delta p_g > 100$ mbar [2]), viscous stresses and surface tension stresses are negligible relative to the pressure differential

Δp_g and the focused jet diameter d_{jet} is only dependent on Δp_g , the liquid flow rate Q and the liquid density ρ_l :

$$d_{jet} = \left(\frac{8\rho_l}{\pi^2 \Delta p_g} \right)^{\frac{1}{4}} Q^{\frac{1}{2}} \quad (6.1)$$

The geometric parameters of the system, namely nozzle internal diameter d_{noz} , orifice diameter d_o and nozzle tip-to-orifice distance H (see Fig. 6.1) were shown to define the stability of the meniscus [137], the minimum achievable flow rate and consequently the minimum jet diameter for which continuous jetting is possible before the onset of the so called ‘dripping’ instability. Different embodiments of the flow focusing principle utilising flame-shaped glass capillary tubes [138], micro-injection moulded nozzles [139] and more recently additively manufactured nozzles [140] to guide the focusing gas stream have been reported and jet diameters of the order of 300 nm have been achieved [141]. In addition to the standard gas-liquid flow focusing, a multi-stage focusing arrangement [142] consisting of a gas stream focusing a viscous sheath liquid which in turn focuses a core liquid stream, has been developed with the objective of improving stability and reducing the minimum jet diameter. A fundamental modification of the flow focusing principle resulted from the use of a sharp hypodermic needle to inject the focused liquid into the focusing fluid stream with the needle tip positioned along the centre line of the focusing orifice [143] – this produced a flow similar to the viscous flow in the gap between two moving surfaces (Couette flow) leading to ejection of a liquid ligament from the sharp tip. Lower stable flow rates than in the standard configuration were reported achieving submicrometric jets emanating from a 160 μm internal diameter needle. Currently, the key application of flow focusing methods is

sample delivery for serial femtosecond X-ray crystallography [144, 145] for structure determination of biochemical molecules where the destruction of the crystal during measurement necessitates continuous replenishment of the sample. Other proposed applications areas include polymer fibre spinning [146], fabrication of microparticles for drug delivery [147] and deposition of polymer films [148]. The compressed air-film encircling jet electrodeposition reported by Xinchao et al. [10] is effectively an application of the flow focusing principle to electrochemical jet deposition. The addition of a focusing orifice downstream of the jet resulted in reduction of a copper deposit diameter of approximately 23% from 1.32 mm to 1.01 mm, produced with a 1 mm nozzle electrode.

This chapter reports the development of a novel end effector for electrochemical jet methods based on the flow focusing principle. It is proposed that through the application of this flow focusing configuration the resolution of electrochemical jet techniques can be decoupled from nozzle diameter, allowing improvement of spatial resolution. The fact that the jet diameter is controlled by the flow rate and the pressure drop across the focusing orifice (Eq. (6.1)) enables continuously variable tool/probe size without the need for a physical tool change, leading to a significant enhancement of process agility. The application of the developed end effector to both the machining and measurement mode of the electrochemical jet apparatus is investigated.

6.2. Development of flow focusing tooling for EJM

The design of the flow focusing end effector was based on the experimental results of Vega et al. [137], who investigated the stability of this configuration

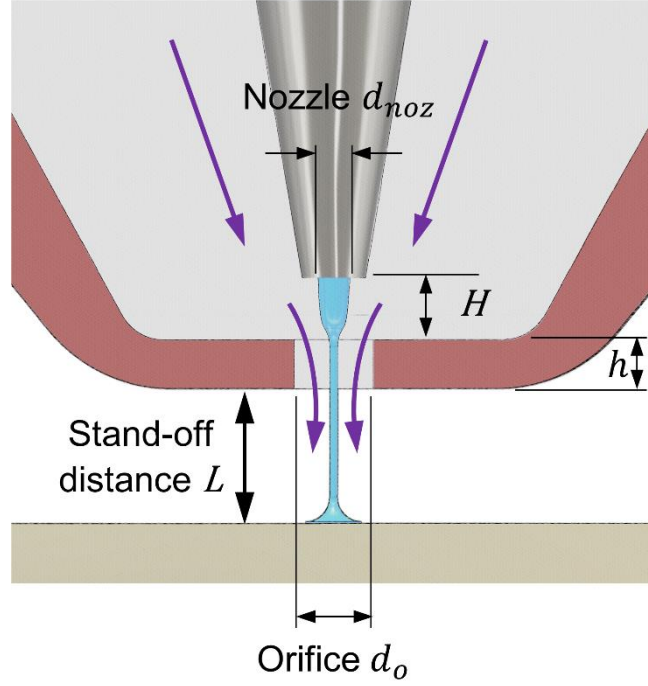


Fig. 6.1. The flow focusing configuration with key geometric parameters.

dependent on the geometric parameters H , d_o and d_{noz} (see Fig. 6.1). The study found that a minimum flow rate Q_{min} exists, which is dependent on geometry, but almost independent of the applied pressure drop, below which the formation of stable meniscus was not possible. The following correlations were derived experimentally in [137] between Q_{min} and the geometric parameters:

$$Q_{min} \approx 2.5 \left(\frac{d_{noz}}{d_o} \right)^{\frac{1}{3}} \frac{d_o \mu}{\rho_l} \quad (6.2)$$

$$\frac{H_{opt}}{d_o} \approx 0.6 \left(\frac{d_{noz}}{d_o} \right)^{\frac{1}{4}} \quad (6.3)$$

In the above H_{opt} is the optimum tip-to-orifice distance at which Q_{min} was observed dependent on d_{noz} and d_o . It should be noted that the stability of the meniscus does not guarantee a stable jet downstream of the orifice. The calculated values for H_{opt} and the minimum achievable jet diameter $d_{jet,min}$ corresponding to Q_{min} at a pressure differential of $\Delta p_g = 150$ mbar for nozzle

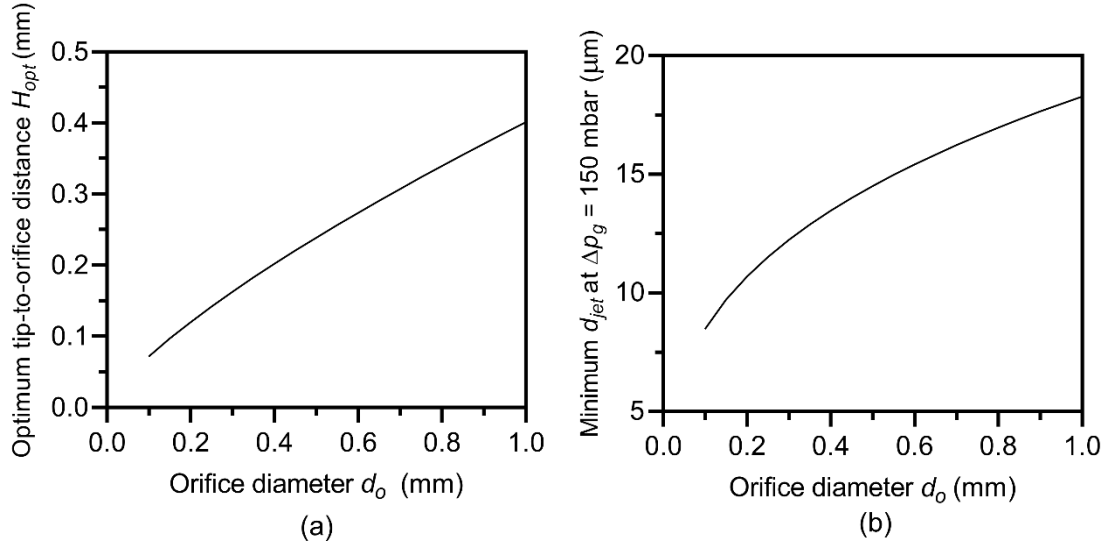


Fig. 6.2. Calculated influence of orifice diameter on the flow focusing performance. (a) Optimum tip-to-orifice distance for minimum flow rate according to Eq. (6.3) . (b) Minimum jet diameter achievable at the optimum tip-to-orifice for $\Delta p_g = 150$ mbar according to (6.2) and (6.1)

internal diameter $d_{noz} = 0.2$ mm are shown in Fig. 6.2. The calculations demonstrate that, in theory, jet diameters smaller than 20 μm are achievable for $d_{noz} = 0.2$ mm and orifice diameter $d_o < 1$ mm with an aqueous electrolyte. To reduce the requirements for precise alignment and small clearance H_{opt} , the value of $d_o = 1$ mm was chosen to conduct this investigation with a corresponding $H_{opt} = 0.41$ mm (Eq. (6.3)). The influence of throat thickness h has not been studied in detail, but it has been suggested that it should be of the order of, or smaller than, d_o to minimise downstream perturbations to the gas flow [2].

The presented design and analysis of the tooling through Eq. (6.1) - (6.3) is developed under the assumption the viscous shear stresses at the jet surface are negligible compared to the applied pressure differential, which according to [2] is valid when $\Delta p_g \gg \mu^2 / (d_o^2 \rho_l)$. The viscosity of common sodium nitrate and sodium chloride aqueous solutions used for ECM is within the range of 1 mPa.s (pure water) to 2 mPa.s (saturation concentration [149]) at 20 °C with density of 1 – 1.3 kg/m³, hence the term on the right-hand side is much smaller than the typical applied pressures > 100 mbar. Hence, the jet diameter is not expected to be significantly affected by electrolyte concentration (Eq. (6.1)) and the developed design would be applicable for a wide range of aqueous solutions although the minimum stable flow rates may depend on viscosity (Eq. (6.2)). Different mechanisms of meniscus stability in flow focusing have been reported with much more viscous liquids [150] (silicone oils with $\mu > 35$ mPa.s), where jet diameter becomes dependent on viscosity and tip-to-orifice distance with lower stable flow rates. This may be relevant for future research with non-aqueous electrolytes in EJM.

A first prototype for the flow focusing nozzle was produced by stereolithography using a Formlabs Form 2 3D printer and this was assembled coaxially with the nozzle electrode as shown in Fig. 6.3a. The focusing orifice was drilled after assembly to ensure concentricity. Initial tests were performed to prove the concept and constriction of the jet diameter by more than 50% relative to the internal diameter of the nozzle electrode was successfully observed (Fig. 6.3b). However, it was established that the focusing air flow is insufficient to clear electrolyte accumulating on the work piece surface. This led to flooding of the interelectrode gap and hindered stable machining and measurement. In

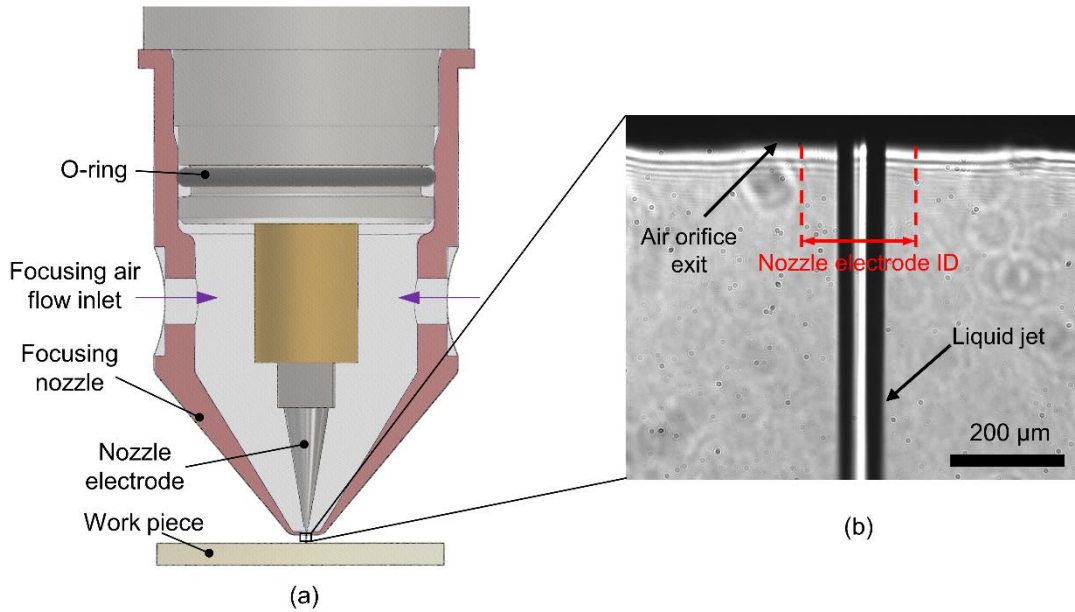


Fig. 6.3. (a) Initial design of the flow focusing arrangement for EJM. (b) Reduction in jet diameter relative to the nozzle electrode internal diameter observed optically at $Q = 2 \text{ ml/min}$ and $\Delta p_g = 150 \text{ mbar}$.

addition, there was some axial float of the focusing nozzle under the influence of the applied air pressure leading to poor control over the value of H . To address these issues a different design was developed with a second pathway surrounding the inner nozzle to deliver compressed air for clearing electrolyte from the work piece surface similar to the arrangement typically used in EJM [20]. The supply pressures of the two pathways are controlled separately by two independent pressure regulators. A metric thread was machined after additive manufacturing to constrain movement in the axial direction and the overall internal profile was produced by turning. The disadvantage of this design is that concentricity is dependent on the machined thread and the fitting of the nozzle electrode. However, it was considered adequate for the purposes of this investigation - the distance between the nozzle electrode centre and focusing orifice centre was 0.035 mm , measured with FVM after assembly. The tip-to-

orifice distance of the finished end effector assembly measured 0.66 mm (± 0.04 mm). The finished thickness of the throat was 0.33 mm.

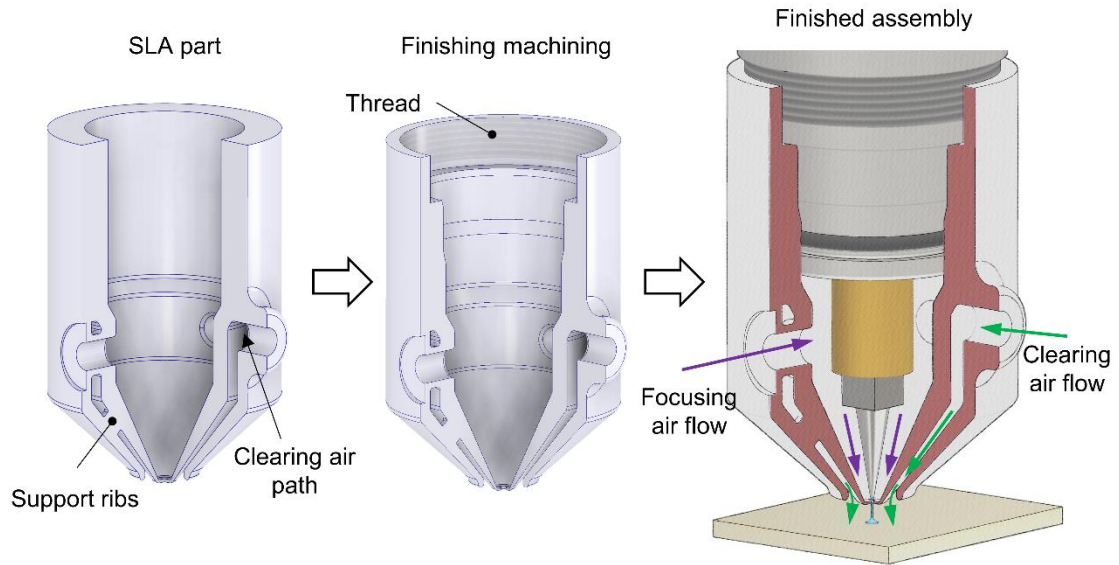


Fig. 6.4. Design and manufacturing of the prototype nozzle used for the investigation.

6.3. Characterisation of the flow focusing end effector

Characterisation of the developed end effector was carried out through electrical resistance measurements and optical imaging as described in Section 3.3.3 and Section 3.6 of the Methodology. This allowed to define the operating regions for the design.

6.3.1. Static resistance measurement in flow focusing

A stable resistance response to variation of stand-off distance is required to apply the electrolyte jet for surface topography measurement. Initial measurements of resistance as a function of time were performed with the nozzle kept static at flow rates of 2 ml/min and 1 ml/min and pressure differential $\Delta p_g = 150$ mbar (Fig. 6.5). A time-dependent sinusoidal oscillation was observed with the period changing dependent on the flow rate. This was also found to be dependent on the syringe diameter. Replacement of the 28.61 mm

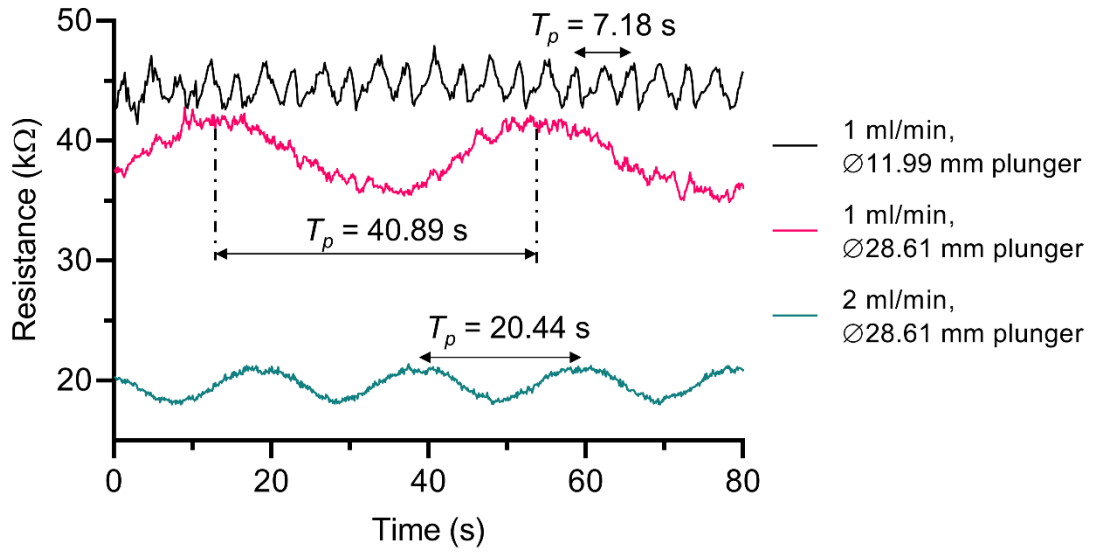


Fig. 6.5. Static resistance measurement of the flow focused jet at different flow rates and plunger diameters with annotated period of rotation of syringe pump.

diameter syringe with an 11.99 mm syringe at the same flow rate resulted in a decrease in oscillation period suggesting that this is related to the pump rotation rate. Considering that period of rotation of the pump t_p is given by $t_p = \frac{\pi x_p d_p^2}{4Q}$ where $x_p = 1.06$ mm is the pitch of the screw thread and d_p is the plunger diameter, Fig. 6.5 shows that this period correlates with the observed oscillation. Since in flow focusing the diameter of the jet is determined by the liquid flow rate, any pulsation in the flow caused by the pump leads to oscillation of jet diameter which is reflected in the measured resistance. Higher frequency components are also likely to be present in the signals corresponding to the steps of the drive motor of the pump. The observed amplitudes equate to 7% – 10% of the mean values which corresponds to approximately $\pm 5\%$ change in jet diameter (± 3 μm in absolute terms for the theoretical jet diameter of 89 μm at $Q = 2$ ml/min). If the amplitude of the observed oscillations was to be attributed to height change in a surface topography measurement this would result in an error of ± 0.15 mm indicating that the system is much more sensitive

to changes in jet diameter compared to changes in surface height. This issue can be addressed by using a pressured reservoir to supply pressure-driven flow to the system instead of the syringe pump. This could eliminate pulses in the flow, to which the system is sensitive. With the current set up, resistance measurements need to be performed in such a way that information content is at a frequency sufficiently removed from the frequency of pump oscillations. This can be achieved with an appropriate choice of scanning speed and plunger diameter. The observed jet diameter oscillation is likely to have negligible influence in terms of machining.

6.3.2. Resistance response of flow focused jets

Resistance response curves were acquired as a function of stand-off distance for the flow focusing set up as described in Section 3.3.3. The influence of flow rate Q and pressure differential Δp_g across the orifice for the flow focusing configuration were investigated and results were compared against optical images of the jet. Considering the observations in the previous section, an 11.99 mm diameter syringe was used, and the speed of the z-axis was adjusted dependent on flow rate to ensure that the flow oscillations had a period shorter than half of the duration of response curve acquisition. Response curves acquired at $\Delta p_g = 150$ mbar and different flow rates are shown in Fig. 6.6a. For flow rates from 0.5 ml/min to 2 ml/min, the curves exhibited an approximately linear region whose slope increased with reduction in flow rate. This is indicative of jet contraction since the reduction of the jet cross-sectional leads to higher resistance per unit length. However, beyond a certain stand-off distance, the resistance of the jet increased sharply, deviating from the initial linear behaviour. Observing optical images generated at the respective conditions

(Fig. 6.6c), the generated flow-focused jets exhibited lateral oscillations which increased in magnitude downstream from the nozzle exit and ultimately lead to jet break-up. This mode of flow focusing, known as “helical jetting” [151] or “whipping” [152], has been previously observed to occur beyond a certain value of applied pressure drop. Recently, Blanco-Trejo et al. [152] demonstrated that the existence of this mode is strongly dependent on the geometric parameters of the focusing nozzle design including the tip-to-orifice distance H and the shape of the throat, but general design rules have not been established yet. The acquired resistance curves reflect the extent of oscillations observed optically, with the scatter evidently increasing at the more unstable conditions.

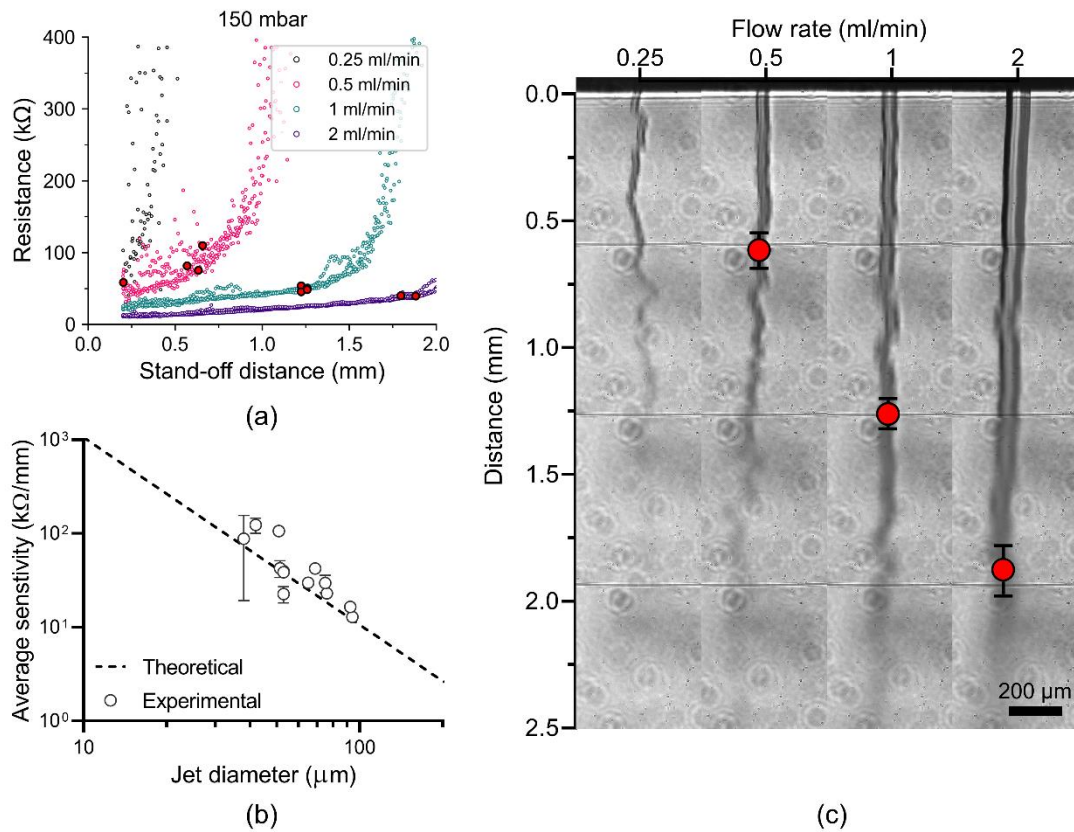


Fig. 6.6. Characterisation of the resistance of flow-focused electrolyte jets. (a) Exemplar resistance response curves at $\Delta p = 150$ mbar. (b) Measured average slope from the linear region of the resistance curve against the optically measured jet diameters. Error bars represent standard deviation from 4 repeated response curves. (c) Optical images of the jets generated at the conditions in a.

The sharp increase in resistance at larger stand-off distances corresponds to disintegration of the jet core and onset of break-up leading to a reduction of jet cross-sectional area. This has previously enabled the measurement of break-up length of liquid jets through electrical connectivity in studies of airblast atomisers [153].

In the context of this work, the break-up length of the jet defines an upper limit on the stand-off distance for electrochemical jet processing since an intact jet is required. To quantify break-up length, least-squares fitting of the function $y = Ax + B + Ce^{Dx}$ was performed to each resistance data set and the break-up location was taken as the point where the value of the second derivative $y'' = CD^2e^{Dx}$ exceeded 300 k Ω /mm². This value was arbitrarily chosen here to define a certain degree of non-linearity and enable quantitative comparison between resistance response curves at different conditions. The break-up locations identified through this method (annotated with red dots in Fig. 6.6a,c) approached the focusing nozzle exit as the flow rate reduced demonstrating that a trade-off exists between achievable jet diameter and useful working range. At 0.25 ml/min, a linear resistance region could not be identified within the investigated range of 0.2 mm to 2 mm stand-off distance, and it was not possible to apply the described fitting procedure. As seen in Fig. 6.6c, the jet at this condition was unsteady along its full length. The average sensitivity of the response curves, obtained by linear fitting for stand-off distances smaller than the break-up length for flow rates 0.5 ml/min – 2 ml/min and pressures 100 mbar – 300 mbar reduced with respect to the jet diameter and demonstrated agreement with the expected trend for a cylindrical conductor (Fig. 6.6b).

6.3.3. Operating regions of flow focusing nozzle

The operating parameter regions and achievable jet diameters of the flow focusing tool determined through optical imaging are summarised in Fig. 6.7a. Jet diameters ranging from $164\text{ }\mu\text{m}$ ($\pm 1\text{ }\mu\text{m}$) at $Q = 7.5\text{ ml/min}$ and $\Delta p_g = 100\text{ mbar}$ to $42\text{ }\mu\text{m}$ ($\pm 4\text{ }\mu\text{m}$) at $Q = 0.5\text{ ml/min}$ and $\Delta p_g = 150\text{ mbar}$ were observed over the investigated parameter window. The latter value represents a 79% reduction of diameter compared to the $200\text{ }\mu\text{m}$ ($\pm 2\text{ }\mu\text{m}$) jet generated without the flow focusing nozzle. The measured diameter values agreed with the expected values from Eq. (6.1) (dashed contours in Fig. 6.7a) with relative error in the range of 0.3% to 13% and mean error of 5%. This demonstrates the capability enabled by the developed tooling to vary the jet diameter to an arbitrary value within the reported range by choosing the appropriate flow rate and pressure. In practice, however, the application of the smallest jet diameters to EJM may be challenging since stand-off distances of approximately 0.2 mm would be required between the nozzle and work piece to utilise the stable portion of the jet. This may interfere with the air flow from the orifice exit. The “whipping” region annotated in Fig. 6.7a represents the parameter domain where lateral oscillations were seen along the full length of the jet and the measurement of stable diameter was not possible. Upon further reduction of flow rate to values lower than 0.25 ml/min , an unstable behaviour consisting of periodic detachment of liquid ligaments from the nozzle was observed, which is

known as dripping and has been described in detail in fundamental studies of flow focusing [154].

Further resistance response curves were acquired at 100 – 300 mbar and 0.25

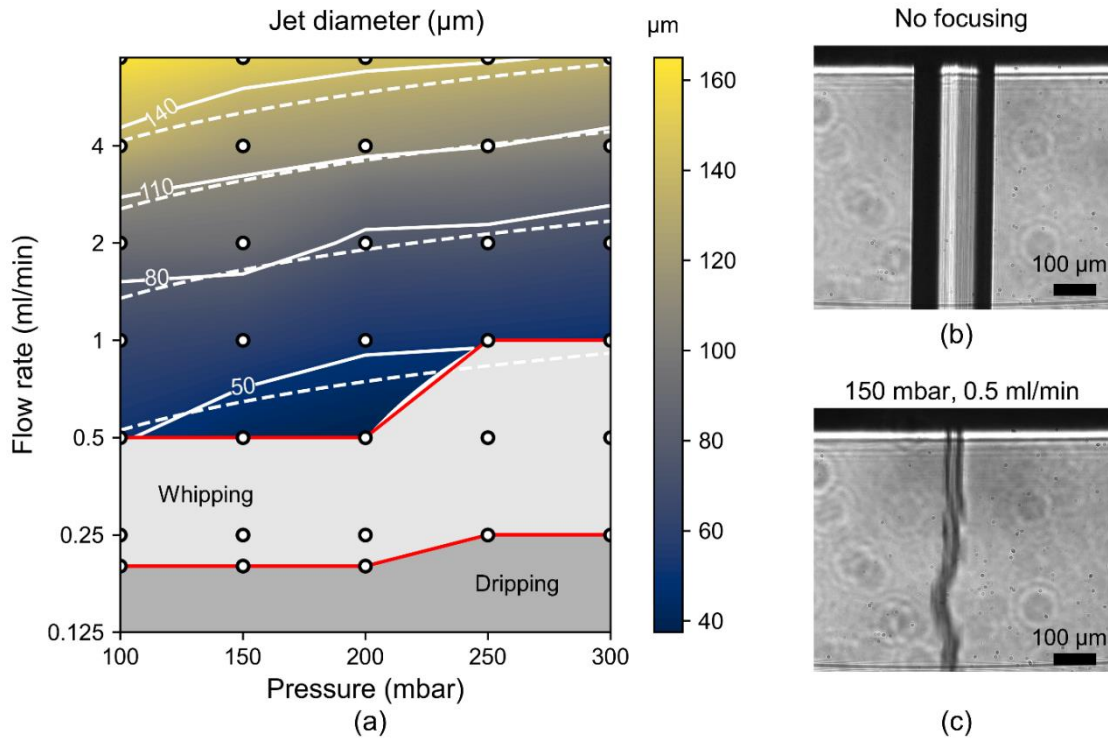


Fig. 6.7. Characterisation of the flow-focusing nozzle. (a) Optically measured jet diameters for the investigated parameter range and regions of stability. The continuous contours are fitted to the experimental data (white markers), while the dashed ones are calculated from Eq. (6.1). (b) – (c) Optical images of the jet at the annotated conditions.

ml/min – 2 ml/min (Fig. 6.8) to allow the characterisation of break-up length and working range at the lower range of observed jet size. These revealed the same behaviour of linear increase of resistance at short stand-off distances followed by rapid increase upon jet disintegration. For $\Delta p_g = 100$ mbar and $Q = 2$ ml/min, the resistance curve appeared linear over the entire stand-off range, indicating a break-up length larger than 2 mm. Upon increase of Δp_g , the high resistance regions appear to shift to lower stand-off values. At pressures above 200 mbar (see Fig. 6.8c-d), the resistance curves at 0.5 ml/min became less stable

without an identifiable linear region, suggesting that high pressures promote instability and earlier break-up.

The break-up length L_b quantified from the resistance curves as described in

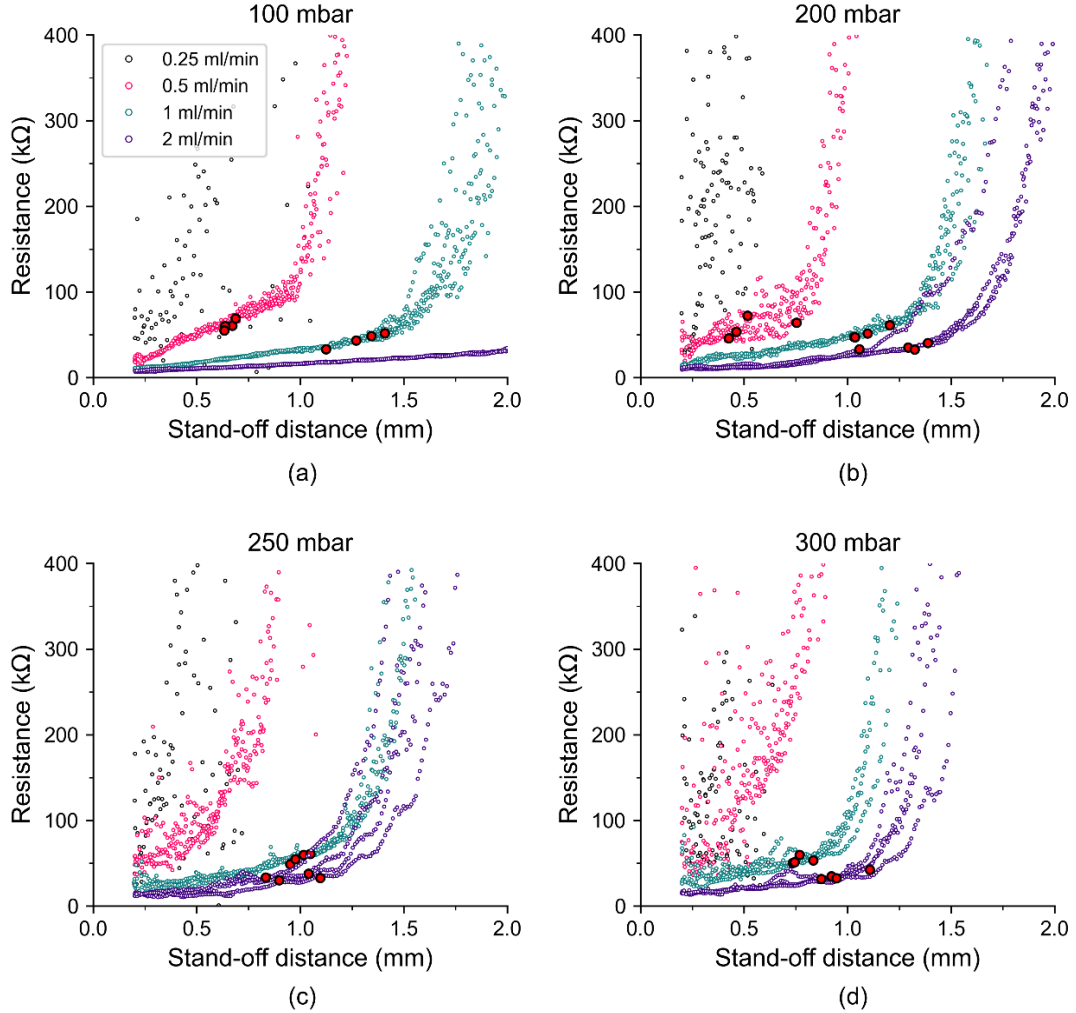


Fig. 6.8. Resistance response curves acquired at pressure differentials Δp_g of (a) 100 mbar, (b) 200 mbar, (c) 250 mbar, (d) 300 mbar.

Section 6.3.3 decreased with reducing liquid flow rate and increasing pressure differential (Fig. 6.9a). The break-up of flow-focused jets and the more general problem of liquid jets with coaxially flowing gas streams have been widely researched. A key dimensionless parameter is the aerodynamic Weber number [155] defined as:

$$We_g = \frac{\rho_g v_g^2 d_{jet}}{\gamma} \quad (6.4)$$

where ρ_g and v_g are the density and velocity of the coaxial gas respectively and γ is the surface tension of liquid-gas interface. The Weber number for the conducted experiments was calculated from the optically observed jet diameter and the pressure differential across the orifice considering that from Bernoulli's law the average velocity of the gas $v_g = \sqrt{\frac{2\Delta p_g}{\rho_g}}$. The surface tension of the electrolyte-air interface was assumed to be 75.6 mN/m² corresponding to an approximate concentration of 2.3 mol/kg of the sodium nitrate solution and a temperature of 20 °C based on published values in [156]. The break-up length was non-dimensionalised by considering the ratio L_b/d_{jet} relative to jet diameter. This exhibited an increasing trend with increasing aerodynamic Weber number up to $We_g \approx 40$ and reached a maximum value of approximately 20 for flow rate

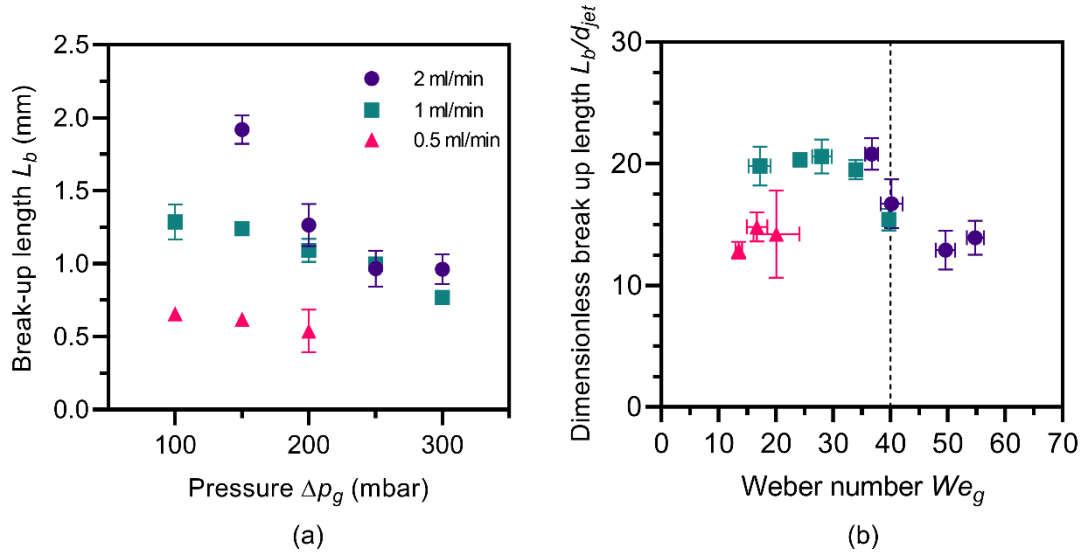


Fig. 6.9. Break-up length of flow-focused jets. (a) Break-up length as a function of applied liquid flow rate and air pressure. Error bars represent standard deviation of break-up measurements of 4 repeated resistance curves (b) Break-up length relative to jet diameter as a function of Weber number We_g

$Q = 1$ ml/min and pressures lower than 200 mbar. For $We_g > 40$, the break-up length decayed again suggesting a change of break-up mode. The existence of transitional Weber number is consistent with previous work by Gañán-Calvo et al. [157], but the authors identified a transition from axisymmetric Rayleigh-type break-up driven by surface tension at low Weber number to a more irregular break-up pattern with non-axisymmetric helical jet motion. As discussed earlier and shown in Fig. 6.6c., helical jet motions were observed in this study even at low Weber numbers (12-39 for the jets in Fig. 6.6c) and this agrees with the observations of Si et al. [151] over a similar parameter range for Δp_g between 100 mbar and 200 mbar and Q between 0.3 ml/min and 1.7 ml/min. These differences in behaviour are likely dependent on the geometry of the flow focusing devices employed. A more detailed study of the geometric configuration could enable a more optimised design and a wider working range for the flow focusing tooling in EJM.

6.4. Measurement with the flow focusing end effector

The application of the flow focusing end effector to the measurement mode of the EJM apparatus was tested by scanning the Rubert 513 reference specimen at $Q = 1$ ml/min and $\Delta p_g = 150$ mbar analogously to the experiments presented in Chapter 4. The measured resistance is compared to the expected response from Eq. (4.2) and (4.4) for the optically-measured jet diameter of $61 \mu\text{m}$ ($\pm 2 \mu\text{m}$) and a sensitivity of $20.2 \text{ k}\Omega/\text{mm}$ (S.D. = $3.1 \text{ k}\Omega/\text{mm}$) in Fig. 6.10a-b. The measured resistance change significantly overestimates the expected one and the signal does not represent the measured geometry. For the $196 \mu\text{m}$ deep and 2 mm wide groove (Fig. 6.10b), it appears that the resistance change of $4.5 \text{ k}\Omega$ is close to the expected value of $4.0 \text{ k}\Omega$ (uncertainty $\nu = 0.6 \text{ k}\Omega$) when

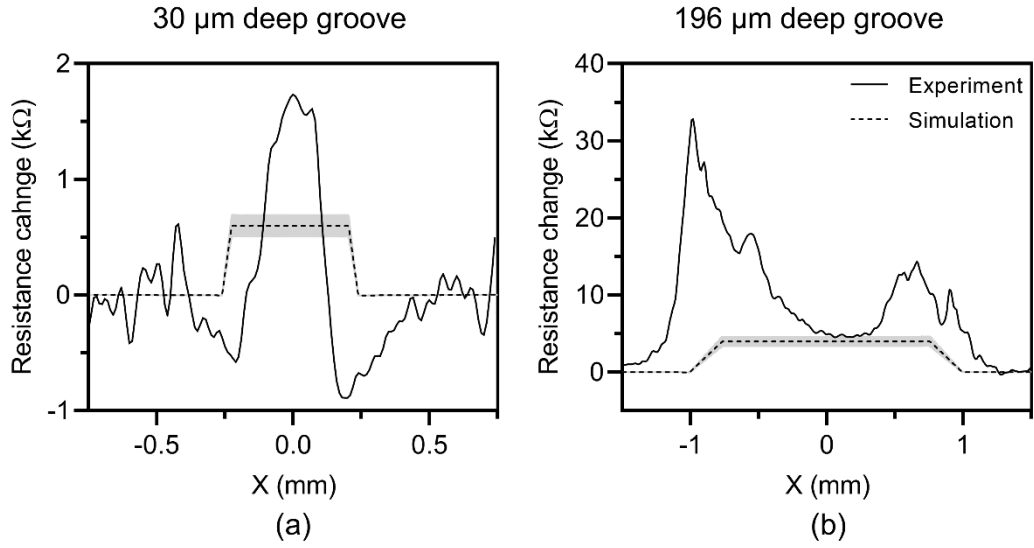


Fig. 6.10. Scanning resistance measurement of the step height reference specimen. (a) 30 μm groove. (b) 196 μm. Dashed lines represent the simulated resistance response expected from the model presented in Section 4.4.2 assuming a sensitivity of 20.2 kΩ/mm and a jet diameter of 61 μm.

the jet is near the centre of the groove. However large spikes are observed at groove edges suggesting the jet was significantly disturbed by the surface feature. This was not observed the experimental work with the conventional EJM end effector presented in Chapter 4. This phenomenon is likely due the interaction of the gaseous focusing flow with the surface and low liquid flow rates used in flow focusing leading to undesired disturbance of jet shape. As discussed in Section 6.3.1. the resistance signal is extremely sensitive to small flow pulsations leading to variation of jet diameter for the flow focusing tooling.

If the sensitivity to jet diameter change is defined as $S_d = \frac{\partial R}{\partial d_{jet}}$, then from Eq.

(2.10) for a cylindrical conductor $S_d = -\frac{8L}{\sigma\pi d^3}$. The ratio of sensitivities:

$$\left| \frac{S}{S_d} \right| = \frac{d_{jet}}{2L} \quad (6.5)$$

could be used to judge how robust the measurement system is to variations in jet diameter where it desired that this quantity is maximised. This reveals that

the system becomes more sensitive to jet diameter variation as the jet diameter is reduced and the working stand-off distance from the surface should be reduced accordingly. The fact that the system inherently becomes less robust to jet diameter and jet shape variation poses significant challenges to the resistive surface topography measurement with the proposed embodiment of the flow focusing method due to the increased requirement for precise, stable and pulse free flow. The present flow focusing tool is clearly not capable of performing surface topography measurement, but investigations of the fluid dynamic interaction with the surface with the aid of computational methods may enable design optimisation.

6.5. Machining with the flow focusing end effector

In the machining mode, the process is expected to be less sensitive to jet diameter oscillations or small variations in jet shape. Machining experiments were performed to assess the effect of flow focusing parameters on the resulting surface geometry.

6.5.1. Selection of parameters for machining trials

A key difference between the proposed application to EJM and previous research in flow focusing, is the fact that tooling in EJM is required to function in close proximity (< 1 mm) to the work piece surface. Hence, the effect of stand-off distance on machining was evaluated by machining channels with simultaneous movement of the Z-axis. The experiment was performed at a pressure of 150 mbar and flow rates of 0.5 ml/min and 1 ml/min with a stand-off from 0.4 mm to 1.2 mm, to cover the working range defined through resistance measurements (average break-up length for 1 ml/min is 1.24 mm with S.D. = 0.02 mm, see Fig. 6.6c). Machining was performed at controlled

current with supplied currents of 8 mA (estimated average current density based on jet cross-sectional area $J \approx 251 \text{ A/cm}^2$) for the 1 ml/min flow rate and 6 mA ($J \approx 376 \text{ A/cm}^2$) for the 0.5 ml/min and corresponding horizontal feed rates of 0.11 and 0.07 mm/s respectively (see Table 6.1). The motion in the Z-direction was programmed so that the Z-axis traverses from 1.2 mm to 0.4 mm stand-off distance over 4 mm of movement in the X-direction. This allows to evaluate the effect that stand-off distance has on machining performance and establish the optimum value for further experiments.

Following this, the stand-off distance was fixed at $L = 0.9 \text{ mm}$ and the influence of flow rate variation on kerf width was investigated at a fixed pressure differential of 150 mbar. For comparison of machining performance across different jet diameters, it is desirable that a constant current density is maintained across experimental conditions, since the dissolution efficiency of stainless steel in sodium nitrate is known to vary with current density [158]. The high electrical resistance of the flow-focused jets, however (see Table 6.1) requires precise current control in the milliamperage range to deliver current densities suitable for EJM ($10 - 400 \text{ A/cm}^2$) across different jet diameters, which is at the resolution limit of the power supply used in this study (Keysight N5751A). Instead, the process was operated in controlled voltage mode. For every applied flow rate, the theoretical jet diameter was calculated from Eq. (6.1) and the expected resistance was estimated by assuming that the geometry of the focused jet can be approximated by two cylinders with different diameters, neglecting the contribution of electrode overpotentials. Following the notation from Fig. 6.1 the estimated resistance R_{jet} is given by:

$$R_{jet} = \frac{4}{\pi\sigma} \left(\frac{\frac{h}{2} + L}{d_{jet}^2} + \frac{\frac{h}{2} + H}{d_{noz}^2} \right) \quad (6.6)$$

The excitation voltage was chosen for an approximately constant target current density of 210 A/cm² across jet sizes estimated based on the theoretical jet diameter (Eq.(6.1)) and resistance. Machining of channels was performed with feed rates according to Table 6.1, which were decreased proportionally to the expected jet diameter. This approach was chosen to compensate for the reduced exposure time to the jet with reduction of jet diameter and aim to maintain a more uniform depth of cut across machining conditions. All machining parameters used in this experiment as a function of electrolyte flow rate are summarised in Table 6.1.

Table 6.1. Machining parameters applied dependent on flow rate.

Flow rate (ml/min)	Expected diameter from Eq. (6.1) (μm)	Estimated jet resistance from Eq. (6.6) (kΩ)	Voltage (V)	Feed rate (mm/s)
7.5	172	6.0	298.8	0.3
5.5	147	7.4	270.1	0.25
4	126	9.4	245.1	0.21
3	109	11.8	230.5	0.19
2.5	99	13.7	224.2	0.17
2	88	16.6	215.9	0.15

1.5	77	21.4	207.6	0.13
1	63	31.0	200.8	0.11

To compare flow focusing against the conventional EJM configuration, channels were also machined with the focusing air flow disabled at 210 A/cm² and a feed rate of 0.35 mm/s. In flow focusing the average jet speed is independent of flow rate and from Eq. (6.1) is given by $v_{jet} = 4Q/(\pi d_{jet}^2) = \sqrt{\frac{2\Delta p_g}{\rho_l}} \approx 5.4$ m/s at $\Delta p_g = 150$ mbar. Hence, for the conventional EJM the electrolyte flow rate was set to 10 ml/min to obtain a similar value (5.3 m/s for a nozzle internal diameter of 200 μ m). The kerf widths reported here were averaged from 3 repeated grooves at each condition with 5 measurements taken at different positions along the groove length.

6.5.2. Influence of stand-off distance

In the stand-off variation study the machined groove visibly became wider and more unstable when the nozzle was positioned closer than approximately 0.7 mm (Fig. 6.11a). At 0.5 ml/min, no material removal was detected at high stand-off distances (larger than approximately 0.9 mm). This is likely caused by impaired electrical continuity due to jet break-up since the break-up length was estimated to be 0.62 mm (S.D. = 0.05 mm). The overall shape of the groove appeared non-uniform, which could be the result of the lateral oscillations of the

jet. The observed widening of the grooves at low stand-off distance can be explained by the interference of the surface with the air flow from the focusing nozzle. As the jet of air impinges on the surface, a stagnation region of high pressure and low velocity is formed at the impingement location [159]. When the tooling is sufficiently far from the surface (Fig. 6.11b), this does not interfere with flow through the orifice and flow focusing can occur. As the nozzle approaches the surface (Fig. 6.11c), the flow is blocked by the surface and in

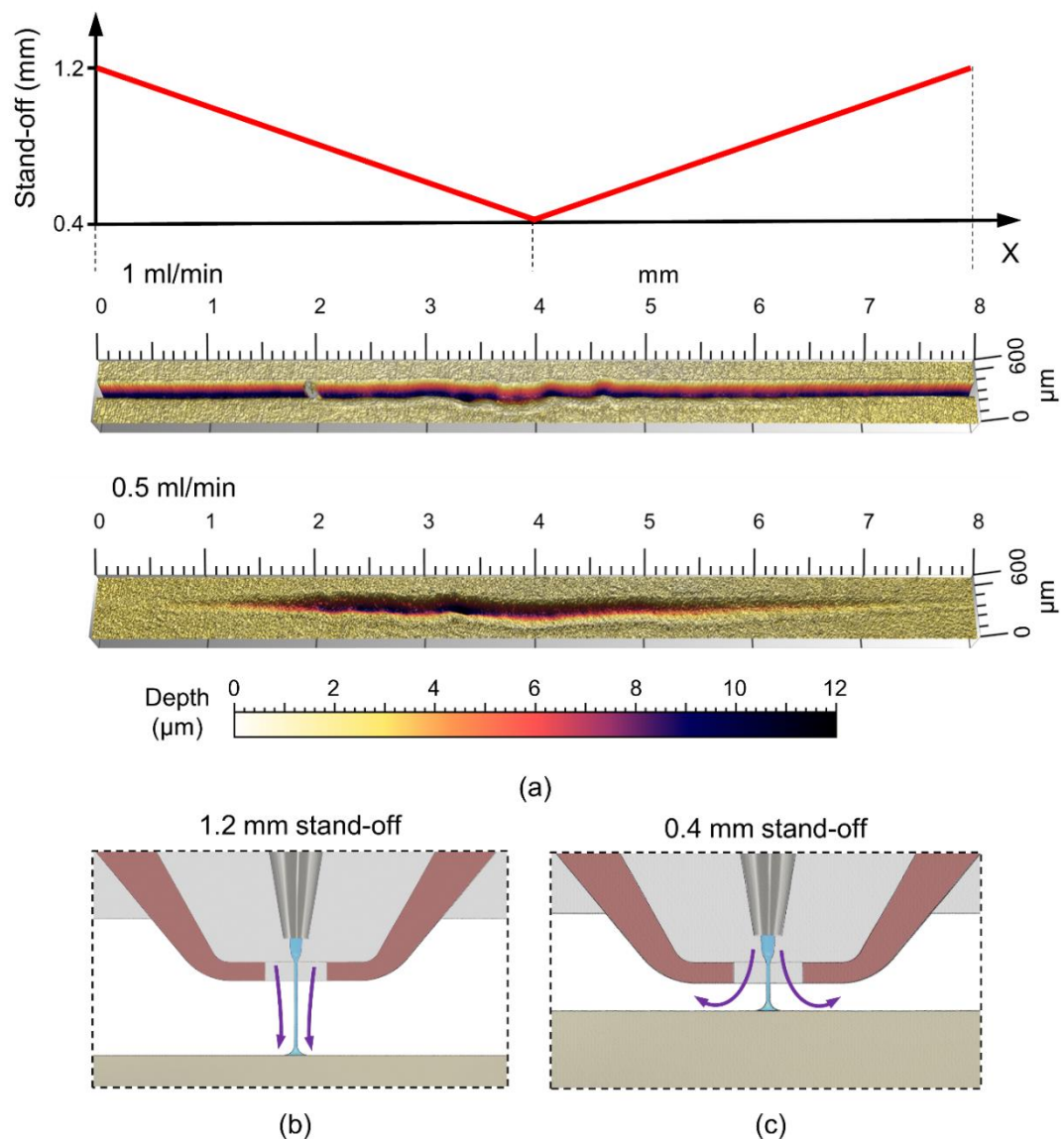


Fig. 6.11. Stand-off distance optimisation for machining. (a) Grooves machined at varying stand-off distance at 1 ml/min and 0.5 ml/min. (c) The proximity of the workpiece surface affects the flow downstream of the orifice and disturbs the flow focusing effect.

the extreme scenario, the focusing orifice could lie within the stagnation zone. It is expected that there will be a reduced pressure drop across the orifice resulting in disturbance of the flow focusing effect and consequently larger jet sizes. This suggests that, in addition to the maximum stand-off defined by the breakup length of the jet, there is also minimum stand-off distance at which the flow focusing becomes significantly disturbed by the workpiece surface. The lower limit can be controlled by reducing the orifice diameter relative to the stand-off distance.

6.5.3. Variable tool size for EJM

Following the optimisation of stand-off distance machining of straight channels was undertaken using the parameters listed in Table 6.1. The average kerf width reduced from 308 μm (S.D. = 5 μm) to 154 μm (S.D. = 7 μm) upon reduction of flowrate from 7.5 to 1 ml/min (Fig. 6.12a-b). The channel machined without flow focusing had a kerf width of 332 μm (S.D. = 6 μm), demonstrating 54% reduction through the developed tooling. However, this was also accompanied by a reduction in volumetric material removal per unit charge (Fig.

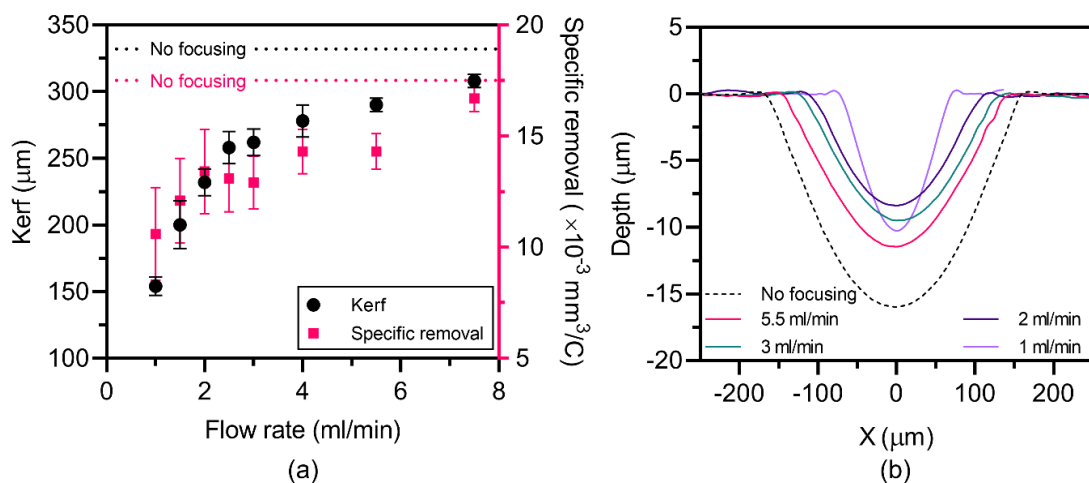


Fig. 6.12. Variable kerf through flow focusing. (a) Kerf width and specific material removal as a function of applied flow rate at a fixed pressure of 150 mbar. (b) Average profiles extracted from the machined channels.

6.12a) and reduction depth of the machined channels (see Fig. 6.12b). The specific removal reduced from $16.7 \times 10^{-3} \text{ mm}^3/\text{C}$ (standard uncertainty $v = 0.6 \times 10^{-3} \text{ mm}^3/\text{C}$) for a flow rate of 7.5 ml/min to $10.6 \times 10^{-3} \text{ mm}^3/\text{C}$ ($v = 2.1 \times 10^{-3} \text{ mm}^3/\text{C}$) for a flow rate of 1 ml/min. The specific removal measured from the conventional EJM configuration at 10 ml/min was $17.5 \times 10^{-3} \text{ mm}^3/\text{C}$ ($v = 0.5 \times 10^{-3} \text{ mm}^3/\text{C}$). It is known that specific removal of stainless steel in sodium nitrate solutions is affected by the applied current density and electrolyte temperature [160], so the current density and the theoretical temperature increase as the electrolyte flows through IEG (neglecting any heat losses) were calculated from the experimentally measured current and set process parameters in Table 6.1 as detailed in Section 3.7.4 of the Methodology and summarised in Table 6.2.

Table 6.2. Calculated average current densities and theoretical temperature increase for the channels machined with the flow focusing end effector.

Flow rate (ml/min)	Measured jet diameter (μm)	Measured current (mA) ($v = 2$)	Estimated current density (A/cm^2)	Predicted temperature increase ($^{\circ}\text{C}$)
10 (No focusing)	197 ($v = 1$)	68	223 ($v = 7$)	26 ($v = 0.8$)
7.5	154 ($v = 1$)	53	285 ($v = 11$)	30 ($v = 1.1$)
5.5	141 ($v = 1$)	38	243 ($v = 13$)	27 ($v = 1.4$)
4	121 ($v = 1$)	28	244 ($v = 18$)	25 ($v = 1.8$)
3	108 ($v = 1$)	22	240 ($v = 22$)	24 ($v = 2.2$)
2.5	100 ($v = 1$)	18	229 ($v = 26$)	23 ($v = 2.6$)
2	93 ($v = 1$)	14	208 ($v = 30$)	22 ($v = 3.1$)
1.5	75 ($v = 2$)	13	294 ($v = 48$)	26 ($v = 4.0$)
1	61 ($v = 2$)	10	342 ($v = 72$)	28 ($v = 5.6$)

The plots of specific removal against current density and theoretical temperature increase (Fig. 6.13) indicate that there is an additional factor determining dissolution efficiency since opposing trends are observed at equivalent parameters. This suggests that the dissolution efficiency for the

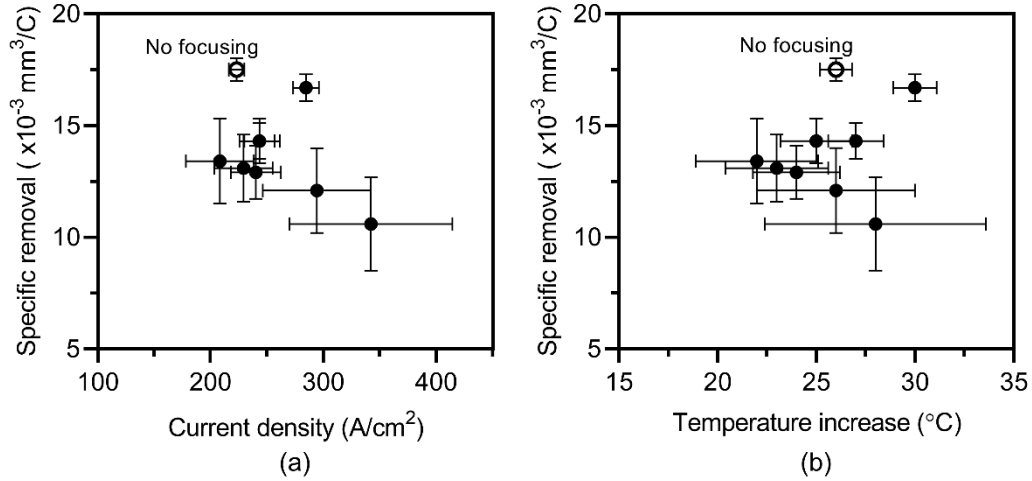


Fig. 6.13. Specific removal volume as a function of (a) estimated average current density, (b) estimated temperature increase of the electrolyte flowing through the IEG due to Joule heating neglecting heat losses.

investigated material-electrolyte system may be fundamentally related to jet size. A possible explanation for the reduction in dissolution efficiency is that the transport-controlled oxygen evolution reaction (OER), which is known to occur in parallel with the anodic dissolution of iron-based alloys in nitrate solutions, is proceeding at an increased rate (consuming a greater proportion of the current). As discussed by Lohrengel et al. [161], the OER is understood to be limited by the depletion of water (dehydration) at the anode surface when electrochemical machining products are formed. The rate of transport-controlled processes occurring at the electrode under an impinging jet has been shown [41] to be proportional to $\sqrt{\frac{v_{jet}}{d_{jet}}}$, as this is a hydrodynamic constant defining the radial flow velocity distribution on the impingement surface. The measured specific material removal is plotted against this quantity in Fig. 6.14a, where the average

jet speed was calculated from $v_{jet} = \frac{4Q}{\pi d_{jet}^2}$ and was in the range of 5.0 – 6.7 m/s.

The observed decreasing trend could indicate that there is an increased supply of water molecules to the electrode surface and respectively higher limiting current density for the OER, leading to reduced dissolution efficiency of the

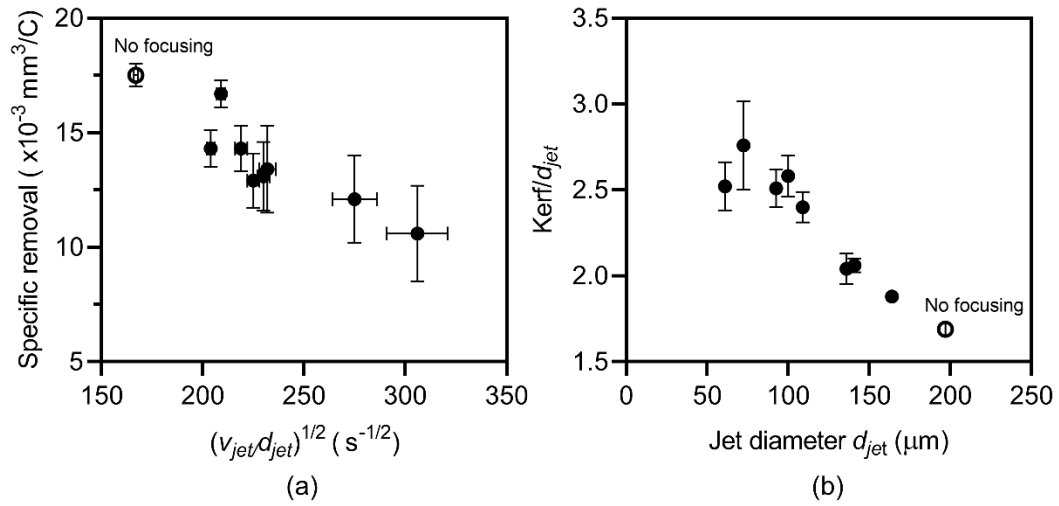


Fig. 6.14. (a) Specific volumetric material removal per unit charge as a function of $\sqrt{v_{jet}/d_{jet}}$. (b) Kerf width of the machined channels normalised relative to jet diameter as function of jet diameter.

work piece material when the flow-focused jet diameter decreased. Decreasing jet diameter also resulted in an increase of non-dimensional kerf widths relative to jet diameter, which varied from 1.88 (S.D. = 0.03) to 2.52 (S.D. = 0.11) (see Fig. 6.14d), demonstrating that kerf width does not scale linearly with jet diameter and could also be the result of the discrete electrochemical processes occurring at the anode surface. It should be emphasised that such effects are specific to the chosen work piece material – electrolyte combination.

Through flow focusing, a wide range of machined kerf widths is made available to the EJM practitioner through simple modification of electrolyte flow rate and air pressure. This reduces the need for different tool nozzles and has the potential to significantly accelerate machining operations where features of

different scale are required. Flow rate and pressure can be automatically controlled by the machine tool control programme as a function of tool path, hence eliminating process interruptions for tool changes. To showcase this new capability, a replica of the painting “The Starry Night” is engraved in a 16 mm × 13 mm area on a stainless-steel workpiece (Fig. 6.15). Different sections of the sample were machined at differing electrolyte flow rates at a fixed air pressure of 150 mbar and the respective parameters from Table 6.1 representing different “brush” sizes required dependent on the feature size. More advanced pump control could enable the dynamic variation of flow rate during machining allowing the fabrication of channels with a controlled non-uniform cross-section, which will significantly expand the surface texturing capabilities of EJM.

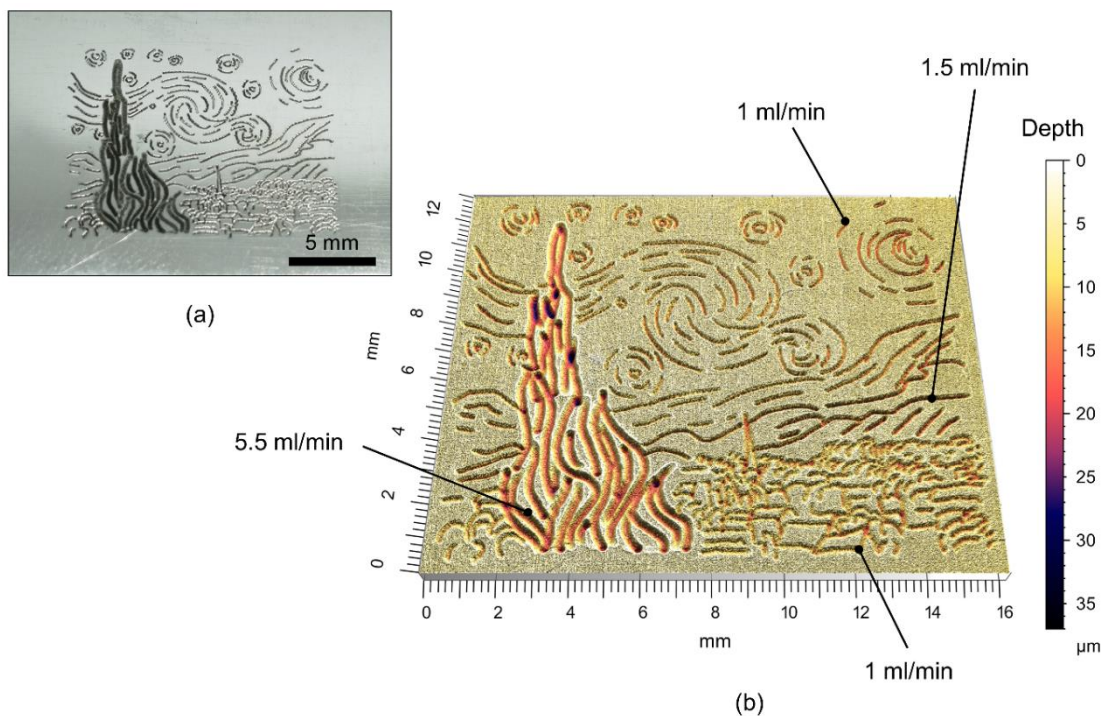


Fig. 6.15. “The Starry Night” engraved on a stainless-steel work piece through flow-focused EJM with a single nozzle electrode. (a) Optical image of the sample. (b) 3-dimensional surface topography reconstruction.

6.6. Summary

The application of the flow focusing principle to a novel end effector design for the electrolyte jet methods was investigated. Optical imaging and electrical resistance measurements were applied to characterise the operating parameter regions for the developed tooling and quantify jet diameter and break-up length. The observed stable jet lengths were in the range of 13-20 jet diameters resulting in a trade-off between achievable jet contraction and working range of the tooling.

The application of the current embodiment to the measurement mode of the electrolyte jet apparatus was shown to be unfeasible due to the instability of the jet upon interaction with surface features. However, the potential of flow focusing to increase the precision and flexibility of the EJM was successfully demonstrated. Compared to the conventional tool nozzle, a 54% reduction in machined kerf width was observed enabling the variation of tool size through selection of electrolyte flow rate or pressure of the focusing gas. The smallest jet size successfully applied for machining was $61\text{ }\mu\text{m}$ ($\pm 2\text{ }\mu\text{m}$) from an initial nozzle orifice of $200\text{ }\mu\text{m}$ ($\pm 10\text{ }\mu\text{m}$).

7. Conclusions

This thesis aimed to advance the capabilities of EJM through on-machine measurement and process monitoring. It was shown that these can be realised successfully by utilising the resistive behaviour of the IEG.

The electrical response of the IEG in EJM to change in vertical distance to the work piece surface was investigated in Chapter 4 and Chapter 5. Chapter 4 demonstrated that the Ohmic resistance contribution to IEG voltage, defined by the geometry of the gap, can be isolated from the contribution of the overpotentials caused by the electrochemical reactions at the surfaces of electrodes through the application of AC excitation with a frequency > 10 kHz. This is a consequence of the capacitive properties of the electrode-electrolyte interface, which is continuously charged and discharged under these conditions, inhibiting the anodic dissolution of the work piece. The measurement of the Ohmic resistance as function of spatial coordinates as the jet is scanned across the work piece is the basis of a novel non-destructive on-machine measurement system using the electrolyte jet apparatus.

The resistance response of the electrolyte jet as a function of stand-off distance was found to be chiefly determined by the conductivity of the electrolyte, the nozzle cross-sectional area and the jet flow speed. Stable calibration curves can be acquired for a set of these parameters. Selection of conductivity and nozzle size allows the tuning of measurement sensitivity, and these represent the key design parameters for optimising the system for a particular application. The sensitivity proved to be robust to work piece material composition without significant effects caused by surface chemistries of commercially pure nickel

and Ti-6Al-4V alloy. Contrary to the frequently applied cylindrical conductor model for the jet, significant non-linearity was found in the relationship between resistance and stand-off distance, and this was attributed to the actual shape of the falling jet. This can be addressed through piecewise linear approximation of the calibration curves. With regards to the spatial resolution of the developed measurement system, this was shown to be defined by the jet diameter while it remained stable with respect to other system parameters. The shortest 5-95% rise distance measured by traversing the jet across a groove achieved with a 0.1 mm internal diameter nozzle was 0.17 mm (S. D. = 0.04 mm) for a groove edge of width 0.03 mm. A simple analytical expression proposed to approximate the resistance response as a function of surface topography provided an adequate agreement with the experimental results predicting the 5-95% distance within 18%. The use of this approximate model provides an alternative to detailed computational fluid dynamics modelling and calculation of potential as the jet interacts with surface features, that could prove too computationally expensive for engineering practice.

Through the developed electrolyte jet measurement system, reference groove depths were successfully measured with an average error of $\pm 4.5\%$ against stylus profilometer measurement values of 30 μm (S.D. = 0.07 μm), 196 μm (S.D. = 0.4 μm), 495 μm (S.D. = 1 μm). Areal surface measurement was also demonstrated by obtaining a topography map with a root-mean-square error of 14.6 μm against focus variation microscopy for a surface with a total height variation of 331 μm .

In Chapter 5, the voltage across the IEG was investigated in the conventional EJM mode under controlled direct current excitation. It was found that, provided

that the machined surface features had a depth-to-width aspect ratio < 0.5 , they did not disturb the jet hydrodynamics significantly, and increasing voltages for a flat work piece can be correlated to increasing depth of cut in machining operations. To aid understanding of the voltage response during machining and enable its prediction, a 3-dimensional geometric model was developed to simulate the surface evolution in EJM, which can be calibrated for a specific material through machining of static pits. This was able to predict the depth of cut in static machining with a mean percentage error of 7.2% and the depth during channel machining with a mean percentage error 12.2%. The percentage error varied with depth of cut since the model did not consider the alteration of the current density distribution and the widening of the machined features as the depth of cut progresses. Coupled with the resistance response model developed in Chapter 5, the model allows for predictive planning of machining operations and the definition of an expected voltage signal, against which the actual process can be monitored. It was demonstrated that localised changes in machining fidelity, such as the depth increase along curved tool paths can be inferred from the voltage response. The results lay the foundations for advanced control of EJM, where in-process resistance measurements can be used to detect machining errors and enable on-the-fly adaptations to process parameters.

An alternative approach to apply in-process resistance monitoring was demonstrated through the implementation of a closed-loop stand-off control algorithm enabling automated surface tracking during machining. This allows the texturing of complicated surfaces without accurate knowledge of the

component shape, relaxes the requirements for accurate path planning and is particularly valuable for the robotic application of EJM.

The results from Chapter 4 and Chapter 5 demonstrate the two possible modes for resistance-based on-machine measurement in the EJM apparatus. Under sufficiently large AC frequency, the electrical response of the IEG is only dependent on geometry and the measurement is non-destructive to the surface which provides a tool for pre- and post-process metrology of surface form and large-scale surface topography. Under conventional DC excitation for surface processing, in-process measurements can be applied to monitor the depth of cut if the geometry of the surface pre-processing is known. The developed approaches can significantly accelerate EJM texturing operations by reducing the need for off-machine inspection, while improving finished part quality.

Chapter 6 addressed the limitations associated with the spatial resolution of electrochemical jet methods through a novel tooling based on the flow focusing method for generation of micro jets. With the configuration presented in this thesis, a minimum jet diameter of $42\text{ }\mu\text{m}$ ($\pm 4\text{ }\mu\text{m}$) was observed representing a 79% reduction of diameter compared to the $200\text{ }\mu\text{m}$ internal diameter of the nozzle electrode used in the experiments. The analysis of resistance response curves allowed the quantification of jet break-up length of jet, which was found to reduce with reduction of jet diameter. Stable jet lengths corresponding 13-20 jet diameters were observed for the investigated range of electrolyte flow rates $0.25\text{ ml/min} - 2\text{ ml/min}$ and gas pressure differentials of $100\text{ mbar} - 300\text{ mbar}$. The break-up length of the jet and the interaction of the focusing air flow with the work piece surface were found to impose the upper and lower limit on the working stand-off distance of the tooling respectively and prevented the

applications of the smallest observed jets to machining. The smallest jet size successfully applied for machining was $61\text{ }\mu\text{m}$ ($\pm 2\text{ }\mu\text{m}$) at a liquid flow rate of 1 ml/min and pressure of 150 mbar, which resulted in a channel kerf width of $154\text{ }\mu\text{m}$ (S.D. = $7\text{ }\mu\text{m}$) representing a 54% reduction compared to the conventional tool nozzle without focusing. The developed end effector delivers the capability to 'select' the kerf width through setting of the liquid flow rate and the gas pressure as part of the machine tool control programme. Surface topography measurement with the flow focusing end effector was not achieved in the present configuration with the acquired resistance signal not representing the measured geometry. This was attributed to the sensitivity to jet disturbances resulting from the two-phase flow interaction with the work piece surface.

The thesis demonstrated for the first time the use of an electrolyte jet as a measurement tool in addition to the known surface processing modes which can open further opportunities for application of electrolyte jet techniques. This allows for low-cost on-machine measurement and verification without the need for additional hardware. Although the currently achievable resolution is inferior compared to established techniques such as stylus profilometry or optical metrology, the developed method can reduce the need for off-machine inspection in a separate set up or location and has the potential to accelerate the overall EJM process flow. For example, overall geometry of the work piece located in the machine tool can be measured before machining to optimise the tool path and machined surface features within the resolution limit can be inspected. This could also enable faster optimisation of parameters for new material-electrolyte systems where experimental material removal

characterisation is required and this usually involves multiple machining and measurement steps.

Further to this, the developed in-process resistance monitoring can be applied to enable more 'intelligent' EJM with real-time feedback of machining performance. This is in contrast to the conventional approach where quality control is only possible after the machining operation has been completed. In-process resistance monitoring can enable detection of machining errors or machine faults at the point of failure. The simultaneous machining and measurement could also be valuable to the finishing of additively manufactured parts with EJM, since the extent of reduction of irregularities on the surface will be reflected in the acquired signals. Apart from prediction of resistance for process monitoring, the geometric model of surface evolution in EJM can be applied for the simulation of finished geometry before machining and optimisation of the tool path and process parameters and could be integrated with computer-aided manufacturing software for planning of EJM operations.

The application of flow focusing to EJM, proven in this thesis, could offer significant advantages against the conventional configuration by dispensing need to fabricate sub-100 μm nozzle electrodes, reducing the risk of clogging due to salt crystallisation, enabling automated control over machining resolution and reducing non-value-added time associated with tool changes when machining features of different sizes. The concept presented in this thesis is a step towards improving the competitiveness of EJM against more established processes such as laser surface texturing in applications requiring high machining resolution and quality surface finish.

8. Future work

Several future research directions that stem from the reported work can be identified.

The resistance-based electrolyte jet measurement system developed in Chapter 4 proved to be a capable method for measurement of large-scale surface topography. Further investigations could help define the limits in terms of measurable geometry before fluid dynamic interactions disturb the measurement. This would also help to better define the scope of validity of the proposed mathematical model for the transfer function of the system. The influence of work piece material on the measurement also requires further research as this has implications for the topography measurement of heterogeneous surfaces.

In Chapter 4, the surface topography measurement was achieved through the acquisition and analysis of the real (resistive) component of the electrical impedance at high frequencies. However, it may be possible to extract further information from the impedance spectrum and in particular the imaginary (reactive) component. The capacitance of the work piece - electrolyte interface expected to depend on the electrochemically active surface area of the electrode and has been used for its approximate determination in conventional electrochemical cells [162]. For highly rough and porous micro-textured surfaces this can be much larger than the projected geometric area. The ratio between the actual area of the interface and the projected area is known as the developed interfacial area ratio and is considered an important surface topography descriptor for functional surfaces. Provided that the impedance

measurement in the electrolyte jet system remains constrained to a controlled area of the work piece defined by nozzle size, at least a comparative appraisal of surface topography on a scale smaller than the jet may be feasible. In addition, the influence of material surface chemistry on the impedance response, which was sought to be minimised for the purposes of the study in Chapter 4, could be valuable for other applications where a localised material inhomogeneity may need to be detected prior to surface processing.

The analytical model of surface evolution in EJM, developed in Chapter 5 proved capable to predict removal geometry resulting from straight and curved tool paths, but only machining on initially flat surfaces was investigated. For applications to realistic engineering components, the simulation of processing on curved surfaces will be of interest. This may pose additional challenges to the presented approach as the current density distribution is affected by the slope and curvature of the surface. Hence, additional corrections of the removal rate function $P(\mathbf{x}', t)$ dependent on the angle of incidence of the jet and local distance between the nozzle and the surface, need to be sought. Model refinement to consider the lateral growth of machined features would also allow for more accurate predictions. A further step in developing the proposed modelling approach is the implementation of optimisation algorithms for solution of the inverse problem to obtain jet trajectory and feed rate based on a specified desired surface geometry.

The on-machine and in-process monitoring tools presented in this thesis create opportunities for further development of advanced closed-loop process control. Machining parameters such as supplied current and feed rate can be adapted automatically during machining to maintain a required process resistance

corresponding to the desired machined geometry. This can enable the machine tool to react to pre-existing surface features in the work piece or material inhomogeneities in order to maintain machining fidelity consistent with the design. The combination of the non-destructive AC jet measurement and DC in-process measurement within the same end effector could further enhance processing capabilities – a “measurement jet” can allow for automated surface tracking, while the “machining jet” is used to monitor the dissolution process. Further research questions can be asked in relation to the information contained within the electric signal during machining beyond the ohmic contribution resulting from the IEG geometry. For example, some noise components may have electrochemical origins and provide feedback for the mechanisms of dissolution of the surface.

Chapter 6 successfully demonstrated the application of the flow focusing principle to EJM, but further work could optimise the tooling design and increase the operating range of the proposed concept. More detailed investigation into the factors determining the break-up of the jet and the liquid and air flow interaction with the work piece surface through experimental and computational fluid dynamics studies could further push the limits of miniaturisation with EJM. Surface measurement with the flow focusing end effector presented in this thesis proved to be challenging due to the high sensitivity of the system to jet instabilities upon interaction with features of the surface. The nature of this interaction deserves further attention to establish whether an operating region and geometric configuration exist that allow measurement with improved resolution relative to the one achieved with conventional nozzles in Chapter 4. If this proves to be feasible, and opportunities to extract chemical information

and surface capacitance from jet-based impedance measurements are exploited, this could deliver a powerful metallurgical characterisation capability. In combination with crystallographic etching capabilities of EJM, a tool can be envisaged which can provide low-cost crystallographic texture analysis based on impedance measurements of jets focused down to sizes comparable to the grain size of the material within a self-contained set-up. This could be particularly valuable in industry for defect detection in the manufacturing of single-crystal components for example.

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Appendix

A. Work piece materials used in this thesis

Table A.1. Nominal composition of Nickel 201.

	Ni	Fe	Mn	Si	Cu	C	S
wt. %	≥ 99.0	≤0.4	≤0.35	≤0.35	≤0.25	≤0.02	≤0.01

Table A.2. Nominal composition of Ti-6Al-4V.

	Ti	Al	V	Fe	O	C	N	H
wt. % max	91.0	6.75	4.5	0.4	0.2	0.08	0.05	0.015
wt. % min	87.6	5.5	3.5	-	-	-	-	-

Table A.3. Nominal composition of stainless steel grade 304.

	C	Cr	Mn	Si	P	S	Ni	N	Fe
wt. % max	0.07	19.5	2.0	1.0	0.05	0.03	10.5	0.11	Balance
wt. % min	-	17.5	-	-	-	-	8.0	-	

B. Reference specimen measurements at varying parameters

Table B.1. Sensitivities applied for reference specimen measurement as a function of process parameters. Nozzle diameter was kept constant at 0.23 mm

Flow velocity v_{jet} (m/s)	Feed rate v_f (mm/s)	Conductivity σ (mS/cm)	Initial stand-off L_0 (mm)	Calibrated sensitivity S within L_0 to $L_0 + 0.5$ mm (k Ω /mm)
7.8	0.5	60.6	0.5	4.36 (S.D. = 0.01)
10.4				4.39 (S.D. = 0.01)
13.3				4.41 (S.D. = 0.03)
15.4				4.38 (S.D. = 0.03)
17.4				4.29 (S.D. = 0.05)
	0.25			
10.4	1.0	60.6	0.5	4.39 (S.D. = 0.01)
	2.0			
	0.5	21.3	0.5	12.08 (S.D. = 0.07)
10.4		100.3		2.64 (S.D. = 0.01)
10.4	0.5	60.6	0.25	4.23 (S.D. = 0.02)
			0.75	4.53 (S.D. = 0.04)
			1	4.66 (S.D. = 0.03)
			1.5	4.78 (S.D. = 0.03)

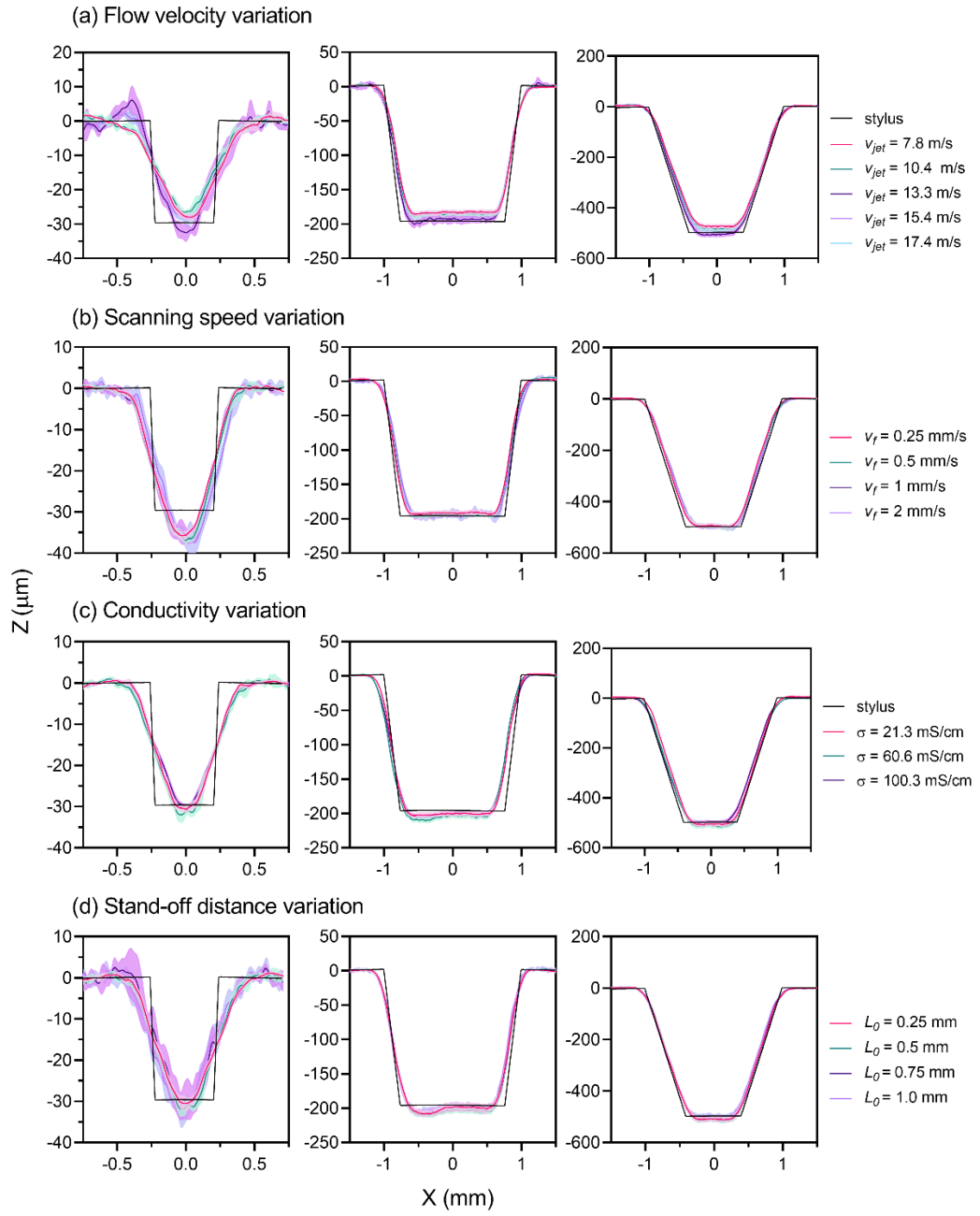


Fig. B.1. Measured profiles of the step height reference specimen at the parameters from Table B.1.