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Novel niobium carbide cermet and manufacturing techniques for moulds towards improving durability and flowability in ceramic injection moulding

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Abstract
Ceramic injection moulding (CIM) enables net-shape fabrication of complex ceramic components for biomedical, aerospace, energy and luxury sectors but requires high injection pressures (up to 2000 bar) due to the limited flowability of ceramic feedstocks with powder volume fraction up to 60%. Combined with the abrasive nature of the feedstock, this leads to severe mould wear and frequent replacement, increasing process cost and limiting precision. This work investigates the process chain of CIM by exploring a new mould material, machinability investigation, laser surface structuring for flowability and mould surface repair.
Niobium carbide with nickel binder cermet (NbC-Ni) was investigated as the mould material which offers a hardness (1468 HV_{30}) comparable to tungsten carbide while avoiding cobalt binders. Initial CIM tests show that NbC-Ni suffers less dimensional wear in depth (~46% lower) and width (~86% lower) after 10,000 shots in comparison to tool steel (Uddelholm Stavax® ESR) with Al_2O_3 feedstock. This shows NbC-Ni is suitable for moulds but its high hardness, modest thermal conductivity and multi-phase microstructure pose significant challenges for machining and surface texturing. Hybrid laser-electrochemical machining (LECM) is explored as a hardness-independent process for machinability evaluation. Compared to conventional electrochemical machining (ECM), LECM demonstrates improvement in shaping precision by 24% and surface quality (Sa) by 30%. In parallel, laser surface texturing with feature sizes below 50 µm is being investigated to enhance feedstock flowability while avoiding texture replication on products. Preliminary experiments have shown it is possible to fabricate micro-dimples with a diameter of 30 µm and a wide range of aspect ratios, with minimal thermal defects at the perimeter on NbC-Ni using femtosecond laser machining. Additionally, a proof-of-concept process chain was developed for bi-directional ECM and electrochemical additive manufacturing (ECAM) using a single-hybrid tool towards repair of mould surfaces.

Keywords: Injection moulding, Electrochemical Machining, Hybrid laser-ECM, Ultrashort pulsed laser, Electrochemical additive manufacturing, ceramics, cermets

1. Introduction

Ceramic injection moulding (CIM) is a near net-shape manufacturing process for producing geometrically complex ceramic components [1]. It is widely applied in biomedical, aerospace, energy and luxury sectors using alumina and zirconia feedstocks. However, CIM faces challenges in achieving high dimensional accuracy, surface quality and process reliability, particularly for components with sub-mm/µm features. A key limitation in CIM arises from the rheological behaviour of ceramic feedstocks, which typically contain up to 60 vol.% ceramic powder. The resulting high viscosity affects flowability [2] and complicates filling of fine mould features, necessitating injection pressures up to 2000 bar. These high pressures in combination with the abrasive nature of ceramic particles, accelerate mould wear and degrade surface integrity on common mould materials like tool steels, making CIM an expensive process.

Cermets such as tungsten carbide-cobalt offer improved wear resistance but rely on Co binders, which are increasingly restricted due to health and environmental concerns. In this context, niobium carbide-nickel binder cermet (NbC-Ni) is a promising alternative as it exhibits hardness (1468 HV_{30}) similar to WC-Co [3]. However, its high hardness, modest thermal conductivity (14 W/mK) and multi-phase composition make it difficult to machine with both conventional and non-conventional processes. Hybrid laser-electrochemical machining (LECM) [4] represents a possible solution to these challenges. It combines µs-pulsed electrochemical machining (ECM) with coaxial ns-pulsed laser to enhance electrochemical reaction kinetics, weaken passivation and improve removal of less reactive phases. Additionally, the process is independent of material hardness and maintains the athermal nature of electrochemical machining (ECM), making it particularly suitable for machinability (shaping) investigation of NbC-Ni.

Beyond material and machining considerations, mould surface functionality plays a critical role in CIM performance. Surface texturing has been proposed as a method to reduce the friction coefficient and improve feedstock flow behaviour [5]. However, surface features on moulds can replicate onto the products. Therefore, texturing is explored in this study with feature dimensions targeted below 50 µm to minimise texture transfer.

Femtosecond laser micromachining was employed for this, as it is hardness-independent and minimises the influence of modest NbC-Ni thermal conductivity to enable precise surface texturing with minimal thermal defects.

Furthermore, given the high costs and lead times of precision mould manufacturing, repair strategies are being researched for resusing dies and moulds [6]. Hence, this work also presents a proof-of-concept repair process chain combining ECM for surface smoothening and exposing surface/subsurface cracks, with electrochemical additive manufacturing (ECAM) for localised and athermal repair-material deposition.

2. Experimental details

2.1. Ceramic powder injection moulding machine

The CIM tests were conducted on a Wittmann® Smartpower 50 machine (**Fig. 1a**) and mould inserts (**Fig. 1b**) were machinined out of tool steel (Uddelholm Stavax® ESR) and NbC-Ni cermet using die-sinking electrical discharge machining at a mould manufacturer without texturing. The NbC-Ni cermet was synthesized in-house through liquid phase sintering at 1420 °C. The CIM feedstock was K1008 from INMATEC® (Al_2O_3, 96 wt% in polyolefine based binder). Experiments were conducted for 10,000 shots at an injection speed of 40 ccm/s and a barrel temperature of 150 °C. To ensure complete filling of the mould, a backpressure of 600 bar was used. The mould inserts were thoroughly cleaned prior to measurements.

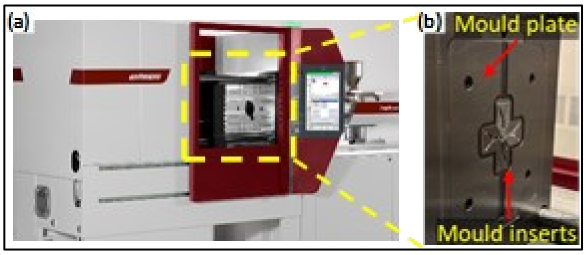

Figure 1: (a) Wittmann® Smartpower 50 machine and (b) mould inserts.

2.2. Mutlifunctional electrochemical machine tool

The in-house developed 3-axis multifunctional electrochemical (EC) machine tool (**Fig. 2**) was used for NbC-Ni ECM/LECM machinability (shaping) investigations and ECM/ECAM experiments for proof-of-concept mould surface repair demonstration. It is equipped with a ns-pulsed green laser source (532 nm, max avg. 30 W) to coaxially and simultaneously supply laser and electrolyte through a tubular steel cathode-tool (1.2 mm O.D., 0.65 mm I.D.) onto the workpiece (anode) surface to realise LECM (**Fig. 2b**). For more details on the process principle and machine tool, the readers are referred to [4]. The tubular tool can be replaced with a hybrid EC-tool (**Fig. 2a**) to enable sequential ECM and ECAM processes by switching the electrolyte and tool/workpiece polarity [6]. It supplies electrolyte through a metal nozzle (0.3 mm I.D.) with an air-column around it to localise machining and deposition.

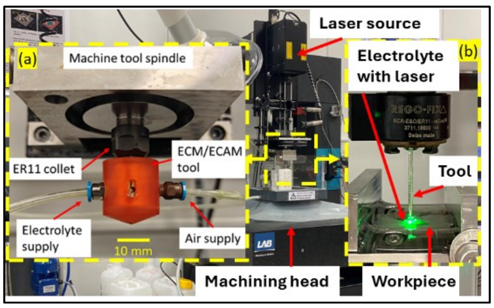

Figure 2: Multifunctional machine tool with (a) hybrid EC-tool for ECM/ECAM and (b) tubular tool for ECM/LECM.

For ECM/LECM experiments, 30 × 10 x 4 (L x W x H) mm bars of NbC-Ni were prepared through wire-EDM on the GF Cut E 600 and the surface was ground to Sa of ~1.2 µm (measured with Senosofar® S Neox, L-filter 25 µm). Preliminary experiments were conducted to find suitable process parameters (**Table 1**) to conduct groove machining experiments using 2.4M $NaNO_3$ electrolyte.

Table 1. Process parameters for ECM and LECM of NbC-Ni.

Parameter	Value
Voltage parameters	30 V, 10 µs pulse on-time, 50% duty cycle
Interelectrode gap	80 µm
Feed rate	80 µm/s
Electrolyte flow rate	0.5 mL/s
Laser parameters	48.5 µJ, 150 kHz, 50 ns pulse on-time

For ECM/ECAM experiments, groove machining of flat stainless steel (SS304) substrates with copper (Cu) deposition on machined grooves with subsequent machining was initially investigated using 1M $CuSO_4.5H_2O$ electrolyte to realise bi-directional process sequencing. Thereafter, nickel (Ni) deposition was conducted using 0.4M $Ni(NH_4)_2(SO_4)_2.6H_2O$ electrolyte in premachined cavities (3 mm width, 1.5 mm depth) on SS304 substrates to check the feasibility on a mould representative feature. The process parameters for these experiments are summarised in **Table 2**, which are based on preliminary experiments on these materials. Afterwards, all samples (ECM, LECM, ECAM) were cleaned in de-ionised water.

Table 2. Process parameters for ECM and ECAM experiments.

Parameter	ECM of SS / Cu	ECAM of Cu	ECAM of Ni
Voltage	20 V / 10 V	5 V	5 V
Interelectrode gap	100 µm	100 µm	100 µm
Electrolyte flow rate	0.45 mL/s	0.1 mL/s	0.1 mL/s
Scan rate	0.8 mm/s	0.8 mm/s	0.8 mm/s
Scan layers	2	40	20
Air pressure	1.1 bar	1.1 bar	1.1 bar
Voltage pulse parameters	20 µs pulse on-time, 50 % dutyc cycle		

2.3. Laser texturing machine

Surface texturing of ground NbC-Ni samples was conducted using a femtosecond laser micromachining system (CARBIDE, Light Conversion Ltd.) [7] (**Fig. 3a**). The system has a laser with an output wavelength of 1030 nm, pulse duration of 250 fs, maximum average power of 20 W, maximum pulse energy of 400 µJ and maximum pulse repetition rate (PRR) of 1 MHz. The laser beam has a Gaussian intensity profile and is focused with a 125 mm focal length lens for a beam waist radius of 17 µm.

A design of experiment approach was employed to produce micro-dimples with base diameters around 30 µm through percussion drilling (**Fig. 3b**).

The experimental matrix involved systematic variation of pulse energy from 2 to 4 µJ in increments of 0.5 µJ and the number of

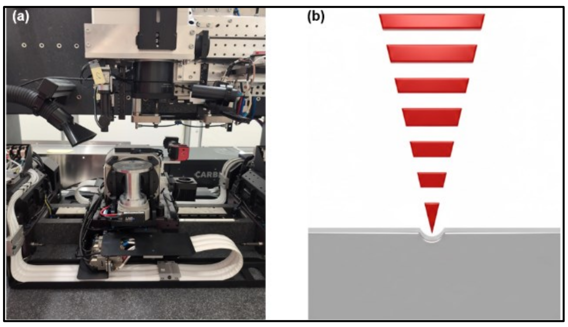

Figure 3: (a) Femtosecond laser micromachining system (b) Schematic of percussion drilling scan strategy.

pulses from 20 to 100 in steps of 10 pulses. Other parameters, such as PRR and pulse duration, were kept constant. The complete set of processing parameters are presented in **Table 3**.

Table 3. Process parameters for micro-dimples laser machining.

Parameter	Values/description
Pulse energy (μJ)	2, 2.5, 3, 3.5, 4
Number of pulses per micro-dimple	20-100 (step size 10)
Pulse repetition rate (KHz)	500
Pulse duration (fs)	250
Scan strategy	Percussion drilling

3. Results and discussion

3.1. Wear assessment of mould insert materials during CIM

The wear behaviour of tool steel and NbC-Ni mould inserts was evaluated after 10,000 CIM shots by analysing the dimensions and surface topography of the uppermost step of the Y-shaped mould cavity (**Fig. 4a**). Dimensional measurements were performed using a Werth VideoCheck® HA coordinate measuring machine equipped with a Renishaw® SP25 tactile probe (MPE 1.5 μm). Surface topography was characterised using a Sensofar S Neox optical microscope operating in confocal mode, with a third-order polynomial F-filter applied to remove form. Three measurements were done before and after experiments to calculate the average and standard deviation for dimensional wear and surface topography metrics (Sa, Sal).

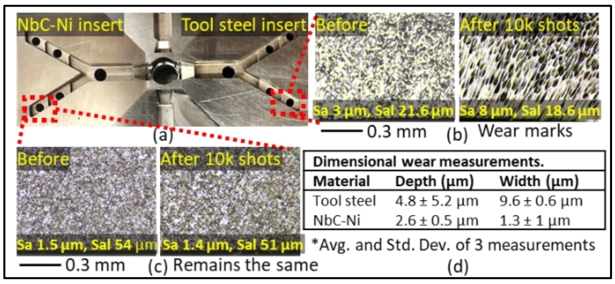

Figure 4. (a) Moulds inserts, surface topographies of (b) tool steel and (c) NbC-Ni cermet, and (d) dimensional wear measurements.

Distinct wear marks were observed on the tool steel mould insert surface after 10,000 shots alongwith a pronounced degradation of the surface integrity. The surface roughness (Sa) increased by approximately 167% compared to the as-manufactured condition (**Fig. 4b**). In contrast, the NbC-Ni mould insert surface exhibited no visually observable wear features and the measured Sa value remained similar (**Fig. 4c**). The autocorrelation length (Sal) of NbC-Ni insert was also higher than for tool steel indicative of less wear marks. The dimensional wear measurements summarised in **Fig. 4d** confirm these observations. The tool steel insert experienced material wear of 4.8 ± 5.2 μm in depth and 9.6 ± 0.6 μm in width. In comparison, the NbC-Ni insert experienced less wear, with an increase in depth of 2.6 ± 0.5 μm and in width of 1.3 ± 1.0 μm.

These results demonstrate the superior wear resistance of NbC-Ni under high-pressure CIM conditions and highlight its potential as a durable mould material. Therefore, its machinability was further investigated with ECM and LECM.

3.2. Machinability evaluation of NbC-Ni cermet with LECM

Apart from the high hardness (1468 HV_{30}) and low thermal conductivity (14 W/mK) of NbC-Ni, which make it challenging to machine (shape) with conventional and non-conventional processes, it also has a high passivation tendency (oxide formation) and different phases with varying reduction potentials that make it difficult to machine by ECM with high precision and surface quality. In LECM, a laser acts as assisting energy source to ECM by locally heating the workpiece surface to focus current density in the center to enhance reaction kinetics, passivation weakening and removal of less reactive phases, leading to improved precision and surface quality [4]. This is demonstrated by the results shown in **Fig. 5**, where the machined grooves and surface topography metrics (Sa, Sdr) were measured (3 measurements) using Sensofar® S Neox. For the surface topography, a F-filter of polynomial order 3 and L-filter of 25 μm were applied. Furthermore, the cross-section was observed by SEM for better visualisation of removal behaviour. The profiles in **Fig. 5a** show improvement in precision by 24% with LECM where the width reduced from 1.95 mm to 1.48 mm along with an increased depth from 0.11 mm to 0.14 mm icompared to ECM. Additionally, due to the current density focusing effect, the residual hill in the groove center with ECM was removed with LECM. The improved dissolution behaviour is further evidenced in **Fig. 5b**, where LECM shows a reduction in inhomogeneous material removal. Preferentially passivating ridges and dissolving crevices observed in ECM were suppressed, indicating a more uniform phase dissolution. This improvement is also reflected in the surface topography measurements as the surface roughness (Sa) and developed interfacial area ratio (Sdr) reduced by ~30% and ~50%, respectively, with LECM (**Fig. 5c**). The passivation weakening capability of LECM on NbC-Ni surface was also verified with energy dispersive X-ray spectroscopy (EDS), which showed reduction in O content by 11 wt.% in comparison to ECM.

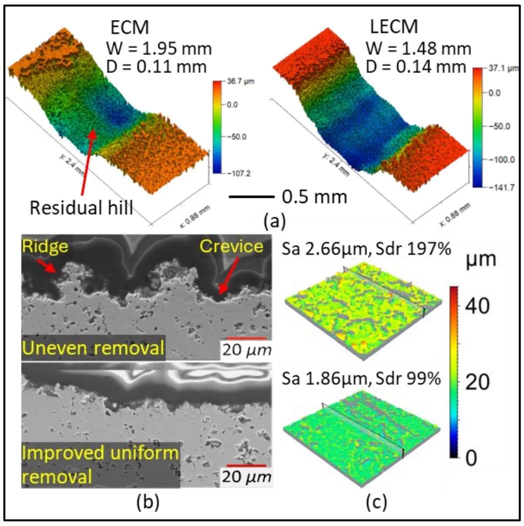

Figure 5. Machining results of ECM and LECM on NbC-Ni. (a) Shape morphology, (b) cross-section views and (c) surface topography.

3.3. Laser surface texturing investigation

The micro-dimples on the NbC-Ni surface were analyzed using a Philips XL 30 FEG SEM microscope and Sensofar® S Neox. Three depth measurements of micro-dimples were taken to calculate the average and standard deviation. The surface topography of the micro-dimples is shown in **Fig. 6**, where **Fig. 6a** shows the SEM image (top view), and **Fig. 6b** shows the 2D profile of the micro-dimples created with 50 pulses of 4 μJ pulse energy.

Minimal thermal defects and recast layer were observed on the periphery of the micro-dimples. The 2D profile shows that the micro-dimples have an inverted conical shape, which is a result of the Gaussian profile of the incident laser beam.

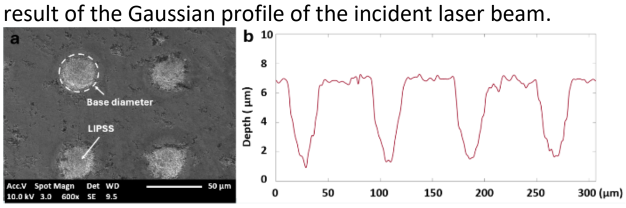

Figure 6. Surface topography of micro-dimples. (a) SEM image showing top view of surface, (b) 2D profile of micro-dimples

The variation of the micro-dimples depth with pulse energy and number of pulses is shown in **Fig. 7**. The dimple depth increased almost linearly with an increase in pulse energy as well as pulse number. However, no significant variation in the base diameter was observed by varying either of the two parameters. Therefore, for a constant base diameter of 30 µm, a wide range of micro-dimple depths (1.5 to 12.5 µm) can be achieved, which corresponds to aspect ratios between 0.05 and 0.45.

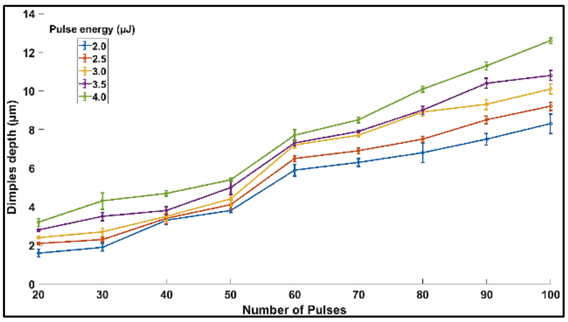

Figure 7. Micro-dimple depth evolution with increasing pulse energy and number of pulses.

3.4. Electrochemical manufacturing method for surface repair

This section presents work in progress results for developing an electrochemical-manufacturing based mould surface repair technique for reusing moulds, thereby improving durability. To assess the feasibility of the repair technique, a sequential processing with ECM and ECAM was evaluated first. The result in **Fig. 8a** shows different process sequences i.e. (i) ECM groove on SS substrate of 14 mm length (red line), (ii) ECAM of Cu in the machined groove up to 10 mm length (green line) and (iii) ECM groove machining of Cu deposit up to 7 mm length (black line). This shows the possibility to achieve bi-directional and sequential material removal and addition in close proximity, which is demonstrated for the first time in mask-free electrochemical manufacturing. The average of 3 measurements indicates that ECAM had a scan layer thickness of ~0.5 µm.

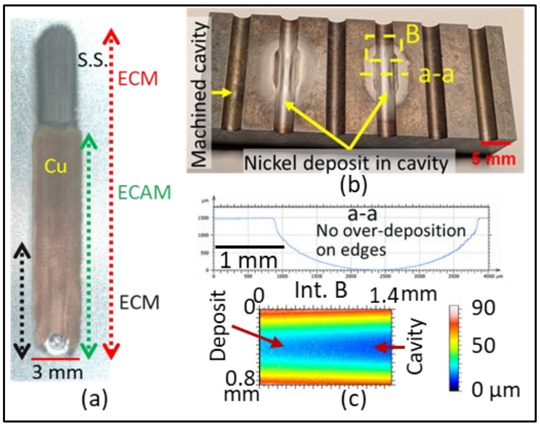

Figure 8. (a) Process sequences of ECM and ECAM. (b) Mould surface repair demo with measurement of (c) 2D profile and 3D interface.

This approach was used to investigate the mould surface repair feasibility using Ni as a repair-material on a SS substrate with premachined cavities representative of mould features (**Fig. 8b**). These initial materials were used to test feasibility to further research nickel-based high performance alloys as repair materials for mould substrates (NbC-Ni, tool steels) in the future. The measurements of the 2D profile and 3D interface in **Fig. 8c** show that with an appropriate ECAM tool scan strategy the shape of the cavity can be maintained while depositing Ni on the SS surface with negligible overdeposition at cavity edges [6] to minimise further post-processing requirements.

4. Conclusion

This work investigated a new cermet and manufacturing techniques to improve mould durability, machinability and surface functionality in ceramic injection moulding (CIM). A work in progress of mould cavity surface repair using simple metals is also presented. A NbC-Ni cermet was evaluated as an alternative mould material to conventional tool steels for CIM. CIM tests with alumina feedstock showed that NbC-Ni exhibits enhanced wear resistance. After 10,000 shots, dimensional wear was reduced by approximately 46% in depth and 86% in width compared to tool steel with negligible changes in surface roughness, demonstrating its suitability for CIM mould inserts.

The machinability (shaping) of NbC-Ni was investigated using hybrid laser-ECM (LECM). Relative to ECM, LECM achieved an improvement in shaping precision by 24% and surface roughness by 30%. This was attributed to laser-assisted current density focusing, enhanced passivation weakening and more uniform dissolution of the multi-phase cermet microstructure.

Femtosecond laser surface texturing on NbC-Ni demonstrated the feasibility of producing 30 µm diameter micro-dimples with controllable aspect ratio and minimal thermal damage. Furthermore, a proof-of-concept mould surface repair strategy combining ECM and ECAM was demonstrated using a single hybrid-EC tool. This approach enables bi-directional process sequences to locally remove and deposit material with a scan layer thickness of ~0.5 µm and minimal postprocessing needs.

Future work will focus on transferring the preliminary findings on laser texturing to a spiral mould for flow length investigations while minimising replication on products, and mould surface repair strategy for depositing advanced repair alloys on mould substrates and assessment of functional properties of the repair layer for adhesion and wear resistance.

Acknowledgment

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