

Comprehensive analysis of cutting temperature, tool wear, surface integrity and tribological properties in sustainable milling of Ti6Al4V alloy: LN₂, nanofluid and hybrid machining

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ARTICLE INFO

Keywords:

Sustainable milling

Ti6Al4V alloy

Nanofluids

Tool wear

Surface integrity

ABSTRACT

Despite being expensive and difficult to process, the Ti6Al4V alloy is a vital component for crucial industries. To improve its machinability and accomplish sustainable production, environmentally friendly cooling and lubricating agencies are used. Studies on the machinability of the alloy are still necessary because of its unique features and significance in vital industries like aerospace, defense, and medicine. Therefore, this investigation focuses on tool wear, temperature, and surface integrity for sustainable milling Ti6Al4V under various machining environments, i.e., dry, pure-MQL, LN₂, hBN, CuO-doped nanofluids, and hybrid methods. The produced nanofluids' thermophysical and rheological characteristics were examined in the study's initial phase. Because of the results from the first stage, machining performance indicators were assessed in the subsequent milling experiments. As a result, CuO-doped nanofluids gave improved results in terms of viscosity and pH. The best results obtained in the LN₂ + CuO hybrid cooling lubrication environment in important machinability outcomes such as tool wear and surface integrity were attributed to the rheological properties of CuO-doped nanofluid and its harmonious cooperation with LN₂-cryogenic cooling.

1. Introduction

Today, the concept of sustainability has become much more important due to reasons such as environmental concerns, a decrease in non-renewable resources, stricter legislation and inflated energy costs, and increasing consumer preference for environmentally friendly products [1]. The efficient use of natural resources and their transfer to future generations, which has become an important concern in all sectors, also has an important place on the agenda of the aviation industry. The significant increase in annual passenger traffic in recent years means more fuel consumption and higher levels of greenhouse gas emissions, deepening concerns about this sector [2]. Therefore, the long-term goals of aviation research councils include the creation of advanced aircraft designs that comply with environmental standards [3]. The basis of

improved design for energy economy is the production of lighter aircraft. However, lighter materials alone will not be sufficient for this. Strength/weight ratio is one of the most important criteria in the selection of aviation materials. In other words, in addition to being light, the materials to be used in aircraft must also have good other properties such as high strength/weight ratio, relatively low modulus, high yield strength, toughness and excellent corrosion resistance [4]. Although it is difficult to find all of these features in a single material, Titanium and its alloys can meet expectations to a significant extent. For this reason, it is a widely preferred material in critical areas of aircraft [4,5]. The most common titanium alloy is Ti6Al4V, which belongs to the $\alpha + \beta$ alloy group and accounts for more than 50 % of titanium alloy production [6]. Ti6Al4V alloy is widely used in many fields, especially medicine, aviation, and maritime, due to its excellent properties. However, these

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<https://doi.org/10.1016/j.jmapro.2024.09.120>

Received 24 June 2024; Received in revised form 28 September 2024; Accepted 30 September 2024

Available online 3 October 2024

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superior properties of Ti6Al4V alloy cause some major problems in its shaping. For example, due to the high melting point, the equipment used for melting and casting is expensive, and their operations are complex [7]. This significantly prevents it from being shaped by the casting method. A similar situation is seen in shaping with machining. Ti6Al4V alloy is in the class of difficult-to-machine materials due to its low thermal conductivity, low modulus of elasticity and high chemical reactivity with most cutting tool materials [8]. In addition, the high temperature resistance and hardness of these alloys require high cutting forces. This causes deformation in the cutting tool during the cutting process. The high dynamic cutting strength during the cutting process causes abrasive sawtooth edges, resulting in notches in the tool [9]. Therefore, the use of cutting fluid is advantageous in many aspects during the machining of difficult-to-cut materials such as Ti6Al4V.

Cutting fluids are frequently used in metal cutting operations because they extend the life of the cutting tool, help with heat transfer by providing appropriate cooling and lubrication, reduce friction at the tool-chip interface, and evacuate the chip from the cutting area [10]. However, the cutting fluids used today are mostly mineral-based and cause adverse effects on the environment, employee health, and production costs [11]. Very serious health problems such as acne, dermatitis, hypersensitivity pneumonia, asthma, and upper respiratory tract disease occur in workers exposed to cutting fluids for a long time. Workers who inhale aerosols of cutting fluids face health problems such as throat, nose, eye, and lung irritation [12]. Therefore, both manufacturers and researchers are working on alternatives to achieve environmentally and employee-friendly processing methods. Among the alternatives put forward here, minimum quantity lubrication (MQL), nanofluid, and cryogenic cooling stand out.

The MQL system works on the principle of spraying a much lower amount of cutting fluid (approximately 1/10000) into the cutting area with compressed air compared to conventional cooling fluid [13]. In this system, cutting oil and compressed air are mixed at one point, atomized in a nozzle, and then delivered to the cutting point in the form of an aerosol spray [14]. However, the MQL system mainly performs better in light and medium difficulty machining conditions [15]. It is discussed in the literature that the MQL system is not sufficient in heavy machining conditions of difficult-to-machine materials such as super alloys, hardened steels and titanium [16–18]. In such a case, there are several alternative methods, one of which is the use of nanofluid. Nanofluids are fluids obtained by adding nanoparticles of 1–100 nm size into a base liquid such as oil and water [19]. Thanks to the advanced thermophysical properties of nanofluids, it can better discharge heat in the cutting zone. By balancing the heat in the cutting zone, the negative effects of high cutting temperature on the workpiece, cutting tool, and machined surface are reduced. Thus, both less fluid and higher efficiency are achieved compared to the flood cooling condition. Previous studies also show that nanofluids provide better results than both flood and pure MQL [20–22].

One of the alternative cooling methods used to eliminate the disadvantages of the MQL system is cryogenic cooling. Inconsistencies regarding initial temperatures in cryogenic cooling have not yet been fully resolved. According to most research and standards organizations, cryogenic cooling is valid for temperatures of 123 K or below. However, the National Institute of Standards and Technology accepts less than 93.15 K as the cryogenic temperature, while the Cryogenic Society of

America considers 120 K as the initial temperature [23]. On the other hand, the process in which liquid nitrogen (LN₂) is referred to as cryogenic cooling. Liquid nitrogen at –196 °C is a commonly used coolant in cryogenic processing industries.

Ravi and Gurusamy [24] compared dry, wet, and LN₂ when machining AISI D2 steel. According to the experimental results, LN₂ offers better processing efficiency. In addition, eliminating the disadvantages of the MQL system in terms of cooling by using LN₂ is also a noteworthy research topic. Sivaiah and Chakradhar [25] compared LN₂ based cryogenic cooling with dry, wet, and pure MQL in the machining of 17–4 steel. Researchers have claimed that LN₂ provides more beneficial results. Chetan et al. [26] compared the effect of cryogenic cooling (i.e., LN₂) and Al₂O₃ added nanofluid on machining performance in turning Nimonic 90 alloy. According to the experimental results, LN₂-based cryogenic cooling offered more promising results. The possibility that the combined use of nanofluids and LN₂ may provide better results in terms of both lubrication and cooling means the emergence of an important alternative, and previous studies offer promising results regarding this. For example, Kim et al. [27] investigated the effect of using LN₂, nanoMQL, and their hybrid on the machinability performance when turning Ti6Al4V alloy. Researchers have claimed that hybrid use produces better results. Yıldırım [28] conducted a series of experiments with combinations of dry, pure MQL, nanofluid, and LN₂ during the turning of Inconel 625 to investigate the effect of cooling conditions on machinability.

In summary, many studies have been conducted to examine the effects of sustainable cooling conditions under heavy processing conditions. However, it has been determined that the studies are still not sufficient due to the existence of many alloys with low machinability, such as Ti6Al4V, and the large number of gases that have both nano additives and cryogenic properties. In other words, there are many cooling parameters whose effects are of interest, and by trying these parameters in different combinations, processing efficiency can be increased much more. So, the aim of this study is to contribute to the literature with the results obtained by using different combinations of environmentally friendly cooling conditions during the processing of Ti6Al4V alloy. In addition, in this study, many output parameters were evaluated in terms of both tribological properties and their effects on machining performance and a comprehensive research was carried out. Thus, it was aimed to present a guiding study for the literature. For this purpose, dry, pure MQL, LN₂, hBN, CuO-doped nanofluids, and hybrid methods were preferred. The temperature analysis in the cutting zone, surface integrity, and wear analysis are discussed in detail.

2. Experimental procedure

2.1. Materials, cutting tool, and machine tool

In the experimental study, Ti6Al4V (standard for ASTM B265) alloy rectangular workpieces with dimensions of 100 × 80 × 15 mm were used. In accordance with the information given by the manufacturer, the chemical and physical properties of Ti6Al4V alloy are tabulated in [Tables 1 and 2](#), respectively.

In milling experiments, Sandvik 490R-08T308E-ML PVD TiAlN coated S30T grade tool with four cutting edges was used. Sandvik 490-025A20-08 L holder was used to mount the cutting tools on the CNC

Table 1
Chemical (%) composition of Ti6Al4V material (from manufacturer).

Designations	C	N	O	H	V	Fe	Al	Ti
Ti6Al4V	0.01	0.01	0.14	0.002	4.00	0.08	5.90	89.86
R56400								
TC4								
Ti64								
Grade 5								

Table 2

Physical specification of Ti6Al4V material (from manufacturer).

Hardness (HV)	Elongation (%)	Yield strength (MPa)	Tensile strength (MPa)
340	10	830	1000

vertical machining center. A new cutting tool edge was used in each experimental condition. In order to avoid runout problems in the tool holder during the milling process, the 35 mm shaft distance was kept constant in all experiments. The Ti6Al4V workpiece is rigidly mounted on the CNC vertical machining center with a precision vise. Milling experiments were carried out on a Delta Seiki CNC 1050 vertical machining center. The CNC vertical machining center has table dimensions of 1000 × 560 mm, spindle norm BT40, and spindle speed of 10,000 rpm. Other details of the experiments performed on the CNC vertical machining center and the experimental setup are given in Fig. 1.

2.2. Stages for nanofluid preparation

In the nanofluid preparation process, heavy metal, chlorine-free vegetable, ester based, and H304 label free Fuchs Plantocut 10 SR oil was used as base fluid. The specification of the oil used is given in Table 3.

Preparation of nanofluid is a critical process, and ensuring the homogeneity of the mixture, as well as its duration and stability, is an extremely important step. In previous studies, recommended mixing stages for nanofluid stability were preferred [29,30]. Firstly, in the mixing step, Fuchs Plantocut 10 SR vegetable oils were filled into glass beakers in 1 L volume. Then, the oils were mixed in a 3-blade mechanical mixer (Daihan HS100D) at the same shaft distance at 750 rpm. Sodium Dodecyl Sulfate (SDS) surfactant was added to the turbulent oil at the rate of 0.25 % of the nanoparticle weight using a precision balance. 0.7 vol% fresh solid nanoparticles (copper oxide-CuO and hexagonal boron nitride-hBN) were added to the oil, which was mixed for 10 min, and the mixture was continued at 750 rpm for 50 min. After the pre-mixing phase, mixing was continued for 30 min through an ultrasonic homogenizer (Bandelin Sonopuls HD 3200) (frequency: 20 kHz, power: 200 W). At the last stage of the mixing, the process was terminated by using a magnetic stirrer (Thermal N11151 – speed: 1500 rpm, time: 120 min). The stages of nanofluid preparation and the equipment

used in the mixing process are given in Fig. 2. The properties of CuO and hBN nanoparticles (supplied from Nanografi (Türkiye)) used in the mixing processes are presented in Table 4, and the nanoparticle and surfactant SEM images are presented in Fig. 3.

2.3. LN₂ storage and experimental parameters

Taylor Wharton XL 45 container was used to store liquid nitrogen gas. These containers are vacuum insulated, stainless-steel containers designed to store and transport cryogenic liquid nitrogen. Produced according to the DOT 4 L standard, the Taylor Wharton container is also safely equipped against possible gas compression. The features of the Taylor Wharton container used in cryogenic experiments are presented in Table 5.

Transfer of N₂ stored in liquid form to the cutting zone is important as there will be heat leaks. Specially produced 1.8 m long vacuum jacketed hoses were used to transfer LN₂ to the cutting zone. LN₂ at 15 bar pressure, carried through vacuum jacketed hoses, was sent to the cutting zone through a nozzle made of stainless steel with a diameter of 3 mm. In all experiments, LN₂ was sent under pressure at a distance of 30 mm from the cutting tool edge. Milling test parameters of Ti6Al4V material are given in Table 6.

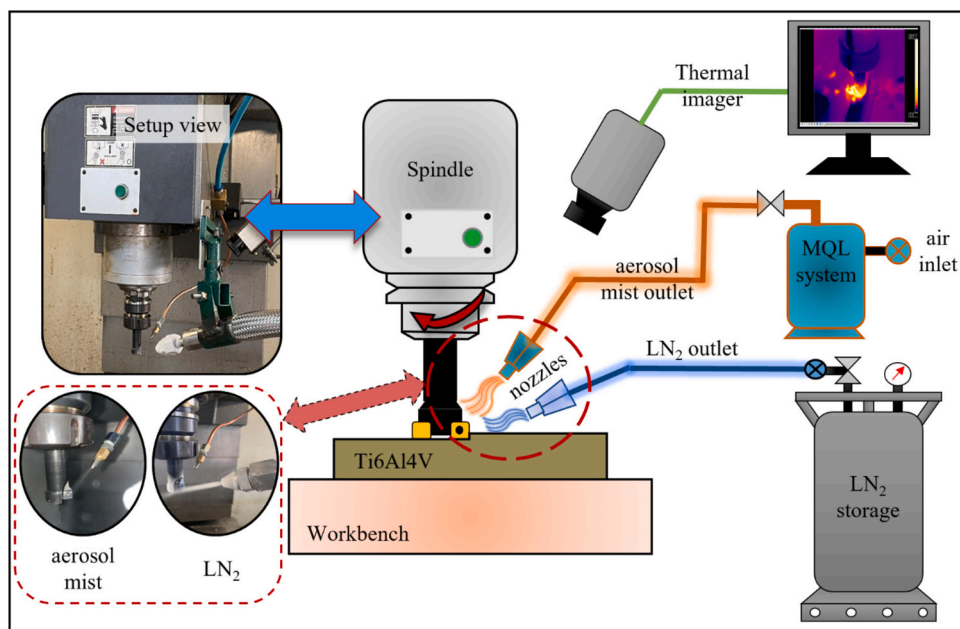
2.4. Critical measurements and methodology

Thermal conductivity coefficients of the mixtures were measured on a Hot Disk TPS 2500 s device at room temperature. Thermal conduction coefficients of the mixtures were determined by taking the arithmetic average of 200 different measurement points. During the measurement, a 5465 cos, 32 mm diameter sensor, specially designed for the thermal conductivity of liquids, was used. Before measurements, the device was calibrated using special solutions. According to the ASTM D2162:2014

Table 3

Specification for vegetable-based fluid (from manufacturer).

Density (at 15 °C – g/mL)	Flash point (°C)	Kinematic viscosity (at 15 °C – mm ² /s)	Cytotoxicity standards (biocompatibility)
0.86	205	10	EN ISO 10993-1, 5, 12

**Fig. 1.** Experimental setup.

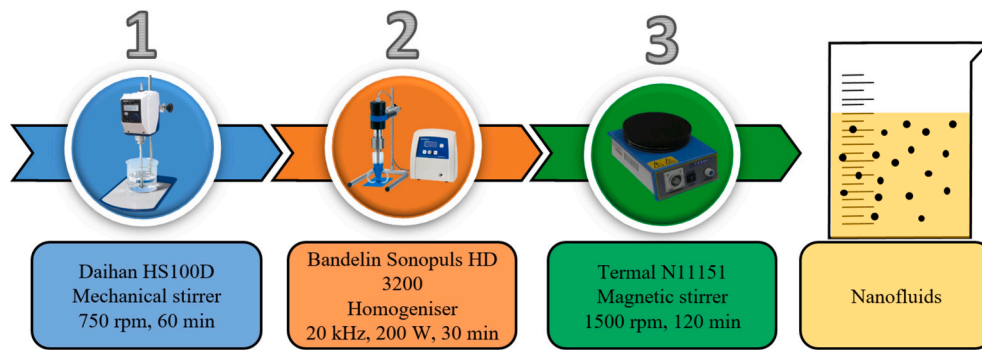


Fig. 2. Stages for nanofluid preparation.

standard, the viscosity values of the mixtures were measured at 40 °C in the AND SV-10 device. pH measurements of the mixtures were determined with a Sartorius PT-10P pH meter at room temperature. The sensitivity of the pH meter is 0.01, and it can measure values ranging from 0 to 15.

Optris PI 450 thermal imaging camera was used to determine the cutting zone temperature. The infrared thermal camera is positioned at a distance of 280 mm from the cutting zone to protect it from chips and adverse conditions. The emissivity value is critical in temperature measurements with a thermal camera and varies depending on the brightness of the materials. In this study, the emissivity value ϵ 0.35 was preferred based on the manufacturer's catalog recommendation. Images from the thermal camera were recorded in video format using the Optris PIX Connect program. Cutting temperature values (maximum) were determined according to the arithmetic average of the highest peak values obtained along the workpiece cutting length (100 mm). After a chip volume of 12,000 mm³ in each cutting environment, the amount of cutting tool flank wear was determined using a DinoLite AM791MZT digital microscope according to the ISO 3685 (1993) standard. The digital microscope magnification scale was set to 50 $\times$, and its calibration was done using DinoCapture 2.0 software. Zeiss LEO 440 SEM imaging device was used to detect wear mechanisms on the cutting tool.

Surface integrity directly affects the performance of components. For this purpose, average surface roughness values (R_a), surface topographies, and subsurface microhardness values were taken into account to determine the surface conditions. R_a values were determined with the Accretch Handysurf+ portable roughness meter device according to the ISO 4287 standard. Before each measurement, the roughness device was calibrated with the device manufacturer's calibration blocks. R_a values were obtained by calculating the arithmetic of measurements made from three different regions. The evaluation length, measurement distance, and sample length were set as 4 mm, 4.8 mm, and 0.8 mm, respectively. Surface topography measurements were determined with the Getphase topography device, and optical imaging was taken from the same regions (middle region) of the milled surfaces. With the SurPhase head, the peaks and valleys of the surfaces in an area of 2.5 mm $\times$ 2 mm were scaled with the Getphase 2.7 software, and image printouts were taken.

Sub-surface microhardness measurements are typically conducted using a microhardness tester. In this study, sub-surface microhardness measurements were utilized according to the ASTM E384–17 standard. Five different microhardness (HV) values were measured at 10 μ m

distances inward from the machined surfaces. Measurements were carried out on the Metkon DuroLine M microhardness measuring device. Before the measurement, chips on the surfaces processed with Ti6Al4V material were carefully removed. Moreover, hardness calibrations were performed with the manufacturer's block gauge before microhardness measurements. HV values obtained as a result of measurements performed according to under 500 g load for 10 s were recorded.

3. Results and discussion

3.1. Thermal conductivity coefficient of nanofluids

Thermal conductivities of the vegetable-based oil and nanofluids used in this study are shown in Fig. 4. As seen in Fig. 4, the lowest thermal conductivity value (0.1987 W/m.K) belongs to the base liquid. The highest thermal conductivity value was obtained in the nanofluid with hBN (0.2161 W/m.K). Compared to the base liquid, the thermal conductivity value of the CuO-added nanofluid increased by 5.84 %, and the thermal conductivity value of the hBN-added nanofluid increased by 8.76 %. It is expected that the thermal conductivity value will increase by adding nanoparticles to the base liquid. Singh et al. [31] stated that the addition of nanoparticles to the base liquid increases the thermal conductivity and that the high thermal conductivity value provides more effective lubrication in the cutting zone and cools the cutting zone. The thermal conductivity value of nanofluids varies depending on the thermal conductivity properties of the added nanoparticles, the amount of concentration, particle size, and surface area [32].

3.2. Viscosity and pH values

Examining the rheological properties of nanofluids, such as viscosity and pH provides guidance in terms of the efficiency of nanofluids. Viscosity and pH values can be related to the stability of nanofluids. In addition, studies have emphasized that the addition of surfactant to the base liquid positively affects the stability and homogeneity properties of nanofluids [33]. It has been observed in studies that the pH value increases by adding nanoparticles to the base liquid [33,34]. Various studies have shown that there is no clumping and a more homogeneous structure, especially in nanofluids with high pH values [34,35]. In addition, the resistance of liquids to microbial action is largely related to the pH value. In other words, too high or too low a pH value can be dangerous for operators and cause problems in waste disposal. Therefore, it is important to know the pH value of the prepared nanofluids. Based on this importance, the pH values of the liquids were measured and are given in Fig. 5 [36].

As seen in Fig. 5, it was observed that viscosity and pH values increased when nanofluid was added to the base liquid. Compared to the base liquid, the pH value of the hBN-added nanofluid increased by 6.49 %, while the CuO-added nanofluid increased by 9.12 %. The reason for this increase can be attributed to the more homogeneous distribution of

Table 4
Specification of CuO and hBN nanoparticles.

Particle type	Purity (%)	Shape	Color	Size (nm)	True density (g/cm ³)
CuO	99.99	Near spherical	Black	30–46	6.50
hBN	99.85	Near spherical	White	65–75	2.30

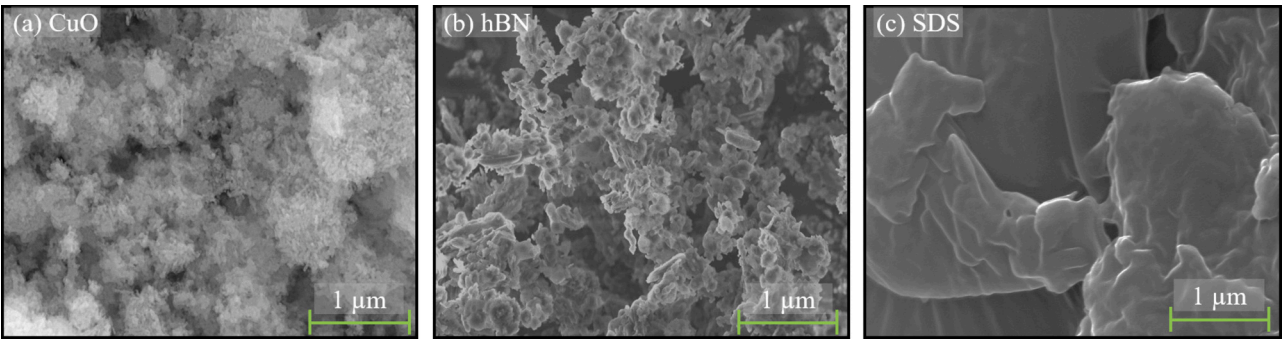


Fig. 3. SEM images of (a) CuO, (b) hBN nanoparticles, and (c) SDS surfactant.

Table 5
Taylor Wharton LN₂ container specification (from manufacturer catalog).

Container type	Useable liquid capacity (L)	Evaporation rate (% per a day)	Flow rate (cu. m/h)	Specification design (TC/DOT)
XL 45	169	1.9	9.2	4LM/4L

Table 6
Experimental parameters for Ti6Al4V material under different environments.

Properties	Comments
CNC Vertical Center	Delta Seiki CNC 1050 A
Cutting tool	Sandvik 490R-08T308E-ML PVD TiAlN
Tool Grade	S30T
Tool holder	Sandvik 490-025A20-08 L
Workpiece	Ti6Al4V Grade 5 (100 × 80 × 15 mm)
Milling parameters	Cutting speed Vc: 120 m/min, feedrate f: 0.25 mm/rev, and cutting depth: ap 1 mm
Basefluid	Fuchs Plantocut 10 SR
Environments	dry, MQL, LN ₂ , LN ₂ + MQL, hBN, LN ₂ + hBN, CuO, and LN ₂ + CuO
MQL	SKF vario, 80 mL/h flowrate, 8 bar pressure, 2 mm nozzle diameter, 25 mm nozzle distance from cutting tool, rake face position
LN ₂ container	Taylor Wharton XL 45, 15 bar pressure, 3 mm nozzle diameter, nozzle distance from cutting tool, rake face position

CuO nanoparticles in the base liquid. It is expected that the addition of nanoparticles to the base liquid will increase the viscosity value of nanofluids. While the lowest viscosity value belongs to the base liquid (10.0 cSt), it is followed by hBN (10.6 cSt) and CuO (11.5 cSt) based nanofluids, respectively. While the viscosity value of the hBN-added nanofluid increased by approximately 6 % compared to the base liquid, the value of the CuO-added nanofluid increased by 15 %. This increase can be attributed to the different densities of hBN and CuO nanoparticles (refer to Table 4).

3.3. Temperature analysis in the cutting zone

In machining operations, approximately 90 % of mechanical energy turns into heat. Thus, a serious cutting temperature occurs in the cutting zone. The resulting high cutting temperature causes excessive wear of the cutting tool, significantly shortening its life. In addition, thermal softening induced in the tool and workpiece disrupts chemical element diffusion and affects the surface quality, dimensional accuracy, and functionality of the machined part [37]. Therefore, it is important to know and control the cutting temperature. In this part of the study, metal removal experiments were carried out under different cooling types, and the effect of cooling conditions on the temperature in the cutting zone was investigated. Dry machining is also included in the

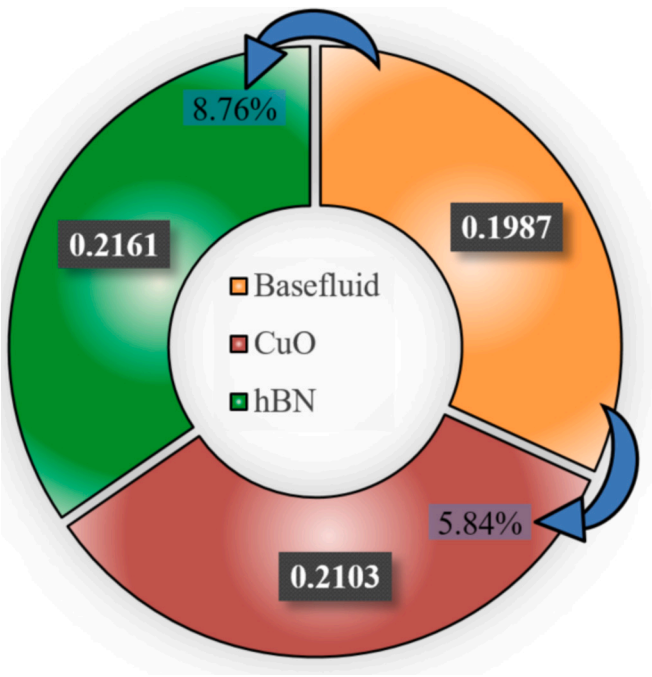


Fig. 4. Thermal conductivity coefficient values.

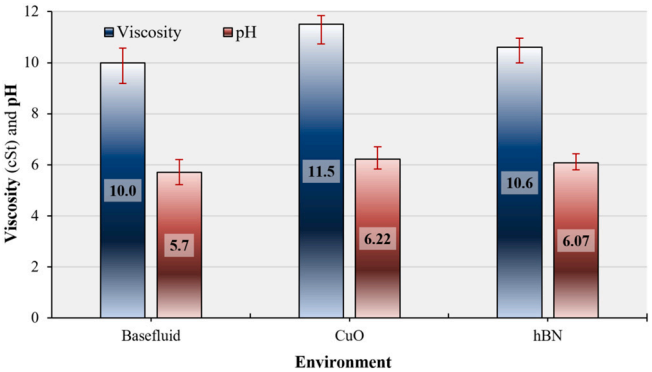


Fig. 5. Viscosity and pH values of base fluid and nanofluids.

system to show the general effect of cooling conditions on the temperature in the cutting zone. Measurement results regarding the temperature in the cutting zone are presented in Fig. 6. When Fig. 6 is examined, it is seen that the highest temperature value is 326 °C under dry machining. This is an expected situation. On the other hand, with the use of cooling systems, the temperature began to decrease. For example,

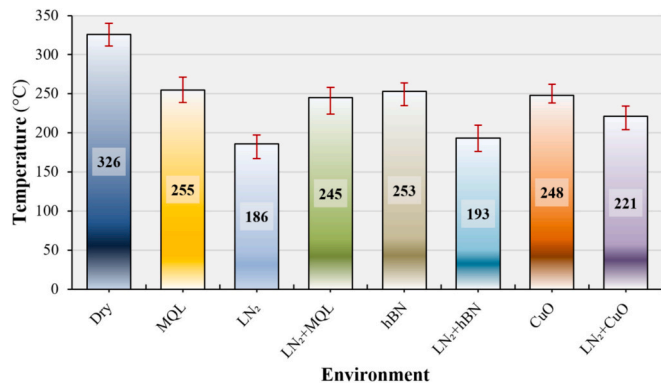


Fig. 6. Cutting temperature results for different environment.

under pure MQL, which gives the highest temperature value among cooling systems, the temperature value is 255 °C, and there is a decrease of 21.78 % compared to the dry environment. The oil delivered to the cutting zone via MQL enters the tool-chip-workpiece interface, reducing friction and partially preventing temperature formation. Moreover, the small amount of cutting fluid used in the MQL system evaporates rapidly under heavy machining conditions, helping to remove the heat in the cutting zone. The decrease in cutting temperature continues in the nanoparticle doped MQL system. While the cutting temperature under hBN-doped nanofluid was measured as 253 °C, the cutting temperature with CuO doping was measured as 248 °C. Compared to dry environment, hBN-doped nanofluid provided 22.39 % improvement, while CuO-doped nanofluid provided a 23.93 % improvement. Nanoadditives increase the thermal conductivity of the liquid and thus enable faster heat dissipation in the cutting zone [38]. In addition, nanoparticles entering the tool-chip-workpiece interface reduce friction by causing a rolling effect, thus reducing heat generation. In this study, the decrease in cutting temperature with nano-added cutting fluids was associated with these properties of nanofluid.

In this study, significant decreases in cutting temperature were achieved with the introduction of LN₂. Compared to dry processing, pure LN₂, LN₂ + MQL, LN₂ + hBN, LN₂ + CuO decreased by 42.9 %, 24.85 %, 40.80 % and 32.21 %, respectively. In the MQL system, when a very small amount of oil is sprayed on the cutting area, the cutting fluid can evaporate quickly. This situation is especially evident under heavy machining conditions such as the machining of Titanium alloys. For this reason, much lower temperatures can be achieved with LN₂, which has obvious cooling properties. Jebaraj et al. [39] claimed that at very high cutting temperatures, oil-based coolants evaporate before reaching the cutting zone and fail to penetrate the sliding interfaces. They also stated that hot steam around the cutting zone reduces heat transfer, and cryogenic gases are more effective at cutting temperature. In another study, Gupta et al. [40] stated that in cryogenic machining, the cryogenic coolant quickly leaves the region by absorbing heat and thus reduces the cutting temperature. Additionally, researchers claimed that the application of cryogenic cooling condition helps break the chips into small pieces, thus reducing the friction of the chips against the cutting tool. Moreover, another interesting result is that the temperature under pure-LN₂ is lower than other hybrid LN₂ (such as LN₂ + MQL and LN₂ + hBN) conditions. It is thought that under the LN₂ + MQL condition, the cutting fluid freezes and acts as a barrier in the cutting zone, so MQL + LN₂ (especially LN₂) cannot effectively penetrate the cutting zone to reduce the temperature. Therefore, when only LN₂ was used, the cryogenic fluid effectively penetrated the cutting zone and reduced the cutting temperature.

3.4. Tool wear analysis

Due to increasing costs, it is desired to obtain maximum efficiency

from cutting tools in terms of efficient use of resources and economic sustainability. Moreover, surface integrity, which is of critical importance in the components used in the workpieces, is directly affected by the cutting tool. Therefore, in this subsection, cutting tool wear, which is very important for economical machining and satisfactory part quality, is studied. Different methods have been used to analyze tool wear in detail. First, the size of the wear was measured with a microscope, and the results were presented graphically. Then, the wear mechanisms that caused the failure of the cutting tool were determined by SEM and EDX analyses.

A fixed volume (12,000 mm³) of chip was removed using a new cutting edge in different cutting environments, and the size of the flank wear was measured at equal intervals (every 3000 mm³). The results are presented in Fig. 7. As expected, the highest flank wear occurred under dry cutting as 470 µm. Remarkable decreases in flank wear have been observed by incorporating different cooling lubrication methods into the cutting environment. To express the decreases as a percentage, the maximum improvement of 61.7 % was reached under LN₂ + CuO-nanofluid, and this was followed by LN₂ + hBN-nanofluid (59.57 %), CuO-nanofluid (57.45 %), hBN-nanofluid (51.06 %), LN₂ + MQL (34.04 %), LN₂ (31.7 %) and MQL (25.53 %) compared to dry machining. It is thought that CuO-based nanofluid shows better dispersion stability compared to hBN. According to the pH results given in Fig. 5, the improved pH value of the CuO-based nanofluid supports this inference. A better dispersion stability allows nanofluids to be used effectively without agglomeration. Jyothish et al. [41] compared the stability of ZnCO₃, BN, and CuO nanofluids and found that CuO showed maximum dispersion stability in sunflower oil for up to two weeks. The higher viscosity of CuO-based nanofluid is also thought to be an important factor in reducing tool wear. Cutting fluid with ideal viscosity values adheres better to and penetrates the tool-chip interface. Moreover, in the machining zone, the friction coefficient is reduced by the rolling effect and thin film layer of CuO nanoparticle cutting oil, resulting in reduced flank wear.

In the hybrid cooling/lubrication (LN₂ + CuO or LN₂ + hBN) process, cryogenic coolant and nanofluid work in a mutual interaction that affects cutting performance. Cryogenic coolant both delays tool wear by reducing the cutting temperature and prevents the evaporation of cutting fluid from the tool-chip-workpiece interfaces by absorbing the heat of the environment. In this way, it is thought that nanofluids and cryogenic cooling liquids penetrate the cutting zone longer and more effectively.

In this subsection, the wear mechanisms that lead to tool failure are also examined with the help of SEM and EDX analysis, as seen in Fig. 8. When the figures are examined, it can be seen that various failure mechanisms, i.e., built-up edge (BUE), built-up layer (BUL), chipping, notching, and peeling of coating, play an active role in the damage to the tools. The predominant wear mechanism leading to tool failure is thought to be adhesion wear. On the rake face, there was a lot of chip adhesion, which resulted in the formation of enormous BUEs and BULs. BUE is a part of the workpiece material that adheres to the tool rake surface and has a noticeable size, especially when machining ductile alloys such as aluminum and titanium [42]. BUE is extremely strain hardened and can grow to a noticeable size and even replace the cutting tool to perform the cutting operation [43]. When machining titanium alloy, the BUE can be observed on every cutting tool [44]. However, its level can be minimized by changing the cutting parameters and improving the cutting conditions, as occurred in this study. Throughout the whole rake face, particles of the workpiece material were found from the cutting edge. The W-rich substrate was revealed as a result of the coating peeling off at and away from the cutting edge due to adhesive wear. The adhesive wear mechanism resulting from the interaction of the tool material and the workpiece material under extreme stress and temperature during cutting weakens the strength of the cutting edge at the end of a certain cycle and causes chipping [45]. Moreover, adhesive wear has been considered the main cause of notch formation. Chip flow

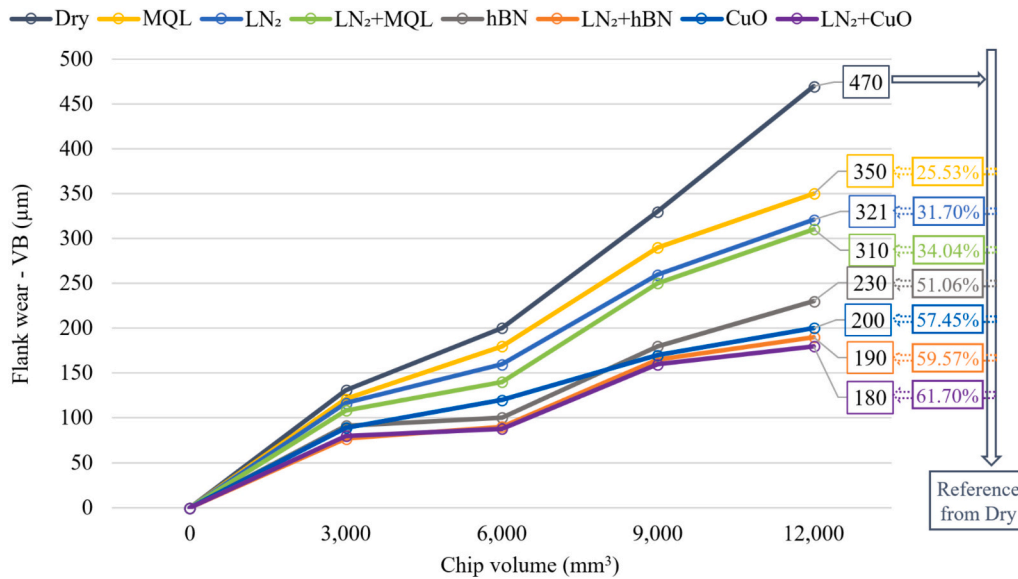


Fig. 7. Flank wear VB trends under different environment (at the end 12,000 mm³ chip volume).

and breakage of the adhesive layer created a slot at the end of the cutting depth, causing notching.

During cutting, the workpiece material tends to weld with the tool surface under high pressure and temperature. However, this process is unstable and may be stripped along with the flowing sawdust under the influence of high friction force. If the tensile strength of the joint is greater than the strength of the tool materials, the ongoing cycle may cause tearing of the tool coating or substrate, triggering adhesion wear and its resulting damage mechanisms. Thus, adhesion and adhesion-induced damages are the result of the combined mechanical-thermal effect at the tool-chip interface [46].

3.5. State analysis of machined surfaces

3.5.1. Evaluation of surface roughness averages

Surface roughness is one of the most important quality indicators that determine product quality in the manufacturing industry and is considered as a machinability criterion. It is an indispensable element of the aviation and space industry, especially where measurement accuracy and surface quality are very important. In this study, a series of tests were carried out to determine the effect of different cooling lubrication conditions on the surface roughness in the milling of Ti6Al4V alloy, which is widely used in the aviation and medical industries. Changes in Ra values obtained as a result of the tests are given in Fig. 9. Among eight different environmental conditions, the highest Ra value of 0.681 µm was obtained in the LN₂ cutting condition. This value was 9.7 % higher than the dry cutting condition, which was the second highest value (0.615 µm). The absence of any lubricating agent in dry cutting causes high friction at the tool-chip interface, which results in an increase in the Ra value, as expected. Similarly, in the use of LN₂, in addition to the lack of a lubricating agent, it is thought that the very high cooling effect (196 °C) causes a kind of adhesion effect at the tool-chip contact point, causing a further increase in friction. Namely, LN₂ applied to the cutting area for a certain period of time creates frost on the part (see Fig. 1). This snow, which melts locally with the heat generated during the deformation of the material, has an adhesion effect by freezing with LN₂ applied between the tool/chip or tool/workpiece pair. This situation caused a higher Ra value compared to the dry cutting condition, which was associated with increased friction. MQL cutting condition resulted in a decrease of 7.3 % and 16.3 % in Ra values compared to dry cutting and LN₂ cutting conditions, respectively. This can be attributed to the reduction of friction by providing effective

lubrication in the tool/chip contact area of MQL [33]. hBN and CuO nanoparticles added into pure oil significantly improved the tribological properties (cooling and lubrication) of the oil, resulting in lower Ra values than pure MQL. Thus, while hBN decreased by 13.2 % in Ra values, CuO decreased by 19.7 %. These decreases not only created an effective oil film in the tool/chip contact area of the nanoparticles used but also increased the surface quality by making mending, polishing, and especially bearing effects due to their spherical forms. The superiority of CuO nanofluid over hBN nanofluid in terms of surface quality is attributed to its tribological and thermal properties. LN₂ cutting condition, which gives a higher Ra value than dry cutting when used alone, interestingly provided a 6.5 % lower Ra value even than MQL when combined with MQL. This result shows that the cooling feature alone, as in LN₂, and the lubrication feature alone, as in MQL, are insufficient to increase the surface quality. The low thermal conductivity coefficient of the Ti6Al4V alloy causes high temperatures in the cutting zone, which negatively affects both tool wear and surface quality [47,48]. Therefore, this study clearly demonstrates the importance of the combination of two different methods with cooling and lubrication features in achieving the desired surface quality. In particular, combining LN₂ with two different nanofluids separately enabled the lowest Ra values to be obtained. While Ra was measured as 0.495 µm in the LN₂ + hBN condition, it reached its lowest value as 0.371 µm in the LN₂ + CuO condition. As a result, the superior lubrication properties of nanofluids, combined with the superior cooling properties of LN₂, played a key role in increasing the surface quality by reducing friction as well as eliminating the high temperatures that occur during the machining of Ti6Al4V alloy.

3.5.2. Evaluation of surface topographies

Unlike Ra, surface topography allows to evaluate the surface from a wider perspective and to see more clearly the effects of the relative movement of the tool and workpiece on the surface. In other words, the machining marks, peaks, and valleys that form on the surface give important clues about the cutting process. In this context, 2D and 3D surface topographies were taken for detailed evaluation of the machined surfaces after milling the Ti6Al4V alloy under different cooling/lubrication regimes, and the relevant images are given in Fig. 10. Among the surface topographies, the presence of red regions (peaks) and blue regions (valleys) is noteworthy, especially in dry and LN₂ cutting conditions. It is seen that the green zones (nominal), which are the desired surface forms, are poor (Fig. 10(a–c)). This shows that the high-strength Ti6Al4V alloy is formed with high deformation under these conditions as

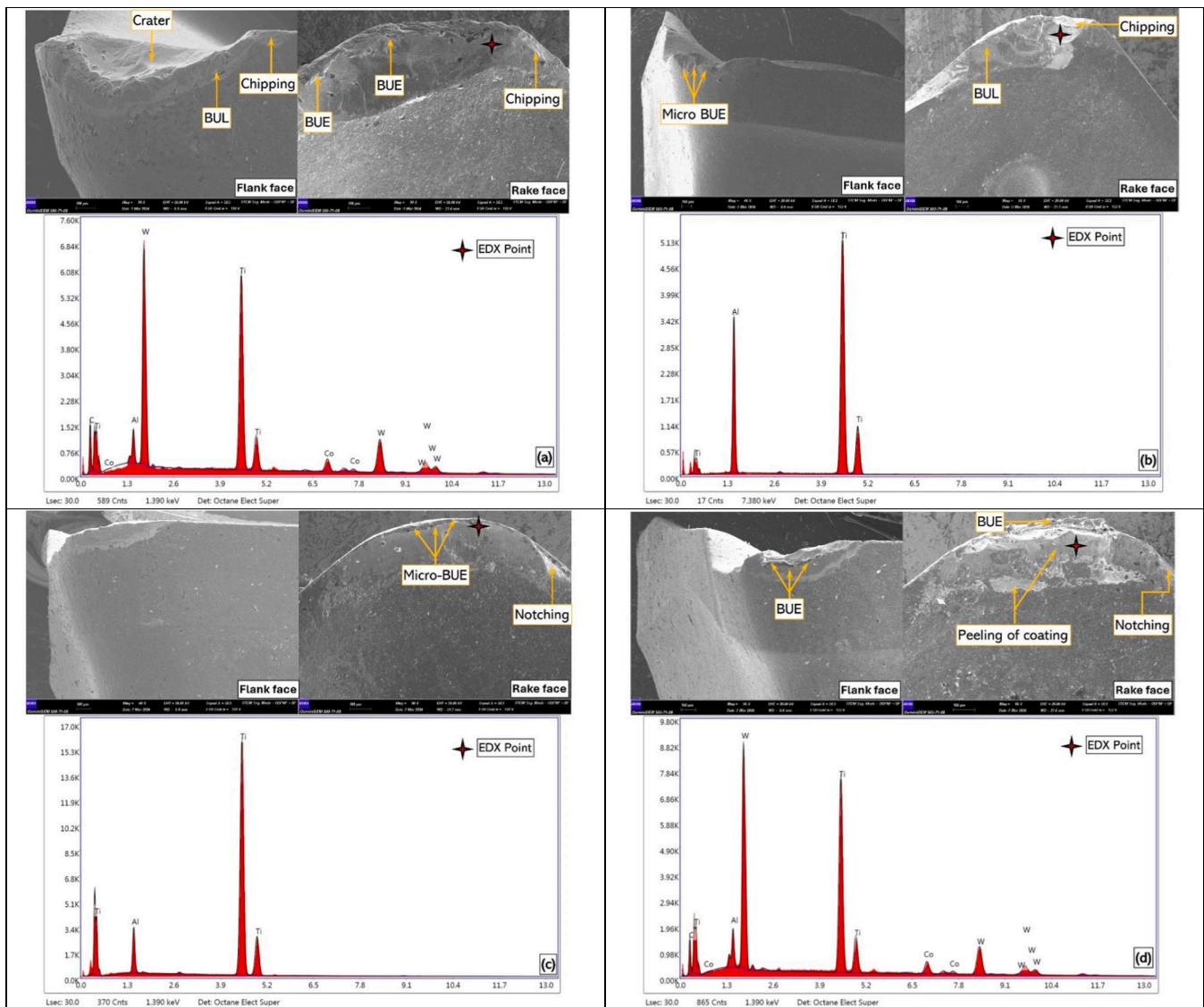


Fig. 8. SEM image and EDX analysis of worn cutting tools at 12000 mm³ chip volume under different environments: a) dry b) LN₂ c) LN₂ + MQL d) MQL e) hBN f) LN₂ + hBN g) CuO h) LN₂ + CuO.

a result of low lubrication and/or low cooling. One of the most important factors that negatively affects the surface quality in machining is the formation of BUE. Ti6Al4V alloy, which has a high tendency to chemically react with the cutting tool, causes BUE formation on the cutting edges of the cutting tool during machining [49]. The large and unstable chip debris on the cutting edge deteriorates the quality of the machined surface and increases the surface roughness, causing topography to deteriorate. As a matter of fact, tool wear pictures under these conditions confirm the existence of BUEs (see Section 3.4). The presence of a lubricating agent that reduces friction and, thus BUE formation under the MQL cutting condition played an important role in improving the surface topography. In addition, the combination of MQL and LN₂ contributed positively to the surface topography by effectively removing heat from the region, which negatively affects the surface quality. On the other hand, nanofluids provided significant increases in the thermal conductivity of the cutting oil, thanks to the nanoparticles added into it, and improved the cooling feature as well as the lubrication feature of pure-oil, resulting in a topography similar to the MQL + LN₂ cutting condition. Here, it cannot be ignored that nanoparticles not only increase the cooling performance but also provide effective lubrication by

creating a barrier between the surfaces in contact. It has been observed that the best (desired) surface topographies among all cutting conditions were obtained in combinations of LN₂ and nanofluids. These results show the importance of the combination of effective cooling and lubrication in reducing cutting temperature and friction, as well as improving the surface topography by suppressing BUE formation and tool wear, in the machining of a material such as titanium, which has high strength, low thermal conductivity and a high tendency to chemical reactions. Although cooling of the cutting zone contributes positively, only sub-cooling (LN₂), which lacks lubrication in terms of surface topography, resulted in negative rather than positive properties.

3.5.3. Evaluation of subsurface microhardness

Surface and sub-surface microhardness values give important clues about the extent to which the processed material is exposed to deformation and how much this deformation penetrates the material. It also helps analyze factors that may cause phase change, such as high temperature and cooling in the cutting zone. This allows better analysis of the conditions under which the cutting occurs and the integrity of the surface. In this context, sub-surface micro hardness measurements were

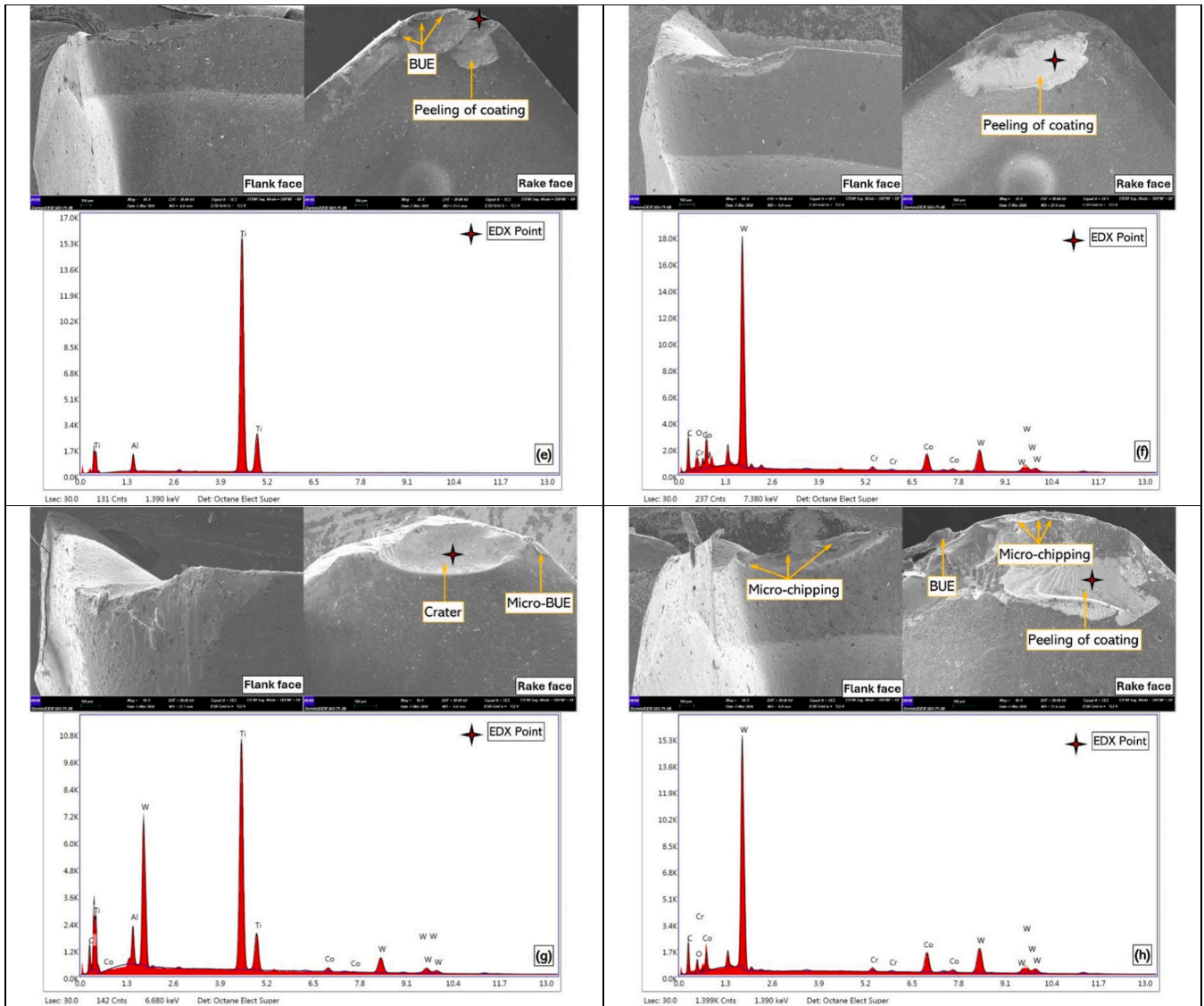


Fig. 8. (continued).

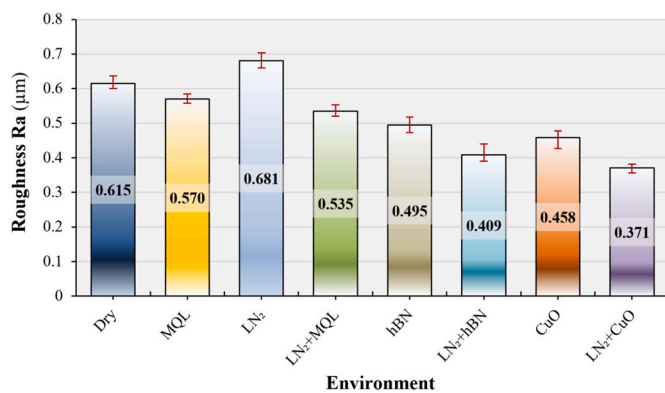


Fig. 9. Surface roughness deviations under different environment.

made at different depths of Ti6Al4V alloy milled under different cutting conditions, and the results are given in Fig. 11. Although there were significant hardness changes between all cutting conditions, it was observed that the microhardness values obtained in all these conditions were higher than the microhardness of the bulk material (342 HV). In

addition, microhardness changes between cutting conditions decreased from the machined surface to the sub-surfaces and were equalized with the hardness of the bulk material when a depth of 50 µm from the surface was reached. This is a clear indication that the milling process causes deformation up to a depth of 50 µm from the surface of the material. Among the cutting conditions, the highest microhardness values were achieved in LN₂ (400 HV) and dry cutting (389 HV) conditions. In fact, a hardness increase of 17.02 % and 13.74 % was recorded in LN₂ and dry cutting, respectively, compared to the bulk material. High heat generation and friction occurring in the cutting zone promote deformation hardening of the material, resulting in high microhardness values [50]. In addition, the cutting process performed with a worn tool causes a change in the microstructure of the material and increases the effectiveness of deformation hardening on the machined surface [51,52]. In the light of these explanations, the fact that the highest cutting temperature and high tool wear were obtained under dry cutting condition, as a result of the high friction in the cutting zone explains the high microhardness value. In the LN₂ cutting condition, although the heat load in the cutting zone is eliminated with the high cooling effect, the absence of any lubricating agent increases the friction between the tool and the workpiece. In addition, the decreasing heat load in the cutting zone reduces the thermal softening effect. This increases the

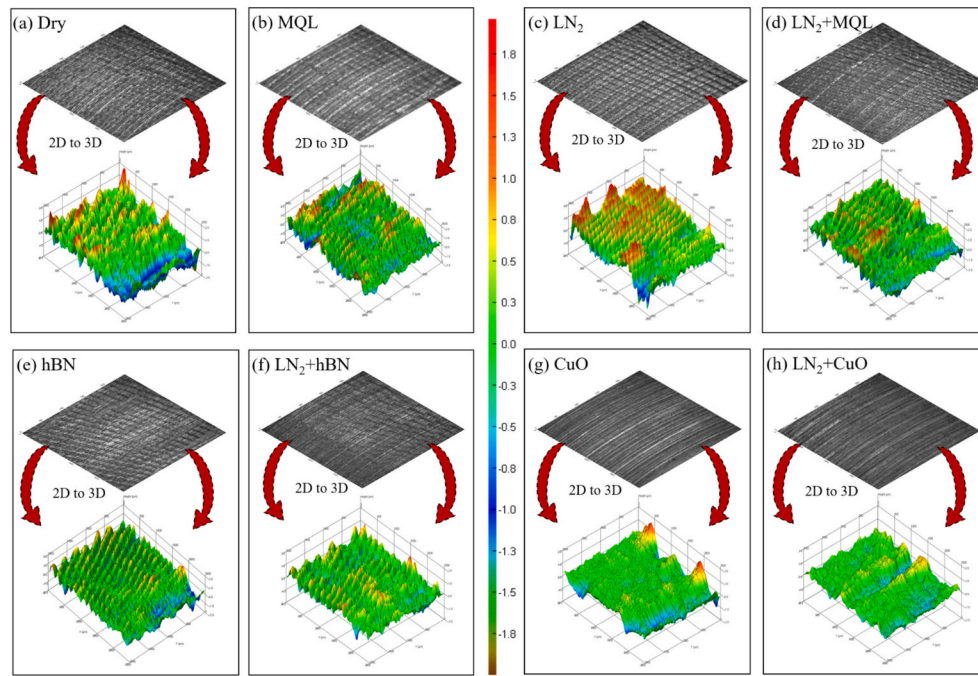


Fig. 10. Machined surface 2D to 3D topographies under: (a) Dry, (b) MQL, (c) LN₂, (d) LN₂ + MQL, (e) hBN, (f) LN₂ + hBN, (g) CuO, and (h) LN₂ + CuO.

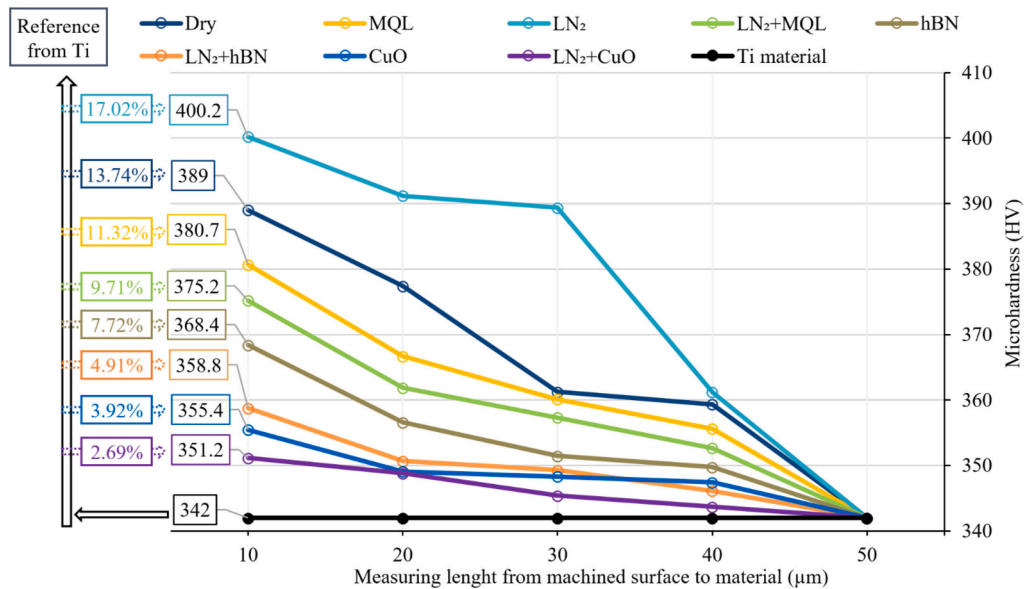


Fig. 11. Microhardness deviation values from machined surface to Ti6Al4V material.

amount of plastic deformation in the upper surface layer of the workpiece. The fact that the highest tool wear value was obtained in the LN₂ cutting condition is another issue that contributes to the increase in hardness. MQL cutting condition helped to reduce friction and partially cutting temperature, thus reducing plastic deformation and achieving lower subsurface hardness. In particular, combining MQL with LN₂ reduced the plastic deformation effect on the machined surface by decreasing temperature and friction, thus playing an important role in reducing the hardness value. Similarly, thanks to the effective lubrication and cooling properties of nanoparticles, they contributed to reducing surface deformations and hardness. Among all cutting conditions, the combined use of nanofluids and LN₂ made the greatest contribution to obtaining subsurface microhardness closest to the bulk material and preserving surface integrity.

4. Overall evaluation and conclusions

In the current study, milling operation was applied to Ti6Al4V alloy under different ecological environments. In total, eight different ecological cooling/lubrication environments (1) Dry, (2) MQL, (3) LN₂, (4) LN₂ + MQL, (5) hBN, (6) LN₂ + hBN, (7) CuO, and (8) LN₂ + CuO were used in the study. Thermal conductivity, viscosity, pH, cutting temperature, tool wear, surface roughness, surface topography, and machined subsurface microhardness were measured to determine the performance of different ecological cooling/lubrication environments. Overall, the key conclusions are listed below.

- Thermal conductivity was found to increase as nanoparticles were added to the basefluid. The highest thermal conductivity value was

obtained in hBN nanofluid (0.2161 W/m.K), followed by CuO nanofluid (0.2103 W/m.K). Compared to basefluid, hBN and CuO nanofluids showed an increase of 8.76 % and 5.84 %, respectively.

- The addition of hBN and CuO nanoparticles increased the basefluid viscosity and pH values. Compared to basefluid, hBN and CuO nanofluids increased pH values by 6.49 % and 9.12 % and viscosity values by 6 % and 15 %, respectively.
- In the cutting zone, the highest cutting temperature was obtained in the dry environment (326 °C), and the lowest cutting temperature was obtained in the LN₂ environment (186 °C). Compared to dry environment, MQL, LN₂, LN₂ + MQL, LN₂ + hBN, and LN₂ + CuO environments reduced the cutting temperature by 21.78 %, 42.9 %, 24.85 %, 40.80 %, and 32.21 %, respectively.
- The highest cutting tool flank wear was recorded in the dry environment (470 µm), while the lowest cutting tool flank wear was recorded in the LN₂ + CuO environment (180 µm). The flank wear values compared to the dry environment was measured: LN₂ + CuO (61.70 %), LN₂ + hBN-nanofluid (59.57 %), CuO-nanofluid (57.45 %), hBN-nanofluid (51.06 %), LN₂ + MQL (34.04 %), LN₂ (31.7 %), and MQL (25.53 %), respectively. BUE, BUL, chipping, notching, and peeling of coating mechanisms were effective in tool wear. The adhesion wear mechanism leading to tool wear was found to be dominant. Especially in SEM images where chips adhering to the cutting tool rake surface were detected, it was understood that BUE and BUL mechanisms are effective.
- According to the surface roughness average Ra results, the highest Ra value was obtained in the LN₂ environment (0.681 µm), and the lowest Ra value was obtained in LN₂ + CuO (0.371 µm) hybrid environment. Compared to LN₂ environment, dry, MQL, LN₂ + MQL, hBN, CuO, LN₂ + hBN, and LN₂ + CuO environments reduced Ra values by 9.7 %, 16.3 %, 21.4 %, 27.3 %, 32.8 %, 39.9 %, and 45.5 %, respectively. Surface topography results followed a trend parallel to Ra results. The lowest Ra value was obtained in the 3D surface topography peak, and the valley was obtained in the LN₂ + CuO environment at relatively lower levels compared to other environments. The highest peak and the lowest valley were obtained in the LN₂ environment with the highest Ra value.
- For the microhardness results taken from the bottom of the milled surfaces, the highest microhardness value was measured in LN₂ environment (400.2 HV). Compared to Ti6Al4V bulk material (342 HV), LN₂ + CuO, LN₂ + hBN, CuO, hBN, LN₂ + MQL, MQL, dry, and LN₂ environments increased the microhardness values by 2.69 %, 3.92 %, 4.91 %, 7.72 %, 9.71 %, 11.32 %, 13.74 %, and 17.02 %, respectively.

Overall, Ti6Al4V material was successfully shaped in the milling process under ecological cooling/lubrication environments. Compared to the other environments, the LN₂ + CuO hybrid environment performed relatively best. The combined condition of the cooling ability of the LN₂ environment and the lubricating ability of the CuO nanofluid environment gave promising results. As authors, we hope that the paper will shed light and provide ideas for future studies.

CRediT authorship contribution statement

Emine Şirin: Writing – original draft, Methodology, Data curation. **Çağrı Vakkas Yıldırım:** Writing – original draft, Resources, Investigation. **Şenol Şirin:** Writing – review & editing, Writing – original draft, Visualization. **Turgay Kıvak:** Writing – review & editing, Visualization, Validation, Supervision. **Murat Sarıkaya:** Writing – review & editing, Visualization, Supervision, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence

the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgement

Murat Sarıkaya acknowledges the Polish National Agency for Academic Exchange (NAWA) under the Ulam Programme (Grant No. BPN/ULM/2023/1/00035) for financial support.

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