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Preparation and Characterization of TiO_2 Nanofluid by Sol-gel Method for Cutting Tools

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Nanofluid; Nanoparticle; Sol-gel method; Milling; Cutting tools; TiO_2

Abstract

In the past few decades, rapid advances in nanotechnology have lead to emerging of new generation of coolants called as nanofluids. Nanofluids are defined as suspension of nanoparticles in a basefluid. Machining experiences high temperatures due to friction between the tool and workpiece, thus influencing the workpiece dimensional accuracy and surface quality. Further, the cutting fluids also incur a major portion of the total manufacturing cost. Nanofluids are containing oxides including MgO, TiO_2, ZnO, Al_2O_3 and SiO_2 nanoparticles. Nanofluids are produced by dispersing nanometer scale solid particles into base liquids such as water, ethylene glycol or oil. In this study, TiO_2 nanoparticles were produced by using sol-gel technique and TiO_2 the nanoparticles were suspended in a specified volume of the base fluid. Regarding the analysis of materials, phase analysis was determined by using X-ray diffactometer (XRD) and the average particle size distribution (APS) using particle size analyzer. The present study summarizes the effect of nanofluid with TiO_2 nanoparticles on the performance of the cutting tools.

Kesici Takımlar İçin Sol-jel Metoduyla TiO_2 Nanosıvıların Hazırlanması ve Karakterizasyonu

Anahtar kelimeler
Nanosıvı; Nanopartikül; Sol-jel yöntemi; İşleme; Kesme takımları; TiO_2

Özet

Yakın zamanda nanoteknolojideki hızlı gelişme nanosıvılar olarak isimlendirilen yeni soğutma sıvılarının ortaya çıkışına neden olmuştur. Nanosıvılar baz sıvı içerisindeki nanopartikül süspansiyonları olarak tanımlanabilir. Takım ve iş parçası arasındaki sürtünmeden dolayı işlemede yüksek sıcaklık meydana gelmektedir bu durum iş parçasının yüzey kalitesi ve hassaslığını etkilemektedir. Buna ilave olarak kesme sıvıları toplam üretim maliyetinin büyük kısmını oluşturmaktadır. Nanosıvılar; MgO, TiO_2, ZnO, Al_2O_3 ve SiO_2 gibi nanopartükülleri içeren oksitlerden oluşmaktadır. Nanosıvılar, nanometre ölçeğindeki katı partiküllerin su, etil glikol veya yağ gibi baz sıvı içerisinde dağılması elde edilmektedir. Bu çalışmada, TiO_2 nanopartiküller sol-jel tekniği ile üretilmiştir ve belirlenen hacimde baz sıvı içerisinde süspansiyon haline getirilmiştir. Malzemelerin karakterizasyonunda ise faz analizi X-Işınları difraksiyonu cihazı ile ortalama partikül boyutu partikül boyutu analiz cihazı ile belirlenmiştir. Çalışma, TiO_2 nanopartiküllerini içeren nanosıvıların kesme takımlarının performansına etkisini özetlemektedir.

1. Introduction

Though exploits of nanotechnology mostly concerns engineering solids either for functional (electronic, magnetic, optical, catalytic, etc.) or structural (strength, hardness, wear/abrasion resistance, etc.) applications, the concept of nanofluid is rather new (Manna, 2009). Nanofluids are a new class of fluids engineered by dispersing nanometer-sized materials (nanoparticles, nanofibers, nanotubes, nanowires, or nanorods) in base fluids. In other words, nanofluids are nanoscale colloidal suspensions containing consendensed nanomaterials. Nanofluids have been found to possess enhanced thermophysical properties such as thermal conductivity, thermal diffusivity, viscosity and convective heat transfer coefficients compared to those of base fluids like oil or water. It has demonstrated great potential applications in many fields. (Tavman and Turgut 2010). The main driving force for nanofluids research lies in a wide range of applications. Although some review articles involving the progress of nanofluid investigation were published in the past several years, most of the reviews are concerned on the experimental and theoretical studies of the thermophysical properties or the convective heat transfer of nanofluids (Tavman and Turgut 2010).

Nanofluid manufacture involves dispersing metallic and non-metallic nanomaterials with high thermal conductivity, into a suitable “working fluid” such as engine oil, water, ethylene glycol, etc., to enhance the heat transfer performance of traditional fluids. According to literature reports, the thermal conductivity of a nanofluid is strongly dependent on the volume fraction and properties of the added nanoparticles. In addition, for the addition of a given volume of particles, the solid-liquid surface contact area between nano-scale particles and the suspension fluid is greater than that for micro-scale particles. Hence, the size and shape of the particles added will have a significant effect on thermal conductivity and heat transfer characteristics (Teng, 2011).

From these reviews, the following key features of nanofluids have been found: (i) they have larger thermal conductivities compared to conventional fluids; (ii) they have a strongly non-linear temperature dependency on the effective thermal conductivity; (iii) they enhance or diminish heat transfer in single-phase flow; (iv) they enhance or reduce nucleate pool boiling heat transfer; (iv) they yield higher critical heat fluxes under pool boiling conditions. Researchers have given much more attention to the thermal conductivities of nanofluids than to the resulting heat transfer characteristics (Rao *et al.* 2005).

Materials used for nanoparticles include chemically stable metals (e.g., gold, silver, copper), metal oxides (e.g., alumina, zirconia, silica, titania) and carbon in various forms (e.g., diamond, graphite, carbon nanotubes, fullerene). Nanoparticles are relatively close in size to the molecules of the base fluid and thus, if properly prepared, can realize very stable suspensions with little erosion and gravitational deposition over long periods of time (Jahanshahi *et al*. 2010).

Titanium dioxide is widely used as TiO_2 nanoparticles and has a large variety of potential applications in biomedical, optical, and electronic fields. Due to their small size, nanoparticles have a very high surface area to volume ratio and are thus of great scientific interest as they are a bridge between bulk materials and atomic or molecular structures. The properties of materials change as their size decreases to nanoscale and the proportion of surface atoms becomes significant. One of these properties, the surface ionization of titanium dioxide nanoparticles in contact with an electrolytic solution, has been studied extensively (Leroy *et al.* 2011).

TiO_2 can be produced by several methods such as chemical vapor deposition precipitation, magnetron sputtering, hydrothermal synthesis, sol-gel, flame spray pyrolysis (Xiao *et al.* 2006; Celik *et al.* 2007; Chaisuk *et al.* 2011) and so on. Among them the sol–gel technique is a powerful method which is employed by researchers to prepare titania in the form of powder, thin films and porous materials. An advantage of the sol–gel technique over other methods, such as hydrothermal synthesis and chemical vapour deposition, is that it produces materials with a high surface area (Monreal *et al.* 2009).

Machining experiences high temperatures due to friction between the tool and workpiece, thus influencing the workpiece dimensional accuracy and surface quality. Machining temperatures can be controlled by reducing the friction between tool-workpiece and tool-chip interface with the help of effective lubrication. Cutting fluids are the conventional choice to act as both lubricants and coolants. But, their application has several adverse effects such as environmental pollution, dermatitis to operators, water pollution and soil contamination during disposal. Further, the cutting fluids also incur a major portion of the total manufacturing cost. The applicability of the fluids as coolants is mainly due to the enhanced thermal conductivity of the fluids due to the solid particle inclusion (VamsiKrishna *et al.* 2010).

A cutting fluid for MQL should be selected not only on the basis of primary characteristics (cutting performance) but also of its secondary characteristics, such as biodegradability, oxidation stability, and storage stability. Those processes, in which the friction and adhesion play a dominant role, generally require the usage of minimal quantities of fluid. MQL refers to the use of cutting fluids of only a minute quantity, which are about three to four orders of magnitudes lower than that used in flooded lubricating conditions (Dhar, 2006).

There are reports, which indicate that MQL in an end-milling process is very much effective (Lopez de Lacalle *et al.* 2001; Rahman *et al.* 2001). This is considered to be because lubricant can reach the tool face more easily in milling operations compared with other cutting operations. MQL with rapeseed oil has only a small lubricating effect in light loaded machining conditions (Itoigawa *et al.* 2006).

In the present research, TiO_2 nanoparticles were produced by sol-gel method for nanofluid application. Afterwards, their machining performance was evaluated.

2. Experimental Studies

Sol-gel-derived nano-powders of TiO_2 solution were prepared from titanium (IV) isopropoxide ($Ti[OCH(CH_3)_2]_4$, Acros) precursor. The precursor was dissolved in xylene ($C_6H_5CH_2CH_3$, Carlo Erba). Glacial acetic acid was used as a chelating agent. The metal concentration of the solutions was set as 0.68 M. Homogeneous solution was obtained by magnetic stirring for 15 minutes at room temperature. In order to chelate the gelation reactions few drops of glacial acetic acid was added in to sol-gel solution. This solution was kept at 70^{o}C for gelation process for 30 minutes and then the obtained xerogel was dried at 100 ^{o}C for 15 minutes on a hot plate. Finally, the dry gel powder was eventually calcined at 530 ^{o}C for 1 hour in air to obtain sol-gel assisted crystalline nanopowders.

X-ray diffractometer (XRD, Rigaku, D/MAX-2200/PC) with a Cu-Kα irradiation, wavelength λ=0.15418nm was used to identify the phase structures. TiO_2 powders were dispersed in acetone media in order to determine the average particle size distribution (APS) using particle size analyzer (Zetasizer Nano ZS) which uses light scattering technique to measure hydrodynamic size of nanopowders.

The TiO_2 nanofluids were produced by dispersing TiO_2 nanoparticles into the base fluid, distilled water (DI water). It had a spherical shape with a mean diameter of approximately 500 nm. Prior to each test, the TiO_2 nanofluids were processed in an ultrasonic bath for 90 minutes to break any possible aggregations of TiO_2 nanoparticles and to keep the nanofluids uniformly dispersed. The dispersing method could ensure that the nanofluids were stable for more than 24 hours without any visible sedimentations and agglomerations. After the TiO_2 nanofluid samples were prepared, they were charged into the test cell for the machining tests.

Milling operations were performed on 7075 aluminium alloys. Cutting speeds up to 350 m/min were employed. The main objective of the present study was to analyze the effect of the coolant environment on tool wear, cutting forces and surface quality of the work-piece during miling operation. The tests were performed using uncoated carbide inserts under three different coolant environments of dry cutting, MQL and flooded coolant conditions. MQL was applied at two rates of 30 ml/h and 70 ml/h. A fully synthetic water soluble coolant (Ecocool S-CO5), containing glycol as a lubricating agent and free of chlorine and mineral oil [Product catalogue, Cutting Fluids, FUCHS 12], was used as the flood coolant in a volumetric concentration of 1:20. As coatings act as a barrier between tool and component and they posses high heat resistance to aggravate the effects of lack of coolant, uncoated carbide inserts (axial rake angle = +5°, helix angle -5°, nose radius of the insert r = 0.4) were selected.

The Vertical Machining Centers with a Taksan TMC 500 electronic control unit was driven by a tri-phasic asynchronous engine. Preliminary experiments were conducted to determine the machining parameters and coolant quantities. From these experiments the depth of cut and feed rate were fixed at 1.0 mm and 0.15 mm/rev respectively.

Tool wear was measured with a Nikon Eclipse ME600 optical microscope and an Omis mini optical measurement inspection system toolmaker's microscope.

Surface roughness measurements were performed by using a Mitutoyo Surftest-B Profilometer with a cut-off length of 0.8 mm and sampling length of 5 mm. Cutting forces were measured with a Kistler three-component dynamometer 9257B linked via a multichannel charge amplifier (type Kistler 5019B) to a chart recorder.

3. Results and Discussion

XRD patterns of the sol-gel derived powders were depicted in Figure 1, which represents typical peaks of anatase TiO_2 and rutile TiO_2. The patterns clearly show the presence of nanocrystalline anatase structure, inasmuch as five high-intensity crystal peaks at 2θ = 25.2–55.1° could be observed and indexed as (101), (004), (200), (105), and (211), respectively (JCPDS, No. 21-1272). In addition, the crystal peaks at 2θ= 27.5 - 54.4° correspond to the (110), (101), (211) planes of the rutile phase (JCPDS 21-1276). By using XRD phase analysis data, anatase/rutile phase ratios were calculated using Rietweld Method. According to the calculations, approximate ratios were found to be as 97.2/2.8 and 87.2/12.8 as given in Table 1.

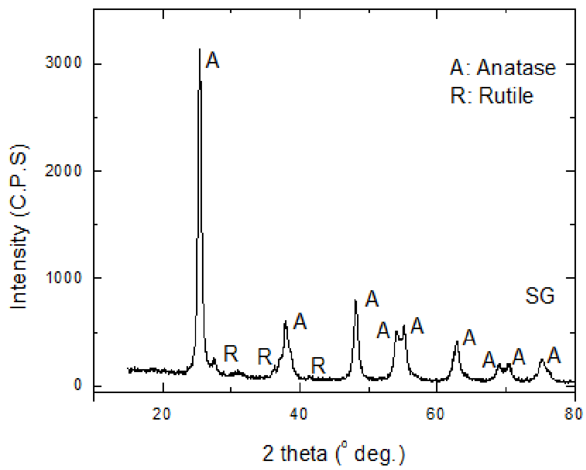


Figure 1. XRD patterns of TiO_2 powders produced via sol-gel methods.

Tablo 1. Some physical and structural properties of TiO_2 powders.

Method	Average particle size (nm)	Specific surface area (m^2/g)	Phase ratios (Anatase /Rutile) (wt%)
SG	541	14.7*	97.2 / 2.8

Correlation coefficient for multipoint BET analyze *0.999944 and **0.999981

Particle size distribution of the produced powders was represented in Figure 2. According to size distribution plots average size is 540 nm; 80 % of the particles are between 477 and 641 nm. This indicates a wide size distribution for sol-gel technique. One must note that the particle size determined by the Zetasizer is the hydrodynamic diameter, which is detected by the scattered laser light and influenced by the viscosity and concentration of the solution (Wu *et al.* 2007).

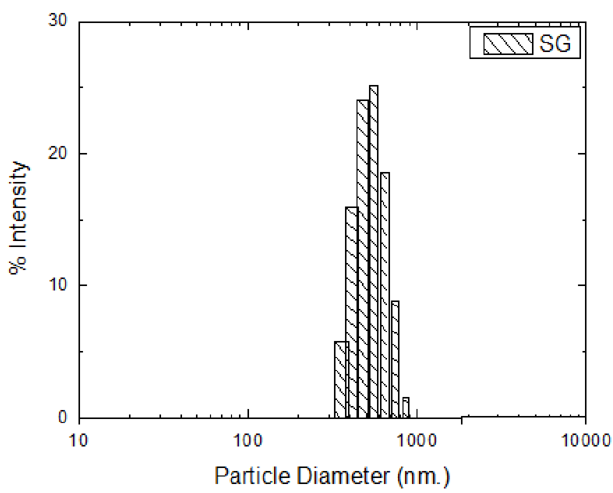
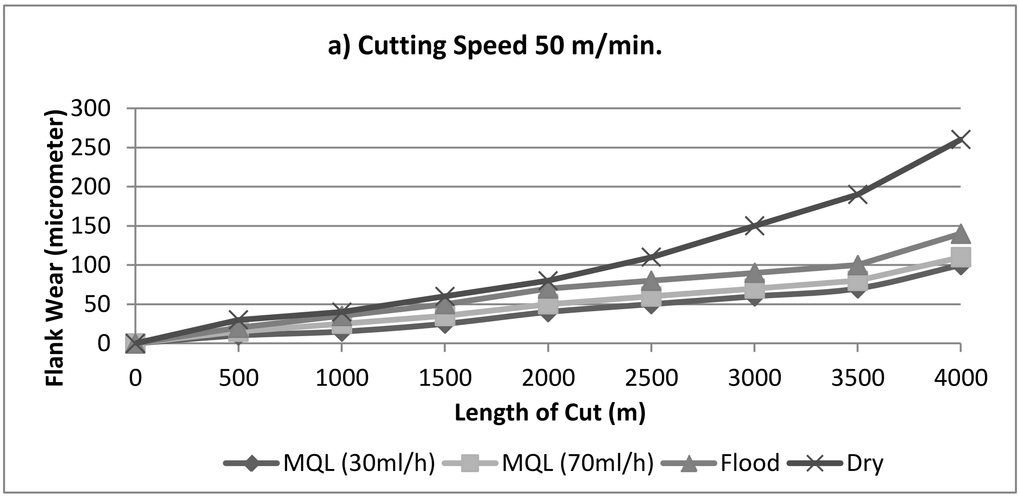


Figure 2. Particle size distribution of TiO_2 powders

Figure 3 shows the development of flank wear curves obtained for nanofluid, flood and dry machining operated at various cutting speeds are given in Figure 4 respectively. It was observed that during milling with nanofluid lubrication, flank wear was observed to increase gradually when lower cutting speeds of 50 m/min were employed



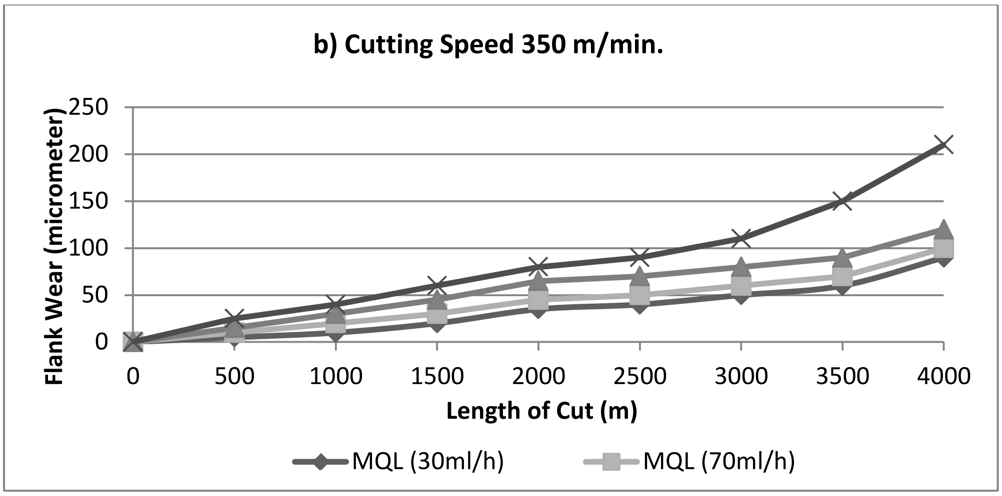


Figure 3. Change in flank wear VB with machining distance at two different cutting speeds.

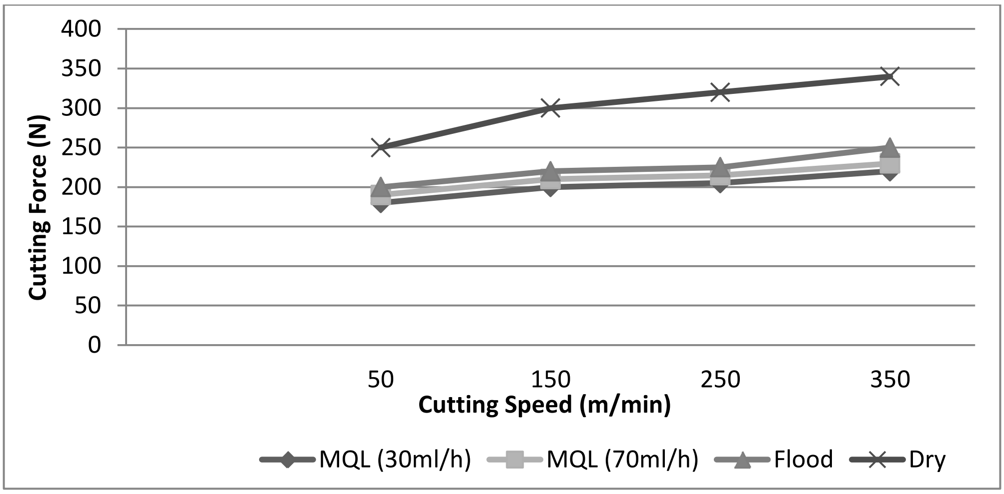


Figure 4. Relationship between the resultant cutting forces and cutting speeds measured under various machining environments.

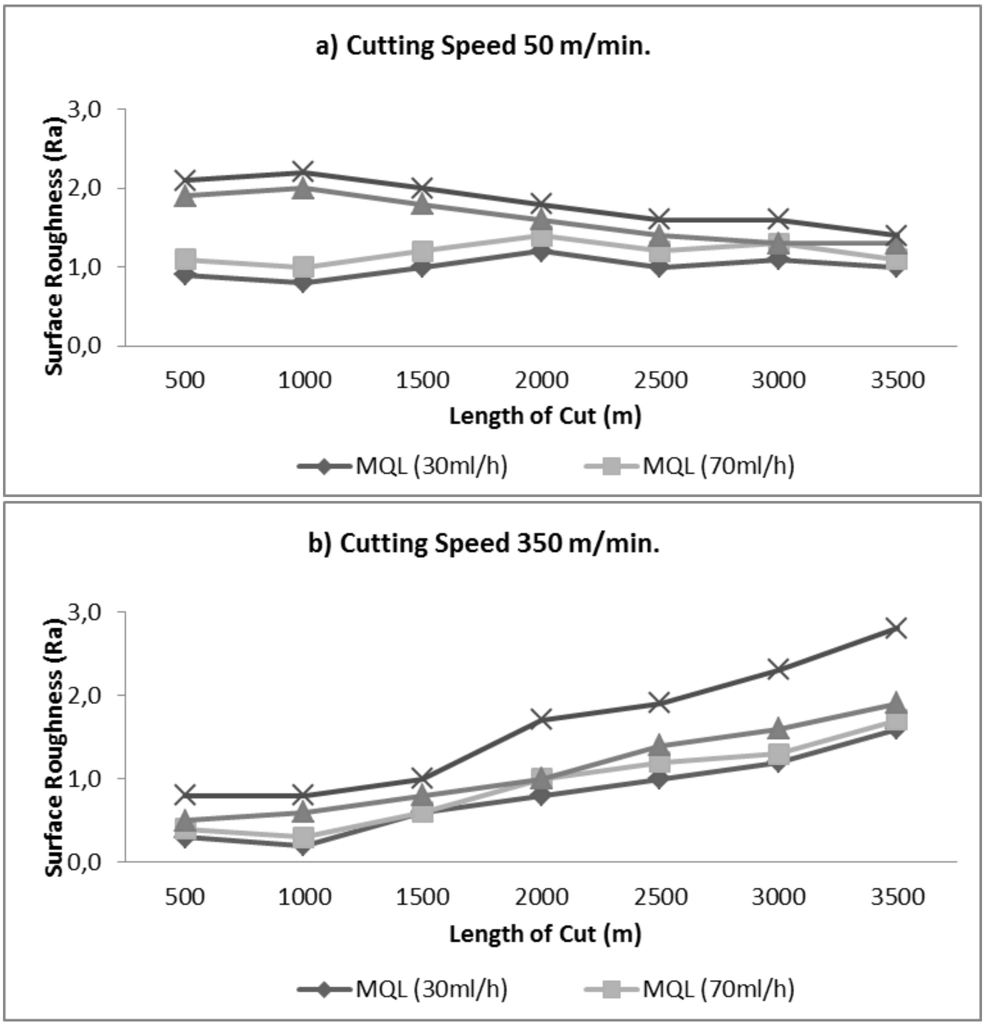


Figure 5. Surface roughness (Ra) values of the work-piece measured parallel to the feed direction.

However, at higher cutting speeds of 350 m/min, the tool worn quite rapidly, resulting in a shorter tool life.

In contrast to the nanofluid lubrication processes, the dry machining worn very rapidly at all cutting speeds tested. Both nanofluid lubrications experienced similar modes of failure throughout the trials. These were non-uniform flank wear and chipping.

Figure 4 shows the relationship between the resultant cutting forces and cutting speeds measured under various machining environments.

It can be seen that the lowest cutting force was obtained using the nanofluid followed by flood (Ecocool S-CO5), whereas highest forces were produced when dry machining, which was an unexpected result, since the higher cutting temperatures generated when machining without cutting fluid would cause the softening of the work material, thus reducing the cutting force. A reason for this behaviour may reside in the high thermal conductivity of aluminium, which would rapidly remove the heat from the cutting zone and lead to the temperature rise. The best performance of the cutting fluid containing nanoparticle as additive may be attributed to the low cutting temperatures achieved when machining aluminium alloys.

Figure 5 shows the surface roughness values (R_a) of the final pass when the tool criteria were met for each test. It is evident that nanofluid lubrication produced better surface finish at most cutting speeds when compared to flood and dry machining. The R_a values obtained for nanofluid lubrication lied between 0.7 and 1.0 µm, while for the dry machining the range was between 0.7 and 1.3 µm for cutting speed of 50 m/min. The wear pattern of the nanofluid lubrication may have an influence on the surface roughness since the damage on the cutting edge was less when compared to dry machining. Results also showed that cutting speed affects the surface finish of the pass. All the processes produced lower R_a values at higher cutting speeds. The R_a value was slightly increased when cutting speed was reduced to 50 m/min for all them.

4. Conclusion

In summary, nano scale TiO_2 powders were successfully produced using sol-gel process. There is a need to develop a simple and efficient method to obtain TiO_2 nanoparticles with a narrow size distribution and high crystallinity. The produced powders were characterized in order to determine phase structure, particle size distributions and surface properties for cutting performances. 7075 aluminum alloy has been machined under different conditions of dry, MQL and flooded coolant/lubricant using uncoated carbide inserts. The process of machining was successful. The performance of the nanofluid, flood and dry machining was studied when face milling 7075 aluminium alloy.

The nanofluid lubrication performed better than the flood and dry machining at the all the cutting speeds. Lower feed forces were obtained using the highest cutting speed (350 m/min) and the lowest feed rate (0.15 mm/rev). Using the highest cutting fluid flow rate (70 ml/h) resulted in lower feed forces only at higher cutting speeds and feed rates. Specific cutting pressure was reduced as cutting speed and feed rate were increased, but increased with cutting fluid flow rate. As far as the surface roughness is concerned, lower R_a values were obtained at higher cutting speeds and lower feed rates, but were not significantly affected by cutting fluid flow rate. Under all the cutting condition, the cutting fluid containing nanoparticle as extreme pressure additive produced lower cutting forces and better surface finish.

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References

Celik, E., Keskin, I., Kayatekin, I, Azem, F. A., Özkan, E., 2007. Al_2O_3–TiO_2 thin films on glass substrate by sol–gel technique. Materials Characerization, **58** 349–357.

Chaisuk, C., Wehatoranawee, A., Preampiyawat, S., Netiphat, S., Shotipruk, A., Panpranot, J., Jongsomjit, B., Mekasuwandumrong, O., 2011. Preparation and characterization of CeO_2/TiO_2 nanoparticles by flame spray pyrolysis. Ceramics International, **37**, 1459–1463.

Dhar, N.B., Kamruzzamman, M., Ahmed, M., 2006. Effect of minimum quantity lubrication (MQL) on tool wear and surface roughness in turning AISI- 4340 steel. *Journal of Materials Processing Technology*, **172**, 2, 299-304.

Itoigawa, F., Childs, T.H.C, Nakamura, T., Belluco, W., 2006. Effects and mechanisms in minimal quantity lubrication machining of an aluminium alloy. *Wear* **260**, 3, 339-344.

Jahanshahi, M., Hosseinizadeh, S.F., Alipanah, M., Dehghani, A., Vakilinejad, G.R., 2010. Numerical simulation of free convection based on experimental measured conductivity in a square cavity using Water/SiO_2 nanofluid. *International Communications in Heat and Mass Transfer*, **37**, 687-694.

Leroy, P., Tournassat, C., Bizi, M., 2011. Influence of surface conductivity on the apparent zeta potential of TiO_2 nanoparticles, *Journal of Colloid and Interface Science*, **356**, 442–453.

Lopez de Lacalle, L.N., Lamikiz, A., Sanchez, J.A., Cabanes, I.,2001. Cutting conditions and tool optimization in the high speed milling of aluminium alloys. *Proceedings of the IMechE, Part B: Journal of Engineering*, **215**, 1257–1269.

Manna, I., 2009. Synthesis, Characterization and Application of Nanofluid-An Overview. *Journal of the Indian Institute of Science*, **89**, 21-33.

Monreal, H.A., Chacon-Nava, J.G., Arce-Colunga, U., Martınez, C.A., Casillas, P.G., Martinez-Villafane, A., 2009. Sol–gel preparation of titanium dioxide nanoparticles in presence of a linear polysaccharide. *Micro & Nano Letters*, **4**, 187-191.

Product catalogue, Cutting Fluids, FUCHS.

Rahman, M., Senthil Kumar, A., Manzoor-Ul-Salam, 2001. Evaluation of minimal quantities of lubricant in end milling. *International Journal of Advanced Manufacturing Technology*,**18**, 235-241.

Rao. K., El-Hami, K., Kodaki, T., Matsushige, K., Makino, K., 2005. A novel method for synthesis of silica nanoparticles. *Journal of Colloid and Interface Science*, **289**, 125–131.

Tavman, I. and Turgut, A., 2010. *Microfluidics Based Microsystems: Fundamentals and Applications*. S. Kakaç et al. (eds.), Springer Science + Business Media, 139-162.

Teng, T., 2011. Preparation and characterization of carbon nanofluid by a plasma arc nanoparticles synthesis system. *Nanoscale Research Letters*, **6**, 293.

VamsiKrishna, P., Srikant, R.R., NageswaraRao, D., 2010. Experimental investigation on the performance of nanoboric acid suspensions in SAE-40 and coconut oil during turning of AISI1040 steel. *International Journal of Machine Tools & Manufacture,* **50**, 911-916.

Wu, Y. L., Lim, C. S., Fu, S., Tok, A.Y., Lau, H.M,. Boey, F.Y.C, .Zeng, X. T, 2007. Surface modifications of ZnO quantum dots for bio-imaging. Nanotechnology, **18**, 1-9.

Xiao, J.R., Peng, T.Y., Li, R., Peng, Z.H., Yan, C.H., 2006. Preparation, phase transformation and photocatalytic activities of cerium-doped mesoporous titania nanoparticles. *Journal of Solid State Chemistry*, **179**, 1161-1170.