



This is a repository copy of *Role of CO₂ cooling strategies in managing tool wear during the shoulder milling of grade 2 commercially pure titanium.*

White Rose Research Online URL for this paper:

<https://eprints.whiterose.ac.uk/195375/>

Version: Accepted Version

Article:

Proud, L., Tapoglou, N., Wika, K. et al. (2 more authors) (2023) Role of CO₂ cooling strategies in managing tool wear during the shoulder milling of grade 2 commercially pure titanium. *Wear*. 204798. ISSN 0043-1648

<https://doi.org/10.1016/j.wear.2023.204798>

Article available under the terms of the CC-BY-NC-ND licence
(<https://creativecommons.org/licenses/by-nc-nd/4.0/>).

Reuse

This article is distributed under the terms of the Creative Commons Attribution-NonCommercial-NoDerivs (CC BY-NC-ND) licence. This licence only allows you to download this work and share it with others as long as you credit the authors, but you can't change the article in any way or use it commercially. More information and the full terms of the licence here: <https://creativecommons.org/licenses/>

Takedown

If you consider content in White Rose Research Online to be in breach of UK law, please notify us by emailing eprints@whiterose.ac.uk including the URL of the record and the reason for the withdrawal request.



eprints@whiterose.ac.uk
<https://eprints.whiterose.ac.uk/>

Role of CO₂ cooling strategies in managing tool wear during the shoulder milling of grade 2 commercially pure titanium

Leon Proud^{1,4,*}, Nikolaos Tapoglou^{1,2}, Krystian K. Wika³, Chris M Taylor¹ and Tom Slatter⁴

1. Advanced Manufacturing Research Centre (AMRC), The University of Sheffield, Wallis Way, Rotherham, South Yorkshire, U.K, S60 5TZ.
2. Industrial Engineering and Management Department, International Hellenic University, Thessaloniki 57001, Greece
3. Nuclear Advanced Manufacturing Research Centre (Nuclear AMRC), The University of Sheffield, Brunel Way, Rotherham, South Yorkshire, U.K. S60 5WG.
4. Department of Mechanical Engineering, The University of Sheffield, Mappin Street, Sheffield, UK, S1 3JD

* Correspondence: lproud@amrc.co.uk

Abstract: As a result of their high hardness, osseointegration and low chemical reactivity titanium alloys have become an invaluable material species for use in medical applications. Unfortunately, many of the material properties which make titanium alloys desirable biomedical materials, also contribute towards difficulties in tool wear and surface quality during machining. Historically, this has been met via the application of robust metalworking fluid (MWF) strategies, e.g. flood, or high-pressure (HP) soluble oil coolant (emulsion). However, this brings with it its own challenges, not least of which the problem of coolant residue contamination. This study describes efforts to minimise and characterise tool wear during the shoulder milling of grade 2 commercially pure titanium (CP-Ti) by using a novel external supercritical CO₂ (scCO₂) coolant strategy. Whilst the results imply that the sole use of scCO₂ coolant contributes to increased tool wear (relative to emulsion), an optimised strategy of combining scCO₂ with minimum quantity lubrication (MQL) is shown to support up to a 245 % increase in tool life. Despite this, machinability outcomes are found to be highly contingent upon proper selection of trial process variables such as scCO₂ mass flow rate and cutting speed, wherein cutting speeds at or below 115 m/min, and 40 kg/h of scCO₂ is generally shown to be the most efficacious parameters for this material/operation. Ultimately, scCO₂ + MQL is found to be an effective compromise between the cleanliness of scCO₂ and the improved function of emulsion.

1. Introduction

Titanium (Ti) alloys have become the material of choice for a multitude of performance driven engineering applications ranging from biocompatible hip implants to aircraft landing gear [1][2]. Whilst the unique properties of titanium, and its alloys make it an extremely attractive engineering material, they also correspond to markedly poor machinability. In addition to their high hardness, Ti alloys exhibit notably low thermal conductivity (7 to 23 W/mK) wherein, during dry machining temperatures at the seized zone of the chip-tool interface can often reach as high as 1000°C [3]. This high temperature environment corresponds to thermal softening of the tool, and subsequently leads to increased abrasive and adhesive wear.

Although alternative MWF strategies, are becoming increasingly commonplace [4][5], historically the challenge of machining Ti alloys has been met via the use of modest cutting parameters [6] and abundant application of emulsion coolant. From a productivity perspective, whilst this often leads to stable cutting processes, it also follows that machined titanium components are generally associated with an extremely sedate lead time, or, are sold at a price point which accommodates the financial burden of high tooling expenditure. Moreover, by narrowing the available cutting parameters for a given component, the freedom to optimise surface properties and fatigue life [7] is removed. In addition to the limitations of modest feeds and speeds, the use of conventional oil emulsion MWFs brings about its own problems [8], not least of which being the burden of post process cleaning of the components, in addition to marked unsustainability and the time/labour burden associated with coolant upkeep.

In response to these concerns, CO₂ MWF strategies have, in recent times been proposed as an alternative to conventional MWFs, with the outlook of both driving improved productivity outcomes and encouraging further development across each of the four pillars of sustainability (human, social, economic and environmental). Currently, much of the available literature, on CO₂ MWF strategies, has focused upon the machining of Ti-6Al-4V [9]–[12], and although this is logical given that Ti-6Al-4V accounts for 50% of titanium production worldwide [13] (as of April 2020), there remains a multitude of other titanium alloy species, with very different machineabilities [14], which are yet to be documented in a CO₂ MWF context. One such alloy being Grade 2 commercially pure titanium (CP-Ti).

In 1969/70 Uehara and Kumagai [6][7] conducted the first (and only) published cryogenic machining research on CP-Ti. Throughout the two papers the authors employed an external liquid nitrogen (LN₂) supply to the workpiece material during outer diameter (OD) turning and documented its impact on cutting temperature, chip formation mechanics, cutting force, surface roughness and tool wear for three distinct materials, carbon steel, stainless steel (both unspecified alloys) and CP-Ti. In their research Uehara and Kumagai found that despite eliciting larger cutting forces at lower workpiece temperature, tool wear was shown to decrease when LN₂ was employed. The authors noted that this phenomenon was a consequence of workpiece embrittlement at lower temperatures, whilst more recent wisdom would add that the reduction in the wear is also likely a result of the reduction in the thermal softening of the tool and a lesser driving force for interdiffusion.

Whilst these results make a compelling case for the use of cryogenic MWF strategies in this context, the use of LN₂ has its limitations. Although LN₂ coolants are extremely effective at cooling the tool/workpiece during the machining operation, they are not capable of providing lubrication to the cutting zone. CO₂ MWF strategies in contrast are able to support lubricant oil via both single-phase dissolution (scCO₂) or dual phase suspension, and (in the case of the former) as a result of their high outlet pressure (and thus penetration) present as an extremely effective method of lubricant delivery.

Currently several different modalities have been employed to provide both scCO₂ and MQL to the cutting zone. As both cooling capacity and MQL dispersion is shown to decrease exponentially with increased distance from the cutting zone [17], through spindle/tool scCO₂ delivery is expected to generate the best tribological performance outcomes (at least those relevant to tool life). It is thus unsurprising that through tool CO₂ delivery has, as of recently, proven to be the subject of growing research interest [18][19][20]. Despite this, through tool CO₂ delivery is not without limitation. Typically, the retrofitting process is limited to modern machining centres, is generally both extremely expensive and time consuming and requires bespoke tooling strategies. External cooling however, can be moved much more freely around the machine shop, does not require invasive retrofit and allows for common place, off the shelf tooling strategies.

Given the above literature review, the work presented here is dedicated to the development of an effective external CO₂ MWF strategy for the milling of Grade 2 CP-Ti. In doing so this work serves to build upon the foundational research of Uehara and Kumagai, in addition to the growing body of external scCO₂ cooling research [21][22], by establishing the value of localised scCO₂ cooling (as opposed to ubiquitous LN₂ cooling) and lubrication (in this context) in addition to contributing to the growing body of machinability data focused upon cryogenic and CO₂ MWF strategies. This research will thus work towards establishing the suitability of scCO₂ coolants for CP-Ti milling and in doing so, will help to qualify if further investment in through tool delivery is merited for this application.

2. Methodology

2.1 Machine, tooling and materials

During trials, four 100×100×100mm cubic blocks of ASTM-F67 Grade 2 CP-Ti were utilised. The typical composition of this material is presented in Table 1, relative to a Ti-6Al-4V reference.

Experimental work was carried out on a Mori Seiki NV5000 α vertical milling centre, utilising Kennametal® HARVI®II KC643M AlTiN coated 4mm diameter 5-flute solid carbide end mills and a Sandvik Coromant CoroChuck™ 930HD tool holder. The tooling employed during trials was selected owing to its recommended use during both the roughing and finishing of titanium alloys. Houghton Hocut 795 mineral oil/hydrocarbon based soluble coolant (6 to 8% concentration) was employed as flood coolant during the emulsion trials.

Table 1

Nominal compositional comparison of grade 2 CP-Ti [23] and grade 5 Ti-6Al-4V [24].

Workpiece Material	Nominal Composition (wt%)							
	Ti	Fe	O	C	N	H	Al	V
Grade 2 CP-Ti	≥ 98.89	≤ 0.3	≤ 0.25	≤ 0.10	≤ 0.03	≤ 0.015	0	0
Grade 5 Ti-64	87.7 - 91	≤ 0.4	≤ 0.2	≤ 0.08	≤ 0.03	≤ 0.015	5.5 – 6.75	3.5 – 4.5

2.2 Experimental setup

Experimental work followed a Design of Experiments (DOE) approach of initial familiarisation and range selection trials followed by process optimisation. Tool life trials utilised straight line shoulder cuts, employing an arc in radius to prevent shock loading of the tooling during cut entry [25]. Similarly, climb milling was employed throughout, both to optimise tool life outcomes and closely replicate industrial best practice for titanium alloy machining. During range selection trials, the initial machining conditions were chosen based upon the tooling manufacturer’s recommendations for the material and tooling strategy (Table 2). The runout at the tool shank and tip was maintained at less than 1 μ m throughout the machining trials.

Table 2

Machining conditions employed during life trials.

Axial Depth of Cut (mm)	Radial Depth of Cut (mm)	Feed per tooth (mm/tooth)	Uncut Chip Thickness (mm)	CO ₂ Set Pressure (MPa)
6	0.4	0.03	0.0095	≈13.8

Tool wear was measured during experimentation initially after each pass, and thereafter at two minute intervals. Wear measurements were taken via the use of a Leica DMS 1000 optical microscope. A tool life of 60 minutes was targeted for the emulsion coolant trials in accordance with common industrial practice for resource efficient use of solid carbide end-mills. Milled surface roughness was measured post process using a focus-variation type non-contact 3D profilometer (Bruker Alicona Infinite FocusSL) with Measuresuite data acquisition software.

2.3 scCO₂ mode of delivery

Liquid CO₂ was stored in an ~510kg Manifold Cylinder Pallet (MCP) and delivered to the cutting tool as a supercritical fluid via the Pure-Cut+® scCO₂ system manufactured by Fusion Coolant Systems (Michigan, USA). The trials made use of a novel external mode of delivery (Figure 1), wherein the CO₂ outlet was connected to small stainless steel (304L) nozzle which in turn directs the fluid flow through a sapphire glass aperture housed within an M4 grub screw. The nozzle was magnetically fixtured to the machining centre and positioned ahead of the leading edge of the tool prior to entering cut. In order to assure repeatability, a datum was marked on both the machining centre and magnetic stand and aligned

prior to each block of cuts. During initial operation for setup familiarisation it was noted that there was a strong negative correlation between tool life and distance between the nozzle outlet and tool. This is a consequence of both a reduction in cooling capacity and increased dispersion of MQL inhibiting effective cooling and lubrication [17]. As a result of this finding, the CO₂ outlet was placed close to the tooling throughout the duration of the experimental work (~10mm).

During trials, the CO₂ mass flow rate was adjusted by incrementally changing the size of the outlet aperture. Flow rates ranging from 15kg/h to 55kg/h were selected in order to generate a centre point mass flow rate (35kg/h) which was consistent with CO₂ flow rates which have previously been employed elsewhere in the literature [20]. Trials were conducted both with and without MQL. When MQL was employed, Blaser Vascomill MMS FA2 MQL oil (fatty alcohol based) was selected owing to its recommended use during titanium alloy machining and the absence of mineral oil (a condition for sustainability). In order to assure that changing performance outcomes were being driven by CO₂ flow rate, and to remain consistent with the recommendations of Fusion Coolant Systems, CO₂ pressure was set at approximately 13.8MPa and a target MQL flow rate of 0.5 ml/min was employed. Within the finite scope of the work, the MQL oil type and its flowrate were held constant and not investigated.

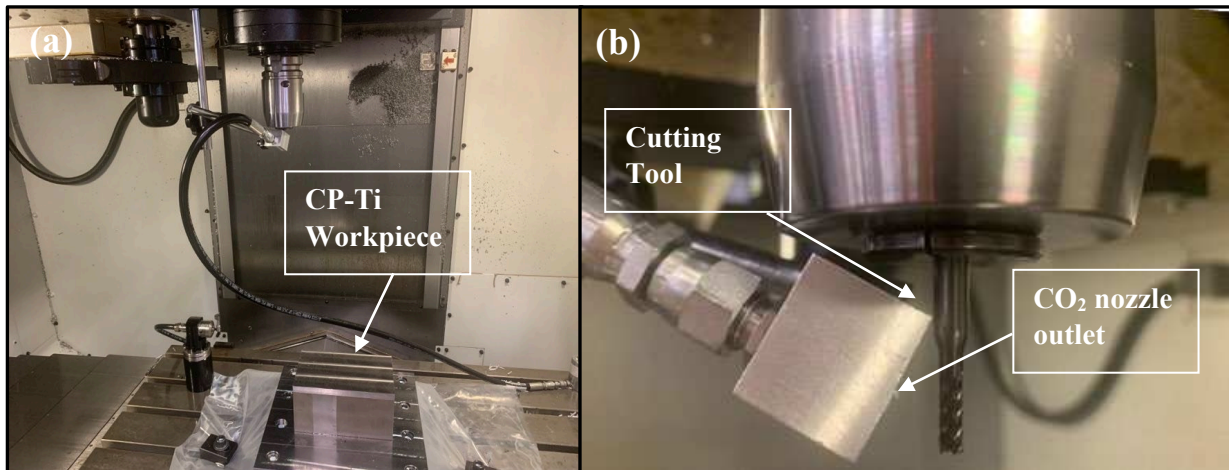


Figure 1 – (a) Machining trial and (b) scCO₂ nozzle setup

3. Results and discussion

3.1 Initial range selection trials

Given the goal of sustainable development, and as the medical sector demands coolant residue free products, the initial focus of this experimental work was to explore if a workable scCO₂ only MWF strategy could be established. Further to this point, much of the early familiarisation work employed an extremely modest CO₂ flow rate (of 15 kg/h), this decision was made with the outlook of optimising sustainability and cost efficiency. In doing so, early machining trials led to near instantaneous tool failure when scCO₂ was employed in isolation and as such, screening was conducted considering additional CO₂ flow rates, feed rates and cutting speeds (Table 3).

Table 3

Life results of scCO₂ screening trials.

Coolant	Flow Rate (kg/h)	Cutting Speed (m/min)	Feed per tooth (mm/tooth)	Tool Life (s)	Material Removed (cm ³)
scCO ₂	15	100	0.03	24.1	1.2
scCO ₂	15	100	0.04	30.2	1.9

scCO ₂	15	100	0.02	12.1	0.4
scCO ₂	35	100	0.03	24.1	1.2
scCO ₂	55	100	0.03	24.1	1.2
scCO ₂	15	130	0.03	6.2	0.4
scCO ₂	15	70	0.03	46.0	1.5
scCO ₂	15	50	0.03	2509.3	59.9
Dry	0	50	0.03	16.1	0.4

The above screening trials show that the performance of the scCO₂ only strategy is relatively insensitive to manipulation of process variables. Changes to feed rate, CO₂ mass flow rate (in isolation) and cutting speed were generally unable to bring about significant changes to machinability outcomes, such that, tool life below one minute was achieved at all but one of the trialled cutting conditions. The exception was at 50m/min which returned a tool life in excess of 40 minutes. These findings are consistent with prior milling trials conducted on Ti-6Al-4V [26] which showed that, devoid of lubrication, scCO₂ cooling was unable to generate tool life outcomes comparative to emulsion. The performance was however far superior to dry cutting, with scCO₂ offering cooling and convection (cut material removal) benefits as well as a modified gas environment for the cutting zone.

It is worthwhile to note that whilst changes to flow rate failed to bring about significant changes in life outcomes at 100m/min, this may not be the case at more modest cutting speeds. It is reasonable to assume that some minimum tool life threshold must be reached before flow rate optimisation would realise a tribological benefit to the process. In this sense, there may be scope to further optimise the life outcomes of the 50 m/min trial, however this was deemed out of scope owing to the necessity of a 50% reduction in cutting speed and thus Material Removal Rate (MRR) relative to the tool supplier's recommended parameter set. Generally, this makes the scCO₂ only strategy untenable for industrial adoption in scenarios where a significant machining element is present in manufacture (i.e., the prototyping of medical implants). However, in a context wherein coolant use is not acceptable, or, where the time burden of post process cleaning exceeds the increase in cycle time of the more sedate machining operation (i.e. limited finish milling of formed surgical tools), a scCO₂ only coolant strategy retains some utility.

Nonetheless, given that low cutting speeds are not desirable, a further machining trial was conducted with scCO₂ + MQL at 100 m/min (15 kg/h) in addition to a corresponding emulsion trial at the same cutting speed (figure 2).

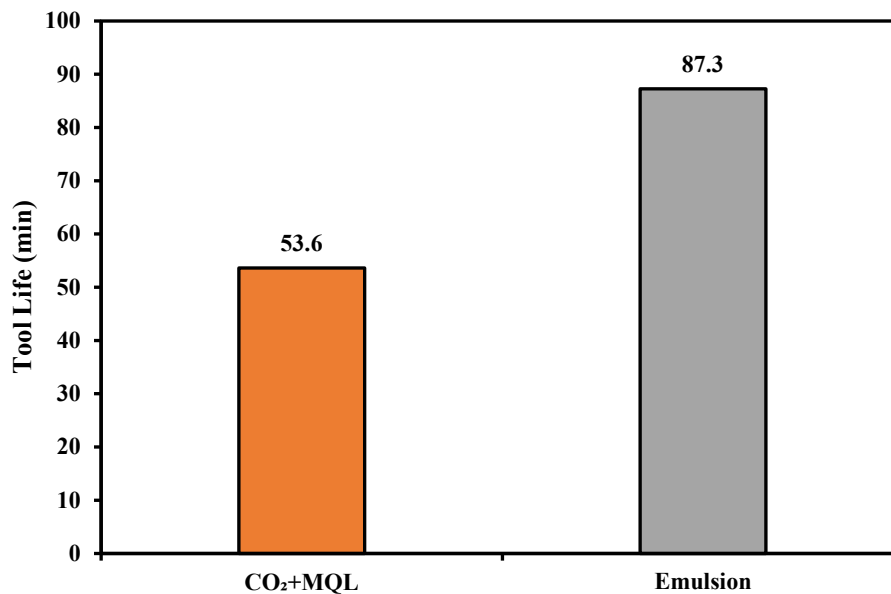


Figure 2 – A graph to show the variation in tool life with MWF strategy during screening trials.

At 100 m/min the scCO₂ + MQL (53.6 min) strategy provided 61 % of the tool life of the emulsion coolant strategy (87 min). Whilst this tool life is significantly lower than was achieved with emulsion coolant, life outcomes for the first time approach the target life of 60 min an observation which serves to validate the experimental methodology. With this in mind and given the inadequate life outcomes obtained during the scCO₂ only trials, the corresponding optimisation works were focused upon improving the performance of the hybridised, scCO₂ + MQL coolant strategy towards something approaching, or in excess of that obtained during emulsion, flood cooling. Moreover, given the target tool life of 60 minutes for the emulsion baseline, cutting speed was increased in subsequent trials.

3.2 Optimisation trial

During optimisation, two primary variables were considered, cutting speed and CO₂ mass flow rate. Cutting speed was selected for analysis as it is an effective means of modulating heat generation in the cut, which in turn, makes it possible to qualify how effective the scCO₂ cooling strategy is at dispelling heat from the shoulder milling process. With this in mind, speeds of 115 m/min and 130 m/min were chosen in addition to CO₂ mass flow rates of 15 kg/h, 35 kg/h and 55 kg/h. In order to vary the mass flow rate of CO₂, nozzle apertures of 0.2mm, 0.4mm and 0.6mm were used respectively. All other experimental variables (MQL flow rate, nozzle location etc.) were maintained at a constant level.

Throughout tool life testing, all tooling was found to fail catastrophically without the onset of significant flank wear, regardless of coolant strategy, CO₂ flow rate or changes to cutting parameters, the mechanistic rationale for which is explored in Section 3.3. Given this rapid onset of wear, tooling was taken to true failure during machining trials, where true failure is defined as the point at which the cutting edge is damaged to an extent such that it is unable to remove material safely or effectively.

During experimentation, the tool life obtained via the hybridised scCO₂ + MQL strategy was shown to be highly contingent upon both cutting speed and CO₂ mass flow rate (Figure 3). At 115 m/min, two of the three CO₂ flow rates were able to return improved tool life relative to the emulsion coolant strategy, where, at 35 kg/h, life was shown to increase by 245 % relative to the flood coolant strategy, whilst 55 kg/h contributed to an increase in tool life of 62 %. In contrast, at the 15 kg/h flow rate, the scCO₂ +

MQL strategy was unable to produce comparative life outcomes relative to the corresponding emulsion trial. These results are in broad agreement with the work of Tapoglou et al. [26] for the inserted milling of Ti-6Al-4V where at 100 m/min scCO₂ on its own performed poorly, whilst scCO₂ + MQL outperformed emulsion coolant significantly, achieving more than double the tool life.

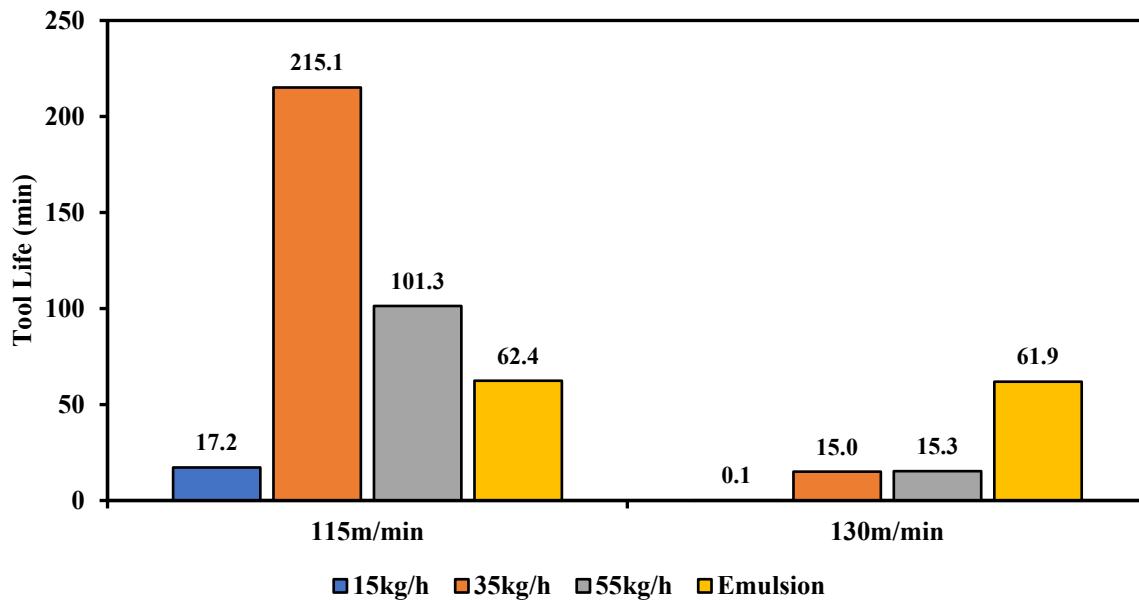


Figure 3 – A graph to compare the tool life outcomes of scCO₂ + MQL (at various flow rates) with emulsion. The plot considers two cutting speeds.

As the cooling capacity of the CO₂ MWF strategy follows the mass flow rate of CO₂, the increase in tool life which is observed at 35 kg/h relative to 15 kg/h is intuitive. Despite this, the reduction in life from 35kg/h to 55 kg/h would seem to suggest that there exists an optimal mass flow rate of CO₂ for a given operation, beyond which performance suffers. This is likely a consequence of excess cooling proximally and thermally hardening the near-surface workpiece material, leading to both elevated load and tool abrasion during the cut. Similar near-surface grain refinement and hardening phenomena has previously been observed during scCO₂ assisted milling of Ti-6Al-4V by Khosravi et al. [19].

At 130 m/min the performance of the emulsion coolant strategy exceeded that of the hybridised approach at all three trialled mass flow rates, wherein near instantaneous tool failure occurred at 15 kg/h, whilst the higher mass flow rates corresponded to markedly similar life outcomes of approximately 25% that of emulsion. In this regard it is noteworthy to remark that whilst the higher CO₂ mass flow rate contributed to a significantly prolonged tool life from 15 kg/h to 35 kg/h, further improvements again were not realised beyond this point.

With regard to tool life outcomes, the hybridised CO₂ coolant strategy is far more sensitive to changes in cutting speed than the equivalent emulsion trials. The mechanistic rationale for this is hypothesised to be a consequence of the following variables:

1. Although both emulsion and scCO₂ + MQL MWF strategies have mass flowrates and heat carrying capacities of similar magnitude, both flow temperature, and their respective area coverage (which flows over the tool and cutting zone) differ. As a comparison, scCO₂ flow is directed into a narrowly focussed, gaussian-type jet, whilst soluble oil coolant is much more ubiquitously present in the cut.
2. At more aggressive cutting speeds, the spatial focus and lower temperature (the snow phase being at -78.5 °C) of the scCO₂ jet will lead to greater thermal cycling (repeated frictional

heating followed by CO₂ cooling) of the cutting edges as they move in and out of cut with each revolution of the tool.

3. The spatial focus of the scCO₂ jet may lead to inferior chip clearance compared to emulsion coolant.

In this regard, emulsion proved to be the most suitable option in this test scenario for achieving high MRR requirements.

3.3 Wear mechanisms and failure modes

Throughout machining trials no notable abrasive flank wear was observed at any point during tool life. Rather, in the absence of flank wear tools failed catastrophically via (most frequently) an adhesive or (rarely) fatigue mechanism. Although limited data exists on the machining of CP-Ti, adhesive wear has been identified previously during the machining of Ti-6Al-4V [27]. When adhesive wear was present on the tool, two failure modalities were observed. In both adhesive modalities, wear begins with the rapid build-up of agglomerated workpiece material on the cutting edge of the tool. This build-up has a deleterious effect on the efficiency of the tools cutting edge (owing to the size effect) which in turn leads to an inability to plastify the workpiece material, and efficiently form chips. Consequently, due to the inability to make a cutting action, the tooling begins to rub/plough the workpiece material. This in turn both generates significant heat and places a localised bending moment on the cutting edge, the combined effects of which often leads to catastrophic thermomechanical failure (Figure 4a). In general, this failure mode leads to fracture of the tool tip and/or breakage of one or more of the teeth.

Less frequently, chip adhesion is localised to one flute of the tool creating a blockage (Figure 4b). In this scenario the cutting capacity of the blocked flute is removed which leads to additional load being placed upon the remaining flutes. This in turn contributes to dynamic instability and poor surface finish, and as such, a tool with a blocked flute should be considered to have failed. These adhesive wear mechanisms occurred in each of the machining trials, with the exception of the scCO₂ + MQL trial conducted at 115 m/min with a 35 kg/h flow rate wherein the tool tip chipped after 3.6 hours into the cut (Figure 4c). In this case, given the prolonged life of the tool, and the efficiency of the coolant strategy, chipping was likely a consequence of both thermal and mechanical fatigue loading, however, this should be explored further.

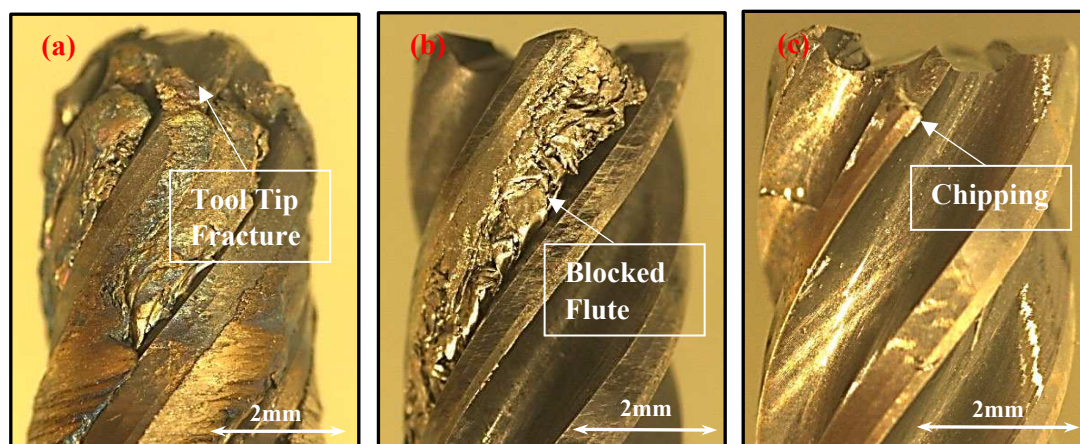


Figure 4 – Tool wear micrographs displaying: (a) adhesive failure, (b) adhesive failure with a flute blockage, (c) tool tip chipping.

In cases of adhesive wear, tool failure generally was shown to follow shortly after a loss of the tools TiAlN coating. The occurrence of delamination is evidenced by both the blistering and dulling of the tool at the cutting edge (Figure 5). Adhesive failure thereafter is an expected result given that coating materials are chosen in part to function as a diffusion barrier between the cemented carbide substrate and the chip material. The tool tip fracture which then often follows adhesive failure is likely influenced

by a combination of the sharp (and thus weaker) cutting edge radius, and small tool geometry. As such, given that tool life was shown to be prolonged significantly via the scCO₂ + MQL MWF strategy (relative to scCO₂ only, and, situationally emulsion coolant), it follows that either chip evacuation, or coating retention is likely improved with the addition of MQL.

Whilst the above makes it clear that the tool coating plays a valuable role in prolonging tool life in this context, it remains to be seen if the coating selected for experimentation is the most appropriate option for CP-Ti. It is reasonable to suggest that given the much higher ductility of CP-Ti relative to Ti-6Al-4V, and similarly, much lower hardness, the most appropriate coating material for each specific alloy may differ. Further to this point, it is likely that the development resources allocated to this tool's design was conducted with Ti-6Al-4V in mind given its frequent use in industry. As such, it is apparent that coating choice should be considered for future CP-Ti machining projects.

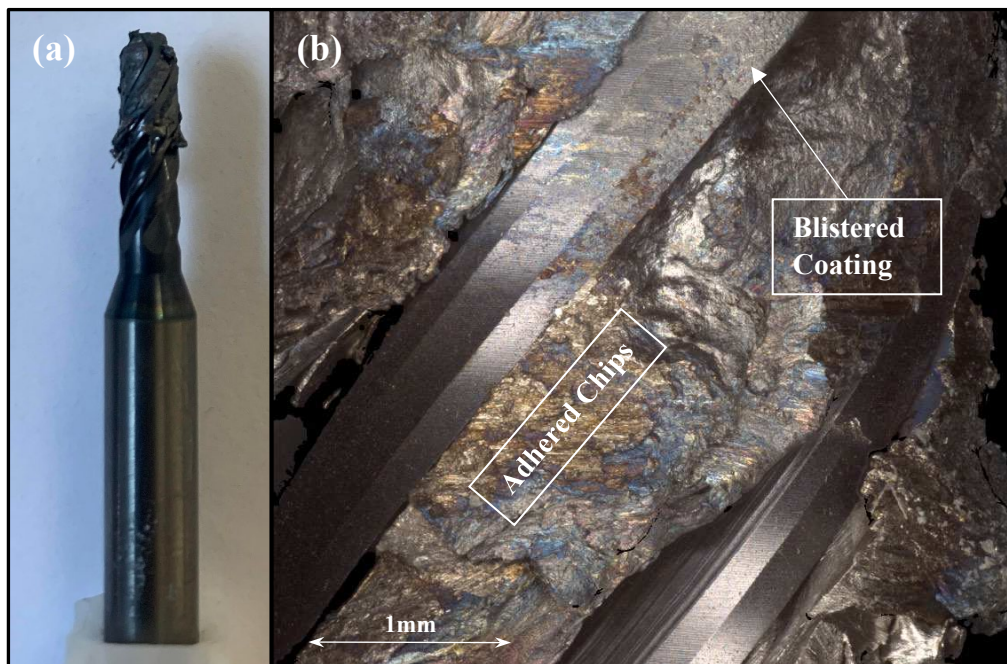


Figure 5 – (a) Micrograph of adhesive failure with (b) the corresponding macro scale image of the tool.

3.4 Surface Roughness

In order to measure surface roughness, milled steps were made with each MWF strategy, flow rate and cutting parameter combination (figure 6). Thereafter, samples of the vertical machined surface (wall) were first sectioned via the use of an electrical discharge machining process (EDM) and thereafter cut to final dimensions on a Secotom-50 CNC machining centre, with a MetPrep type T5 cut-off wheel. In scenarios where the tooling failed during the cut, samples were removed at a point prior to the onset of tool failure. Mean surface roughness (R_a) was calculated based upon three repeat measurements taken equidistantly across the wall of the machined surface.

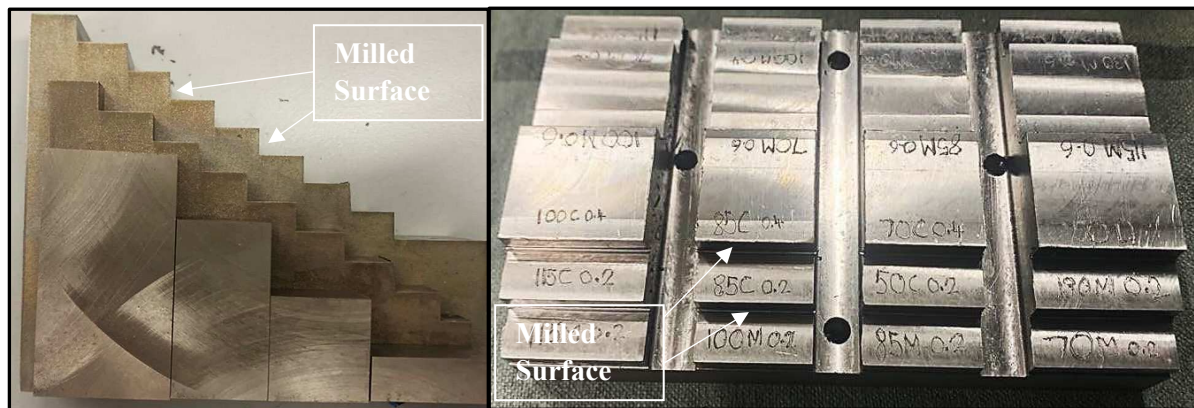


Figure 6 – Images of the stepped workpiece geometry used to measure surface roughness (pre and post sectioning).

Samples were taken at 15 m/min cutting speed intervals from 70 to 130m/min inclusive, with CO₂, CO₂ + MQL and emulsion coolant strategies (Figure 7). In the case of the CO₂ and hybridised coolant strategies, sample were made with each of the CO₂ mass flow rates employed during machining trials (15 kg/h, 35 kg/h and 50 kg/h).

During analysis, surface roughness readings ranged from 0.26 to 1.96 μm , whilst the majority of samples tested returned an R_a value of less than 1 μm . At a cutting speed of 115 m/min all but one of the scCO₂ and scCO₂ + MQL strategies generated a lower surface roughness than the corresponding emulsion coolant strategy (0.98 μm). Of the 115 m/min samples, the 35 kg/h scCO₂ + MQL trial led to the lowest surface roughness (0.5 μm) in contrast to the 55 kg/h scCO₂ only trial which corresponded to the largest R_a value (1.96 μm). Interestingly, the 35 kg/h scCO₂ + MQL trial also corresponded to the longest tool life at the 115 m/min cutting speed, implying that stable cutting conditions have been reached.

When emulsion coolant was employed, sub 0.5 μm surface roughness was achieved at cutting speeds of 70, 100 and 130 m/min, whilst markedly higher roughness was observed at both 85 and 115 m/min. Although this was not the case for each MWF strategy, the 130 m/min cutting speed samples generally were of lower, or similar surface roughness to the other trialled speeds. This finding supports current industrial best practice of using higher cutting speeds when undertaking finishing operations.

Whilst the two lowest values of surface roughness were achieved at a flow rate of 15kg/h with the scCO₂ only (0.26 μm), and scCO₂ + MQL (0.32 μm) strategies respectively, it is important to note that these MWF strategies lead to unsustainably low tool lives, and as such, are not viable for industrial adoption. In these cases, it is possible that the notably low surface roughness is a product of the cutting tool burnishing the machined surface as a consequence of ineffectual cutting, however this requires further evidence.

Of the MWF strategies which have been shown to generate industrially tenable tool lives in excess of a few minutes, the best performing strategy, from a surface roughness perspective is the 100 m/min emulsion trial (0.39 μm), whilst the best hybrid approach is the 70 m/min, 15 kg/h strategy (0.48 μm). It is thus noteworthy that a large percentage of the scCO₂ and scCO₂ + MQL strategies are not available for roughness optimisation as a result of the associated inadequate tool life. As a consequence, emulsion remains the best practice for robustly optimising surface roughness in this context.

Further, it is possible that when milling with unfavourable cutting conditions, machined surface roughness could be artificially high due to premature tool wear changing the geometric profile of the cutting edges, which in turn, leads to an unfavourable kinematic cut profile on the material surface. Equally, it is worthwhile to note, that when tenable cutting conditions are employed, higher CO₂ flow rates are, in general shown to contribute towards increased surface roughness. Given the findings of Khosravi and colleagues [19], one potential justification for this observation could be lower cutting zone temperatures leading to grain pull-outs from the potentially, harder, more brittle material regime at the surface. However, this should be validated by high resolution imaging of both the surface, and subsurface, in addition to nano-hardness testing, prior to drawing any conclusions.

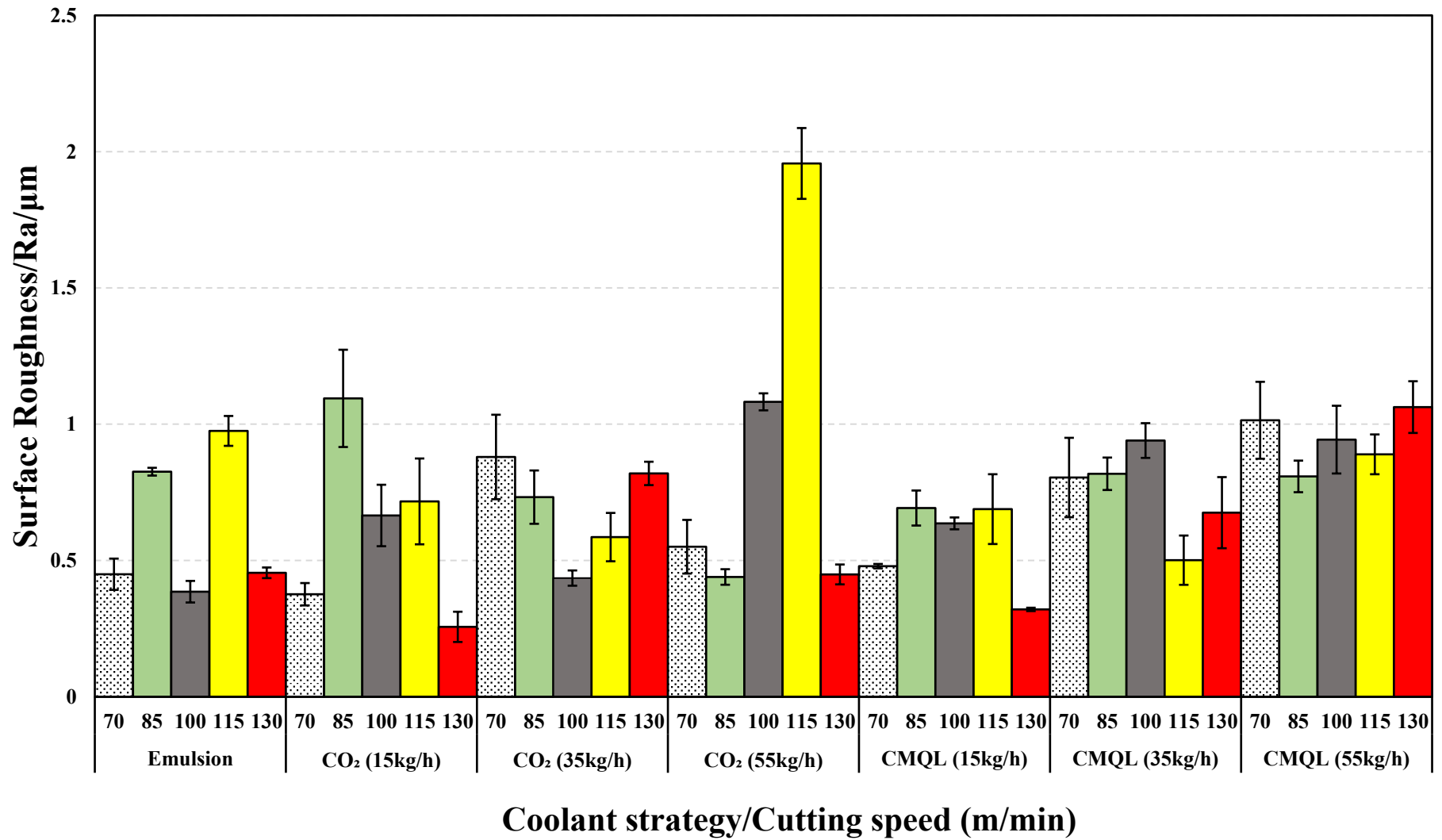


Figure 7 – A comparison of the surface roughness outcomes achieved via various MWF strategies at a range of cutting speeds. CMQL denote scCO₂ + MQL and CO₂ denotes scCO₂ in isolation.

4. Conclusions

The conclusions which should be drawn from this work are as follows:

1. Despite retaining some function at lower cutting speeds (< 50 m/min), the use of external scCO₂ in isolation when machining CP-Ti generally leads to marked reductions in tool life relative to emulsion coolant.
2. In contrast, given appropriate setup and parameter selection, a hybridised external scCO₂ + MQL approach is able to generate a 245 % increase in tool life (at 115 m/min) relative to conventional MWF strategies.
3. The tool wear performance outcomes of scCO₂ assisted cooling and lubrication strategies are shown to worsen significantly (relative to emulsion coolant) at inappropriate CO₂ mass flow rates, elevated cutting speeds (in excess of 115 m/min) and inadequate nozzle proximity (to the tool).
4. At 115m/min, given the reduction in tool life which is observed as CO₂ mass flow rate is increased from 35 kg/h to 55 kg/h, overcooling has been identified as a potential problem. As such, the relationship between a given material's properties and the in-process temperatures should be considered when selecting an appropriate mass flow rate of CO₂.
5. Adhesive failure mechanisms are identified as a cause for concern during small diameter solid carbide shoulder milling of CP-Ti.

It is acknowledged that the MQL oil type, flowrate and nozzle design were not factors within this work, and that there remains significant scope to optimise these variables in this context. In addition, the potential role of thermal cycling and chip clearance warrants further consideration.. Finally, given the clear utility of external scCO₂ + MQL application, future investment in integrated, through tool scCO₂ cooling is supported in this context.

Author Contributions: Conceptualization, L.P. and N.T.; methodology, L.P., N.T. and K.W.; investigation, L.P. and T.S. ; writing—original draft preparation, L.P.; writing—review and editing, L.P., N.T, K.W., C.T. and T.S.; visualization, L.P., N.T. and K.W.; supervision, K.W., N.T and T.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the EPSRC Industrial Doctorate Centre in Machining Science (EP/L016257/1).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Conflicts of Interest: The authors would like to declare no conflicts of interest.

References

- [1] M. Semlitsch, "Titanium alloys for hip joint replacements," *Clin. Mater.*, vol. 2, no. 1, pp. 1–13, 1987.
- [2] J. D. Cotton *et al.*, "State of the Art in Beta Titanium Alloys for Airframe Applications," *Jom*, vol. 67, no. 6, pp. 1281–1303, 2015.
- [3] R. Li and A. J. Shih, "Tool Temperature in Titanium Drilling," *J. Manuf. Sci. Eng.*, vol. 129, no. 4, pp. 740–749, Apr. 2007.
- [4] M. Dogra *et al.*, "State of the art review on the sustainable dry machining of advanced materials for multifaceted engineering applications: progressive advancements and directions for future prospects" *Mater. Res. Express*, 2022.
- [5] V. S. Sharma, M. Dogra, and N. M. Suri, "Cooling techniques for improved productivity in turning," *Int. J. Mach. Tools Manuf.*, vol. 49, no. 6, pp. 435–453, 2009.
- [6] R. Komanduri and B. F. Von Turkovich, "New observations on the mechanism of chip formation when machining titanium alloys," *Wear*, 1981.
- [7] A. Cox, S. Herbert, J. P. Villain-Chastre, S. Turner, and M. Jackson, "The effect of machining and induced surface deformation on the fatigue performance of a high strength metastable β titanium alloy," *Int. J. Fatigue*, vol. 124, no. February, pp. 26–33, 2019.
- [8] L. Proud, N. Tapoglou, and T. Slatter, "A Review of CO₂ Coolants for Sustainable Machining," *Metals (Basel)*, vol. 12, no. 2, 2022.
- [9] K. Wika, O. Gurdal, P. Litwa, and C. Hitchens, "Influence of supercritical CO₂ cooling on tool wear and cutting forces in the milling of Ti-6Al-4V," *Procedia CIRP*, vol. 82, no. ii, pp. 89–94, 2018.
- [10] N. S. Ross, P. T. Sheeba, M. Jebaraj, and H. Stephen, "Milling performance assessment of Ti-6Al-4V under CO₂ cooling utilizing coated AlCrN/TiAlN insert," *Mater. Manuf. Process.*, vol. 36, no. 1, pp. 1–15, 2021.
- [11] M. I. Sadik, S. Isakson, A. Malakizadi, and L. Nyborg, "Influence of Coolant Flow Rate on Tool Life and Wear Development in Cryogenic and Wet Milling of Ti-6Al-4V," *Procedia CIRP*, vol. 46, pp. 91–94, 2016.
- [12] Q. An, C. Cai, F. Zou, X. Liang, and M. Chen, "Tool wear and machined surface characteristics in side milling Ti6Al4V under dry and supercritical CO₂ with MQL conditions," *Tribol. Int.*, vol. 151, no. May, p. 106511, 2020.
- [13] Supra Alloys, "Titanium Grade Overview," *Supra Alloys*, 2020. [Online]. Available: <http://www.supraalloys.com/titanium-grades.php>. [Accessed: 21-Apr-2020].
- [14] P. J. Arrazola, A. Garay, L. M. Iriarte, M. Armendia, S. Marya, and F. Le Maître, "Machinability of titanium alloys (Ti6Al4V and Ti555.3)," *J. Mater. Process. Technol.*, vol. 209, no. 5, pp. 2223–2230, 2009.
- [15] K. Uehara and S. Kumagai, "Chip Formation, Surface Roughness and Cutting Force in Cryogenic Machining," *Annals of the C.I.R.P.*, vol. 17, pp. 409–416, 1969.
- [16] K. Uehara and S. Kumagai, "Characteristics of tool wear in cryogenic machining," *CIRP Ann.*, vol. 18, no. 1, pp. 273–277, 1970.
- [17] D. Gross, M. Appis, and N. Hanenkamp, "Investigation on the productivity of milling ti6al4v with cryogenic minimum quantity lubrication," *MM Sci. J.*, vol. 2019, no. November, pp. 3393–3398, 2019.

- [18] D. A. Stephenson, S. J. Skerlos, A. S. King, and S. D. Supekar, "Rough turning Inconel 750 with supercritical CO₂ -based minimum quantity lubrication," *J. Mater. Process. Tech.*, vol. 214, no. 3, pp. 673–680, 2014.
- [19] J. Khosravi, B. Azarhoushang, M. Barmouz, R. Bösinger, and A. Zahedi, "High-speed milling of Ti6Al4V under a supercritical CO₂ + MQL hybrid cooling system," *J. Manuf. Process.*, vol. 82, no. July, pp. 1–14, 2022.
- [20] K. K. Wika, P. Litwa, and C. Hitchens, "Impact of supercritical carbon dioxide cooling with Minimum Quantity Lubrication on tool wear and surface integrity in the milling of AISI 304L stainless steel," *Wear*, vol. 426–427, no. January, pp. 1691–1701, 2019.
- [21] F. Zou, B. Zhong, H. Zhang, Q. An, and M. Chen, "Machinability and Surface Quality During Milling CFRP Laminates Under Dry and Supercritical CO₂-Based Cryogenic Conditions," *Int. J. Precis. Eng. Manuf. - Green Technol.*, vol. 9, no. 3, pp. 765–781, 2022.
- [22] L. Tu *et al.*, "Machinability improvement of compacted graphite irons in milling process with supercritical CO₂-based MQL," *J. Manuf. Process.*, vol. 68, no. PA, pp. 154–168, 2021.
- [23] Allvac, "Titanium Grade 2," *MatWeb*, 2022. [Online]. Available: <https://www.matweb.com/search/DataSheet.aspx?MatGUID=24293fd5831941ec9fa01dce994973c7&ckck=1>.
- [24] Allvac, "Titanium Ti-6Al-4V (Grade 5), Annealed Bar," *MatWeb*, 2022. [Online]. Available: <https://www.matweb.com/search/DataSheet.aspx?MatGUID=10d463eb3d3d4ff48fc57e0ad1037434>.
- [25] Sandvik Coromant, "8 tips on machining titanium and its alloys," 2019. [Online]. Available: https://www.sandvik.coromant.com/gb/news/technical_articles/pages/8-tips-on-machining-titanium-and-its-alloys.aspx. [Accessed: 27-Sep-2022].
- [26] N. Tapoglou, C. Taylor, and C. Makris, "Milling of aerospace alloys using supercritical CO₂ assisted machining," *Procedia CIRP*, vol. 101, pp. 370–373, 2021.
- [27] A. Zahedi, "High-speed milling of Ti6Al4V under a supercritical CO₂ + MQL hybrid cooling system," *J. Manuf. Process.*, vol. 82, no. July, pp. 1–14, 2022.