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Graphene Nanoplatelets-Assisted Minimum Quantity Lubrication In Turning To Enhance Inconel 718 Surface Integrity

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Abstract

Inconel 718 is regarded to be a difficult-to-cut nickel alloy due to its low thermal conductivity, high hardness, and chemical reactivity with tool materials at high temperature, which, overall, cause the generation of extremely high temperature at the cutting edge, contributing to deteriorate the surface quality of the machined workpiece. With the aim of improving the alloy machinability, the present paper evaluates the effect of Minimum Quantity Lubrication (MQL) on the turning performances of Inconel 718 compared to dry and flood lubrication conditions. In particular, for the first time, the feasibility of using graphene nanoplatelets as additives to a vegetable oil to form MQL mist is assessed. After having defined the optimal concentration of two graphene nanoplatelets of different size, the nanofluids were prepared and their stability as a function of time was assessed; afterwards, they were characterized by means of viscosity and specific heat capacity measurements as a function of temperature. The cutting performances were evaluated in terms of surface integrity and chip morphology. Results showed that the use of the nanofluid with the lowest graphene nanoplatelets size provided the best surface integrity compared to the ones obtained when using just MQL without any additive and standard lubricating conditions.

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Keywords: Inconel 718; Turning; Nanofluids; Surface finish.

1. Introduction

Inconel 718 is a nickel-based super alloy characterized by high strength, hardness and corrosion resistance even at very high temperatures. For these reasons, it is widely used for static and rotating parts, like blades, blisks and shafts in high pressure compressor and turbine stages [1]. These unique characteristics combined with its low thermal conductivity make Inconel 718 one of the most difficult-to-cut metal alloy, since very high amount of heat develops and accumulates at the cutting zone [2].

To minimize the heat produced during machining, the flood lubri-cooling regime is usually utilized. However, the indiscriminate use of conventional cutting fluids is detrimental to the environment and dangerous for the health of the operators [3]. To reduce the environmental impact of this procedure, Minimum Quantity Lubrication (MQL) techniques, which consist in providing the optimal amount of cutting fluid mixed with compressed air, have been used for a few years [4]. Unfortunately, these techniques cannot remove the excessive heat generated when machining difficult-to-cut alloys.

Within this framework, in order to enhance the MQL technique efficiency, several researchers tried to add nano-sized metallic or non-metallic particles to the base oil. Vasu et al. [5] applied three different cutting conditions, namely dry, MQL with vegetable oil, and MQL with vegetable oil and Al₂O₃ nanoparticles, to machine the Inconel 600 nickel alloy. They found that the surface roughness, cutting temperature, cutting force, and tool wear could be remarkably decreased by using the nanoparticle-enriched vegetable oil. This was ascribed to the enhanced thermal properties of the nanofluid, which presented higher thermal conductivity and heat transfer

coefficient than the ones of the pure oil.

Yi et al. [6] used different graphene oxide nanoparticles in different concentrations added to a semi-synthetic cutting fluid to machine the Ti6Al4V titanium alloy. They found that smoother surfaces with less scratches and reduced plastic deformation were obtained when using the nano-particles, especially when sprayed at the highest concentration. Such amelioration was attributed to lower cutting temperatures at the cutting zone, which induced a higher material resistance to deformation.

Hegab et al. [7] investigated the effect of two nanofluids on the tool wear and chip morphology during turning Inconel 718. Multi-walled carbon nanotubes and alumina nanoparticles were used as nano-additives. The obtained results showed that higher shear angles together with lower cutting forces were achieved by using the nano-additives. Furthermore, carbon nanotubes showed better performances compared to alumina nanoparticles, thanks to their higher thermal conductivity.

Nevertheless, in none of the previous studies, graphene nanoparticles were used as additives to the base oil. The graphene has a thermal conductivity as high as 5000 (W/m*K) [8], thus it can be a great heat transferring intermediary, and, as a consequence, can represent an ideal candidate as additive to MQL. To this aim, in the present study, graphene nanoplatelets of 5 μ m and 15 μ m size were utilized as nanoparticles to be added to pure oil in MQL mode when machining Inconel 718. The machined surface integrity and chip morphology were compared with the ones obtained when using standard MQL, conventional flood and dry conditions at three different cutting speeds.

Nomenclature

MQL_Pure minimum quantity lubrication with just vegetable oil

MQL_5 nanofluid with graphene platelets of 5 μm size MQL_15 nanofluid with graphene platelets of 15 μm size

 $\begin{array}{lll} \textbf{Vc} & & \text{cutting speed (m/min)} \\ \textbf{t}_c & & \text{chip thickness (mm)} \\ \textbf{t}_0 & & \text{uncut chip thickness (mm)} \\ \boldsymbol{\lambda} & & \text{chip compression ratio} \\ \boldsymbol{\varnothing} & & \text{chip helix angles} \\ \end{array}$

2. Experimental

2.1. Workpiece material

The material under investigation was the AMS 5663 Inconel 718 supplied in the precipitation hardened condition. The latter treatment is usually carried out to develop maximum strength by precipitation of dispersed phases throughout the matrix. The material was supplied in form of bars of 41 mm of diameter and 1000 mm of length. The bars were cut, polished and etched for 35 s using the Kalling's reagent to observe the as-delivered microstructure. The grain size was then measured by using a MatlabTM script. The optical microscopy image of the microstructure and related grain size distribution are reported in Fig. 1 a) and b), respectively.

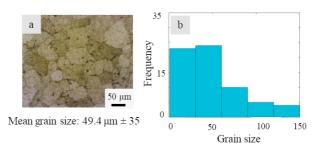


Fig. 1. (a) Inconel 718 microstructure in the as-delivered condition; (b) grain size distribution.

2.2. Machining tests

Turning operations were conducted on a NL 1500 Mori SeikiTM lathe. The adopted cutting tool was a TiAlN-coated CNMG 12 04 04-SM 1105 insert supplied by SandvikTM.

The insert was chosen as suitable to cut heat-resistant alloys and it was characterized by a cutting radius of 0.397 mm. In order to avoid the influence of the tool wear, a new insert was used for each cutting condition.

The bars were first rough machined up to a final diameter of 38.8 mm using cutting speed of 100 m/min, depth of cut of 1 mm, and feed of 0.3 mm/rev. Then, a finishing pass was carried out using fixed depth of cut of 0.2 mm and feed of 0.2 mm/rev, using three values of the cutting speed, namely 70 m/min, 100 m/min and 130 m/min.

The following five lubricating-cooling conditions were adopted: i) dry; ii) conventional flood; iii) minimum quantity lubrication with vegetable oil (MQL_Pure); iv) nanofluid with graphene platelets of 5 μ m size (MQL_5), and v) nanofluid with graphene platelets of 15 μ m size (MQL_15). Conventional flood refers to the use of the commercial semi-synthetic cutting fluid MonroeTM Astro-Cut HD XBP oil mixed with water (1:20 mixing ratio). In this regime, the oil is sprayed on the cutting zone without any kind of regulation.

MQL refers to the use of only a limited amount of lubricant, which is mixed with compressed air before being sprayed on the cutting zone. In this study, the DropsaTM MQL system was used by selecting an air pressure of 7 bar and oil flow rate of 1320 ml/h. A single nozzle placed 2 mm far from the cutting edge with an angle of approximatively 45° from the flank surface of the cutting tool insert was used (see Fig. 2). As a result, the jet of lubricant effectively fell on the cutting zone. The same system was used for the nanofluid solutions, but with the characteristics and preparation procedure described in § 2.3. All the experiments were conducted three times, and the average of the acquired data was considered.

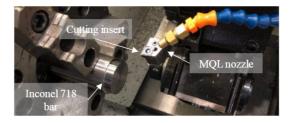


Fig. 2. MQL experimental setup.

2.3. Preparation and characterization of the nanofluids

The LB 2000 cutting fluid provided by Partelli Group[™] was used as the base oil. It is a non-toxic and biodegradable fluid

produced using natural ingredients. Graphene nanoplatelets, purchased from Sigma AldrichTM, were used as additives to the base oil. These nanoplatelets are short stacks of graphene sheets having a platelet shape, held together by Van der Waals bonds that let them slip easily relative to each other under shear loads.

In this study, two different dimensions of the nanoparticles were used, namely 5 μm and 15 μm . In both cases, the average thickness was approximately 6-8 nanometers, whereas the surface area ranged between 50-80 m²/g for the 5 μm size particles and 120-150 m²/g for the 15 μm size particles. Exemplary SEM images of the nanoplatelets are shown in Fig. 3 a) and b).

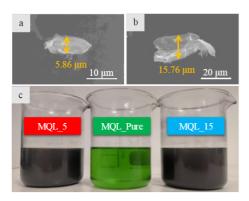


Fig. 3. Exemplary SEM images of the (a) 5µm size and (b) 15µm size graphene nanoplatelets; (c) nanofluids appearance after preparation.

The nanofluids preparation procedure included two steps. First, the graphene nanoplatelets were weighted using a KERNTM analytical balance (accuracy of 0.1 mg) and subsequently added to the base oil. Afterwards, they were mixed for 30 min using an IKATM magnetic stirrer. Finally, the nanofluids mixture was placed into an ElmasonicTM ultrasonic vibration bath for 1 hour. The weight concentration of the graphene nanoplatelets into the base oil was fixed at 0.1 wt.% for both types of nanoplatelets. The appearance of the nanofluids obtained with this procedure is visible in Fig. 3 c).

The stability of the nanofluids and the absence of sedimentation was visually inspected after 24 h. Visual inspection is the most extensively used technique to investigate the presence of sedimentation, since there are not standard methods available [9].

The density of the nanofluids was calculated according to the law of mixtures ([9]), acquiring the density of the pure oil and the one of the graphene nanoplatelets from datasheets (920 kg/m 3 for the pure oil and 65 kg/m 3 for the graphene nanoplatelets).

The dynamic viscosity of the investigated cutting fluids was measured by means of the ARES TM viscometer as a function of the shear rate and temperature. The former was varied from 0.1 s^{-1} to 1000 s^{-1} and the latter was set equal to 40°C and 100 °C.

A DSC Q200TM differential scanning calorimeter was used to measure the specific heat capacity of the investigated cutting fluids using the sapphire method [10].

2.4. Evaluation of the surface integrity and chip morphology

The quality of the machined surfaces was evaluated using a Sensofar PLu NeoxTM optical profiler with a 20x magnification NikonTM confocal objective. The evaluation of the areal surface texture parameters was performed according to ISO 25178-2:2012 standard [11].

Chips were collected, cleaned and then inspected by using a FEITM QUANTA 450 Scanning Electron Microscope (SEM) with the Secondary Electron (SE) probe. Images at different magnifications, namely 80X and 500X, were acquired for each cutting condition. The chip thickness was measured ten times with a micrometer and then the average value was calculated. Afterwards, the chip compression ratio was calculated as the ratio between the chip thickness (t_c) and the uncut chip thickness (t_c), according to Eq. (1):

$$\lambda = \frac{t_c}{t_0} \tag{1}$$

After turning, a portion of the samples was cut, then metallurgical inspected by using the procedure described in § 2.1. The size of the grains was measured every 20 μ m till a distance of 200 μ m below the machined surface.

The Leitz DurimetTM micro-hardness tester was used to perform micro-hardness measurements every 20 μ m from the machined surface to a depth of 200 μ m. 490 mN was applied as a load for 30 s; five values were recorded for measurement and then the average value calculated.

3. Experimental results

3.1. Thermo-physical properties of the nanofluids

The prepared nanofluids were stable and no significant sedimentation was observed even after standing for 24 h.

The calculated density of both the MQL_5 and MQL_15 is 918.29 Kg/m³, which is slightly lower than the one of the pure oil.

Fig. 4 shows the dynamic viscosity of the investigated cutting fluids as a function of the shear rate at 40°C and 100°C. A common trend can be seen for both the testing temperatures: at low shear rates, all the cutting fluids present a shear thinning behavior that converts into a Newtonian one at higher shear rates. In addition, as expected, the viscosity always decreases at increasing temperature. Only the addition of 15 µm size nanoplatelets played a role in modifying the viscosity, contributing to increase it of 7% and 1% at 40°C and 100°C, respectively, compared to the one of the base oil. Whereas, by introducing the 5 µm size nanoplatelets, the viscosity marginally decreased at both the testing temperatures. Reasonably, the conventional cutting fluid presented the lowest viscosity, since it is mainly composed by water. It is worth noting that, in case of flood conditions, it was not possible to draw the curve at 100°C due to the boiling of the water.

Fig. 5 shows the specific heat capacity of the investigated cutting fluids as a function of the temperature. A marginal increase in the specific heat capacity at increasing temperature is appreciable regardless of the lubricating-cooling conditions. However, it can be noticed that the specific heat capacity is lower for the MQL_15 compared to the MQL_5, as a consequence of the nanoplatelets bigger size that allows less heating exchange. A similar trend was reported for silver nanoparticles by Xiong et al. [12], who ascribed the enhanced heat capacity of smaller nanoparticles to the larger atomic thermal vibrational energy of surface atoms.

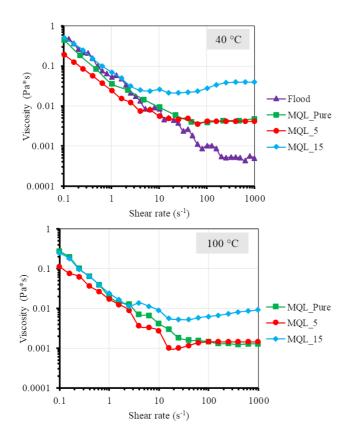


Fig. 4. Dynamic viscosity of the investigated cutting fluids at 40° C and 100° C as a function of the shear rate.

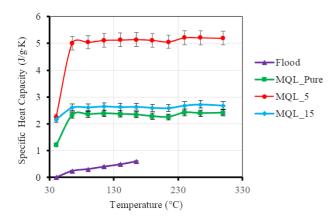


Fig. 5. Specific heat capacity of the investigated cutting fluids as a function of the temperature.

3.2. Machined surface roughness

The effect of the investigated cutting fluids on the surface roughness at varying cutting speed is presented in Fig. 6, where a twofold effect can be seen. First, the surface roughness always increases at increasing cutting speed for all the investigated lubricating-cooling conditions. Further, the effect of the lubricating-cooling condition is visible regardless of the cutting speed. As expected, dry cutting led to the formation of the roughest surfaces at any cutting speed. Whereas, for example at a fixed cutting speed of 130 m/min, the surface roughness reduction is 21% for flood, 7% for pure MQL, 26% for MQL_5 and 4% for MQL_15 compared to dry cutting. Overall, the use of MQL_5 always makes achieve the best surface finish.

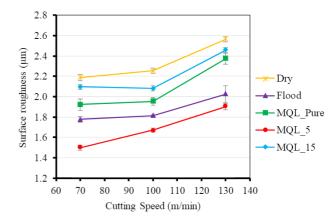


Fig. 6. Surface roughness as a function of the cutting speed and lubricating-cooling conditions.

3.3. Chip morphology

Fig. 7 displays the SEM images of the chips morphology at 130 m/min of cutting speed and varying lubricating-cooling conditions. Overall, spiral and semi-continuous serrated chips were always produced. The chip curling can be attributed to the bi-metallic spring behaviour of the chip surface sliding on the tool rake face and the free one due to the thermal gradient present between them. Therefore, higher temperature gradients lead to curler chips, as proved in the case of dry cutting that provoked the most curled chips (see Fig. 7 a)). This is confirmed also by the fact that the chip helix angles (Ø in Fig. 7 a)) is the lowest in the case of dry cutting, as a consequence of the absence of a hydrodynamic layer between the chip and the tool rake face. Except for the dry condition, no sensible differences in the chip curling degree and helix angle were found when comparing the other lubricating-cooling conditions. Magnified images of the chip underside surface (Zone 1) are reported in Fig. 7 f), g), h), j) and k). Friction tracks, micro particle deposits, adhered material are present regardless of the lubricating-cooling conditions, indicative of the fact that the mechanism of chip formation was not altered by using the nanofluids.

On the contrary, the chip thickness varies as a function of the cutting conditions, as witnessed by the chip compression ratio λ that can be considered a machinability index, being influenced by the nature of the chip-tool interaction, chip contact length and morphology [13]. The values of λ as a function of the cutting speed and lubricating-cooling conditions are reported in Table 1. The lowest value of λ was obtained when using the MQL_5 condition, while the highest value corresponded to dry cutting. On the contrary, a clear trend of the effect of the cutting speed could not be highlighted.

Table 1. Chip compression ratio as a function of the cutting speed and lubricating-cooling conditions.

70 (m/min)	100 (m/min)	130 (m/min)
2.5	2.3	2.5
1.8	2	2
1.9	1.9	2.2
1.7	1.7	1.9
2.2	1.9	2.1
	2.5 1.8 1.9 1.7	2.5 2.3 1.8 2 1.9 1.9 1.7 1.7

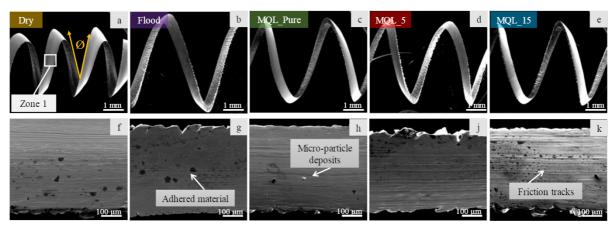


Fig. 7. a), b), c), d), and e): Morphology of the chips obtained at 130 m/min as a function of the lubricating-cooling conditions. f), g), h), j) and k): Magnified images of the chips underside surface as a function of the lubricating-cooling conditions (zone 1).

3.4 Mechanical and microstructural characterization

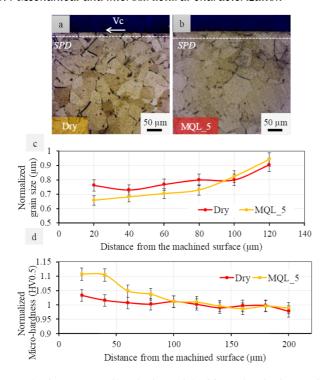


Fig. 8. a) Microstructures, b) grain size, and c) Vickers micro-hardness under the machined surface for the samples machined at 130 m/min.

Fig. 8 a and b shows the optical images of the microstructure of samples machined at 130 m/min under dry and MQL_5 conditions, respectively. Even if only two specific cases are here reported, a grain refinement below the machined surface was recorded in all the samples, regardless of the adopted cutting parameters. The size of the grains gradually increases from the machined surface towards the bulk, attaining the value of the as-delivered material at approximately 120 μm below the machined surface (see Fig. 8 c).

Fig. 8 a and b highlights also the presence of a Severe Plastic Deformed (SPD) layer in which the reduced size grains are deformed towards the cutting direction. The thickness of such layer is higher in the MQL_5 condition compared to the dry case. This can be ascribed to the high temperatures reached during dry cutting which favours thermal softening and reduce

the degree of grains deformation.

In general, strain hardening due to the refinement of the grains leads to a micro-hardness increase up to 100 m below the surface (see Fig. 8 d). However, the use of MQL_5 guaranteed an increment of 7% in micro-hardness compared to dry case near the machined surface. No differences were found between MQL_5 and other cooling conditions, even at different cutting speeds.

4. Discussion

The higher the specific heat capacity the higher the heat dissipation and therefore the cooling at the tool-workpiece interface, whereas the higher the viscosity the lower the machinability as a consequence of the reduced pressure loss at a given flow rate. [14]. Controlling the temperature rise helps keep both the hardness and sharpness of the tool cutting edge. On the contrary, low viscosity values help reduce the friction forces between the chip and the tool. Figs. 4 and 5 show that the MQL_5 presents the best aforementioned characteristics among the cutting fluids under investigation, which is witnessed by the surface roughness results in Fig. 6.

As evidenced in [15], there are other reasons for the efficiency of the nanofluids, besides considering their viscosity and specific heat capacity, namely: i) the rolling mechanism thanks to which the nanoparticles act as a nano-ball bearing reducing friction at the chip-tool interface; ii) the protective effect thanks to which the nanoparticles likely deposit on the generated surface forming a protecting layer; iii) the mending effect played by the nanoparticles that can easily fill scars and groove of the machined surface; iv) the polishing effect thanks to which the roughness of the machined surface is reduced by the nanoparticle-assisted abrasion. Nevertheless, the results reported in this study evidenced that the size of the nanoparticles plays a fundamental role in affecting the performance of the nanofluids. In fact, even if the MQL 15 is characterized by a higher viscosity than the one of the pure oil, its specific heat capacity does not increase significantly, making its performances lower than the ones of the MQL Pure. A further comparison between the results of the MQL Pure and MQL 15 shows that the importance of a low viscosity prevails at lower cutting speeds, whereas, the specific heat capacity becomes more important at the highest cutting speed, since the

surface roughness values get closer at highest cutting speed. Fig. 6 shows that the surface roughness of the Inconel 718 workpieces machined under MQL condition was higher than that obtained with the conventional flood method. The better performances of the MQL compared to the ones of the conventional flood is still a controversial issue in literature. As example, different machining conditions were applied to machine the Inconel 718 alloy in [16], showing that the MQL had the ability to lower the surface roughness and cutting force by 38 and 59%, respectively, compared to conventional flood. On the contrary, the ineffective cooling performances of the MQL were highlighted when high speed machining of Inconel 718 in [17]. In this study, the conventional flood performed better than the MQL Pure, because, even if the former is characterized by lower specific heat capacity compared to the latter, it was sprayed with a higher flow rate, which contributed to limit the temperature rise in the cutting zone.

Data of the chip compression ratio (see Table 1) show that the thickest chips were obtained under dry cutting while the thinnest ones in the case of MQL_5. A reduced chip thickness is a consequence of a larger shear angle, induced, in turn, by a reduced friction coefficient, therefore further proving the lubrication efficiency of the MQL_5.

Fig. 8 a, b and c show a grain refinement together with the formation of a SPD layer near the surface were observed, regardless of the machining conditions. This leads to strain hardening near the machined surfaces, according to the results of [18]. These metallurgical alterations are due to intense thermo-mechanical loads that occur during turning. Since the mechanical counterpart is the same at fixed cutting parameters, the surface integrity changes obtained with different cooling conditions can be mainly attributed to the thermal load. The use of MQL 5, which inhibits temperature rises, leads to the formation of harder surface characterized by thicker extension of the SPD layer compared to the dry case. In addition, it is shown that the application of both nanofluids as well as pure MQL led to the same microstructural and mechanical alterations of flood condition, which still represents the most currently used lubricating-cooling technique.

5. Conclusions

The performances of different cutting fluids were evaluated in terms of obtainable surface integrity and chip morphology when machining Inconel 718. These cutting fluids were characterized in terms of viscosity as a function of the temperature and shear rate as well as specific heat capacity up to 300° C.

The following conclusions were reached:

- The introduction of 5 μm size graphene nanoplatelets into the vegetable oil contributed to increase the nanofluid specific heat capacity without increasing its viscosity; on the contrary, opposite results were achieved when adding 15 μm size particles.
- The application of the MQL_5 resulted in the best surface roughness thanks to its improved efficiency as metalworking fluid. On the contrary, the MQL_15 did not improve the surface finish compared to the MQL_Pure, which also worsened it compared to the conventional flood condition.

- The chip compression ratio data showed that thinnest chips were obtained when applying the MQL_5, whereas the highest were obtained in the case of MQL 15.
- All the samples showed a grain size refinement and the presence of a SPD layer, whose extension depended on the adopted cooling conditions.
- All the samples showed hardening in proximity of the surface. However, softer surfaces were obtained under dry condition compared to the ones obtained under all the other lubricating-cooling conditions.

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