

## Preparation and Characterization of SiO<sub>2</sub> Nanofluid by Flame Spray System for Machining Process

N. Funda Ak AZEM<sup>1,\*</sup>, Işıl BIRLIK<sup>1</sup>, Recep YIĞIT<sup>2</sup>, Mustafa EROL<sup>1,3</sup>, Serdar YILDIRIM<sup>1,3,4,5</sup>, Erdal ÇELİK<sup>1,4,6</sup>

<sup>1</sup>Dokuz Eylül University, Department of Metallurgical and Materials Engineering, Buca, Izmir

<sup>2</sup>Dokuz Eylül University, Izmir Vocational School, Programs of Technique, Buca, Izmir

<sup>3</sup>Dokuz Eylül University, The Graduate School of Natural and Applied Sciences, Buca, Izmir

<sup>4</sup>Dokuz Eylül University, Center for Production and Applications of Electronic Materials, Buca, Izmir

<sup>5</sup>TEKNOBİM Nanoteknolojileri Araştırma ve Geliştirme Dezenfektan San. Tic. Ltd. Şti., Urla, Izmir

<sup>6</sup>Dokuz Eylül University, Graduate School Natural and Applied Sciences, Department of Nanoscience and Nanoengineering, Buca, Izmir

\*e-posta: funda.ak@deu.edu.tr

Geliş Tarihi:22.10.2012; Kabul Tarihi: 11.11.2013

### Abstract

Nanofluids are a relatively new class of fluids which consist of a base fluid with nano-sized particles suspended within them. Nanofluids showed a large enhancement of thermal conductivity in comparison with their base fluids. 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. These particles that used in nanofluid applications, generally a metal or metal oxide, increase conduction and convection coefficients, allowing for more heat transfer out of the coolant. In this study, SiO<sub>2</sub> nanoparticles were produced by using flame spray method and they were suspended in a specified volume of the base fluid. Regarding the analysis of materials, phase analysis was determined by using X-ray diffractometer (XRD) and the average particle size distribution (APS) using particle size analyzer. Besides, successful synthesis of the SiO<sub>2</sub> nanoparticles were confirmed using Fourier transform infrared spectroscopy (FT-IR). The present work shows the effect of nanofluid with SiO<sub>2</sub> nanoparticles on the performance of the cutting tools.

### Key words

Nanofluid;  
Nanoparticle; Flame  
Spray Method; Milling;  
Cutting tools; SiO<sub>2</sub>

## Mekanik İşleme Prosesi Alev Sprey Sistemiyle SiO<sub>2</sub> Nanosivuların Hazırlanması ve Karakterizasyonu

### Özet

Nanosivular nano-boyutlu partiküllerin (1-100 nm) süspansiyon halinde olduğu baz sıvısından oluşan yeni tip sıvılardır. Baz sıvıları ile karşılaştırıldığında nanosivular termal iletiminde büyük iyileştirme göstermektedir. Takım ve iş parçası arasındaki sürtünmeden dolayı işlemede yüksek sıcaklık meydana gelir bu durum iş parçasının yüzey kalitesi ve hassaslığını etkilemektedir. Buna ilave olarak, kesme sıvıları toplam üretim maliyetinin büyük kısmını oluşturmaktadır. Nanosivü uygulamalarında kullanılan partiküller genelde metal veya metal oksit olup, ısı iletim ve yayılım katsayılarını arttırmakta böylece soğutucu akışkandan daha fazla ısı transferi olmasını sağlamaktadırlar. Bu çalışmada, SiO<sub>2</sub> nanopartiküller alev püskürtme tekniği ile üretilmiş ve belirlenen hacimde baz sıvı içerisinde süspansiyon haline getirilmiştir. Malzemelerin karakterizasyonu ise, faz analizi X-ışınları difraksiyonu cihazı ile ortalama partikül boyutu partikül boyutu analiz cihazı ile belirlenmiştir. Buna ilave olarak, SiO<sub>2</sub> nanopartiküllerin başarı ile üretimi Fourier Dönüşümlü Kızılötesi spektrometresi (FT-IR) ile doğrulanmıştır. Çalışma, SiO<sub>2</sub> nanopartiküllerini içeren nanosivuların kesme takımlarının performansına etkisini özetlemektedir.

### Anahtar kelimeler

Nanosivü; Nanopartikül;  
Alev püskürtme;  
İşleme; Kesme  
takımları; SiO<sub>2</sub>

© Afyon Kocatepe Üniversitesi

### 1. Introduction

In the past few decades, rapid advances in nanotechnology have lead to emerging of new generation of coolants called "nanofluids". Nanofluids are a relatively new class of fluids which

consist of a base fluid with nano-sized particles suspended within them. These particles, generally a metal or metal oxide, increase conduction and convection coefficients, allowing for more heat transfer out of the coolant. 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. (Saidura *et al.* 2011; Tavman and Turgut, 2010). Nanofluids are dilute suspensions of functionalized nanoparticles composite materials developed about a decade ago with the specific aim of increasing the thermal conductivity of heat transfer fluids, which have now evolved into a promising nanotechnological area. Such thermal nanofluids for heat transfer applications represent a class of its own difference from conventional colloids for other applications (Saidura *et al.* 2011; Manna, 2009).

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). Silica nanoparticles occupy a prominent position in scientific research, because of their easy preparation and their wide uses in various industrial applications, such as catalysis, pigments, pharmacy, electronic and thin film substrates, electronic and thermal insulators, and humidity sensors. The quality of some of these products is highly dependent on the size and size distribution of these particles (Sreenivasa *et al.* 2005).

At present, nanoscale silica materials are prepared using several methods, including vapor-phase reaction, sol-gel and thermal decomposition techniques (Singh *et al.* 2008). Flame Spray Pyrolysis technique (FSP) has many advantages over the other methods as it is low-cost, easy to control particle size, simple processing, high production yield and ease of conversion to mass manufacturing (Tok *et al.* 2006).

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).

When considering machinability parameters, the use of coolant is indispensable in metal cutting operations. However, stricter environmental regulations are making the use of an ample amount of conventional coolant impossible because of its negative impact on the environment. Consequently, the use of minimal quantities of lubricant (MQL) can be regarded as an alternative solution in which the functionality of cooling and lubrication can be achieved by a tiny amount of cutting oil (Rahman *et al.* 2001).

This paper reports on a novel flame spray pyrolysis method to produce nano-sized silica particles for nanofluid application and then their machining performance was evaluated.

## 2. Material and Method

Precursor solution was prepared by dissolving appropriate amounts of Tetraethyl orthosilicate (C<sub>8</sub>H<sub>20</sub>O<sub>4</sub>Si, Acros) in ethanol. The metal concentration of the solution was set as 1 M. Homogeneous solution was obtained after stirring 15 min at room temperature on a magnetic stirrer. The obtained solution was fed into the precursor syringe of flame spray pyrolysis device (TETHIS, Np10). In order to clarify the process some parameters must be identified. In a typical run of the flame spray reactor, the liquid precursor mixture was fed into the center of a methane/oxygen flame by a syringe pump (at precursor feed rate of 5 ml/min in this experiment) and dispersed by oxygen (Linde Industrial Gases Co.), to forming the fine spray. At the nozzle, the pressure drop at the capillary tip was kept constant at 2 bar by adjusting the orifice gap area. The spray flame was ignited by a smaller flame ring issuing from an annular gap. This premixed methane/oxygen (Linde Industrial Gases Co.) supporting flame was kept constant with flow rate of 5 L/min and fuel/oxygen ratio of 1.5/3 L methane/L oxygen. A sintered metal plate ring provided an additional oxygen sheath flow (5 L/min) surrounding the supporting flame. Particle products were collected on a cellulosic filter with the aid of a vacuum pump.

Particle size distribution was measured using Malvern Zeta Sizes Nano S90 which uses light scattering technique to measure hydrodynamic size of nanopowders. In order to eliminate random errors at least five measurements were made at 25°C. The bond integrity is monitored by Fourier Transform Infrared (FTIR) spectroscopy (Perkin Elmer) from 650 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>. In order to get accurate element distribution and determine the possible phase on surface, XPS and XRD analysis were performed. X-ray diffractometer (XRD, Rigaku, D/MAX-2200/PC) with a Cu-K<sub>α</sub> irradiation, wavelength  $\lambda=0.15418$  nm was used to identify the phase. Nanoparticles element distribution is studied by X-ray photoelectron spectroscopy (XPS) (K-alpha, Thermo).

The SiO<sub>2</sub> nanofluids were produced by dispersing SiO<sub>2</sub> nanoparticles into the base fluid, distilled water (DI water). It had a spherical shape with a mean diameter of approximately 213 nm. Prior to each test, the SiO<sub>2</sub> nanofluids were processed in an ultrasonic bath for 90 minutes to prevent aggregations of SiO<sub>2</sub> 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 SiO<sub>2</sub> 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 milling 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

The produced SiO<sub>2</sub> nano powders were dispersed in water in order to determine particle size using particle size analyzer (Zetasizer Nano ZS) which uses light scattering techniques to measure hydrodynamic size of nanoparticles. Particle size distribution of the produced powders was represented in Figure 1. According to size distribution approximate particle size was found to be in the range of 70–500 nm and the average particle size was about 213 nm. The produced particles can be identified as monodispersed due to small particle size distribution range.

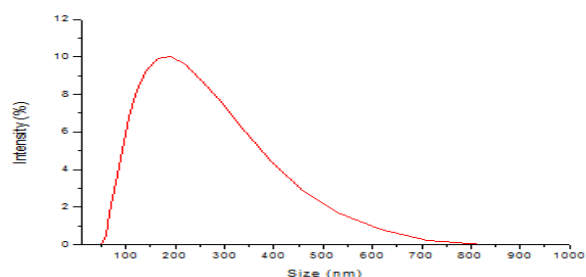


Figure 1. Particle size distribution of SiO<sub>2</sub> nanopowders

Figure 2 shows the FT-IR spectrum of SiO<sub>2</sub> nanoparticles the strong peak at 1100 cm<sup>-1</sup> is observable, indicating Si–O–Si bonding in SiO<sub>2</sub> nanoparticle. It is clear from this graph that there are no organic groups in SiO<sub>2</sub> nanoparticles.

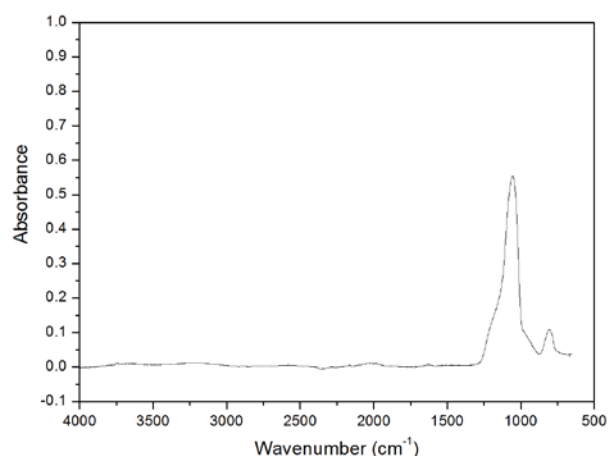


Figure 2. FT-IR spectrum of SiO<sub>2</sub> nanoparticles.

XRD patterns of the produced powders were depicted in Figure 3, which represents the silica (SiO<sub>2</sub>) showed a broad peak centered at 22.8°, indicating its amorphous structures.

Figure 4 shows the wide scan XPS spectrum of SiO<sub>2</sub> nanoparticles. The peaks at 527.2 and 94.4 eV are assigned to O 1s and Si 2p in the nanoparticles, respectively. Figures 5 (a) and (b) present the XPS spectra of O 1s and Si 2p narrow scan on the SiO<sub>2</sub> nanoparticles, respectively.

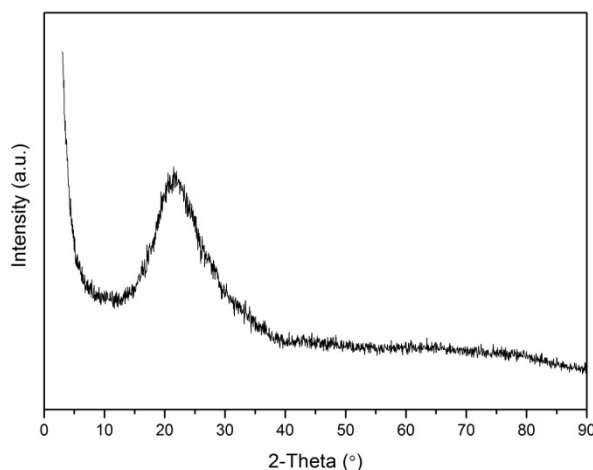
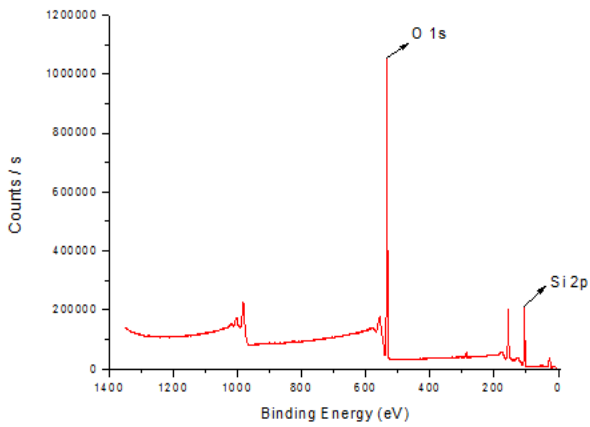


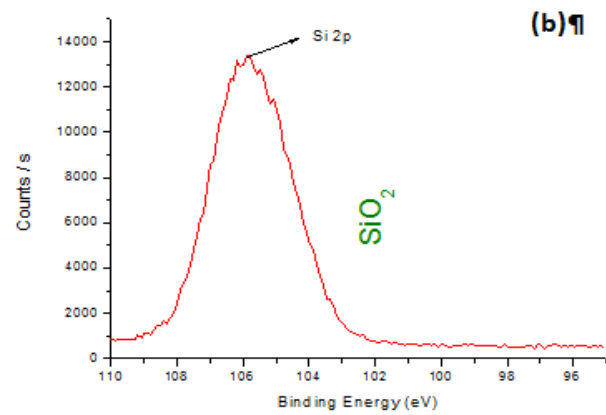
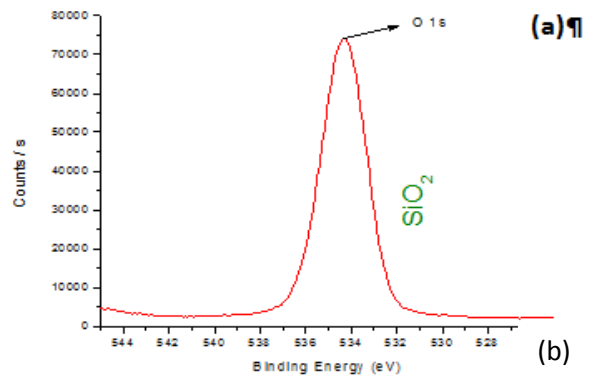
Figure 3. XRD pattern of SiO<sub>2</sub> powders produced via flame spray pyrolysis.



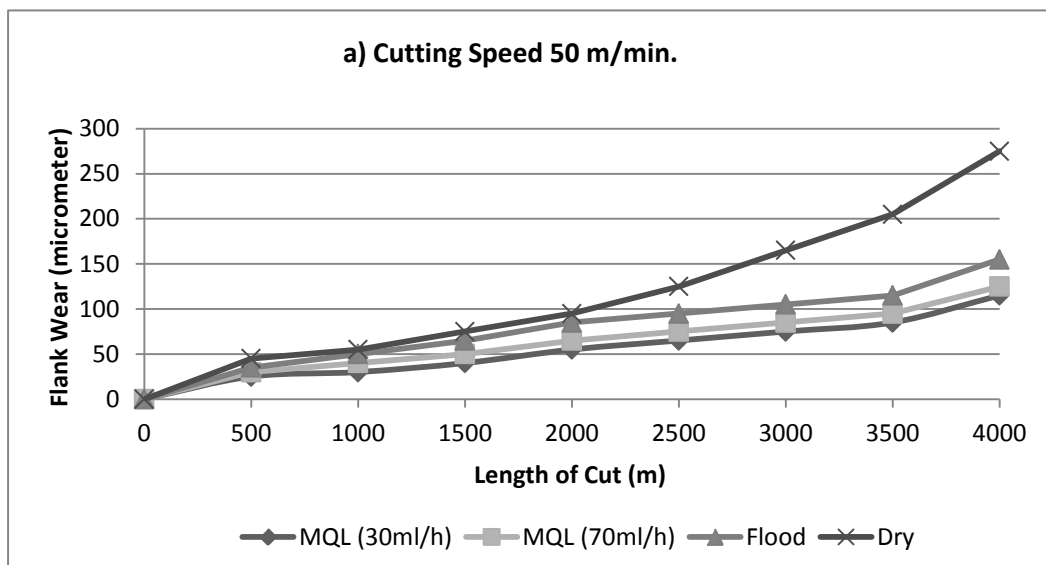
**Figure 4.** Wide scan XPS spectra results of SiO<sub>2</sub> nanoparticles.

Figure 6 compares the progression of flank wear for dry, flood and nanofluid milling. It is noted that at high cutting speeds (Figure 6(b)), the nanofluid reduces the flank wear. This is not the case, at low cutting speeds (Figure 6(a)). In fact, at low cutting speed, for some cases, the nanofluid slightly increased the flank wear. At low cutting speed, the heat generation is low and tool-process temperature is not as high as in speed machining. Therefore, the forced convective heat transfer is not as effective as in speed machining and one should not expect any significant reduction in tool wear due to reduction of tool-tip temperature. Instead, sometimes in dry milling at low speed, a slight increase of the process temperature causes a slight reduction in tool wear due to softening of the material.

Figure 7 shows the variation of main (vertical) cutting force with the cutting time for four cutting conditions. In all the cases of speed mill (a) the vertical cutting force is less in nanofluid milling due to less flank wear.



**Figure 5.** Narrow scan results of XPS (a) O 1s spectra and (b) Si 2p, SiO<sub>2</sub> nanoparticles.



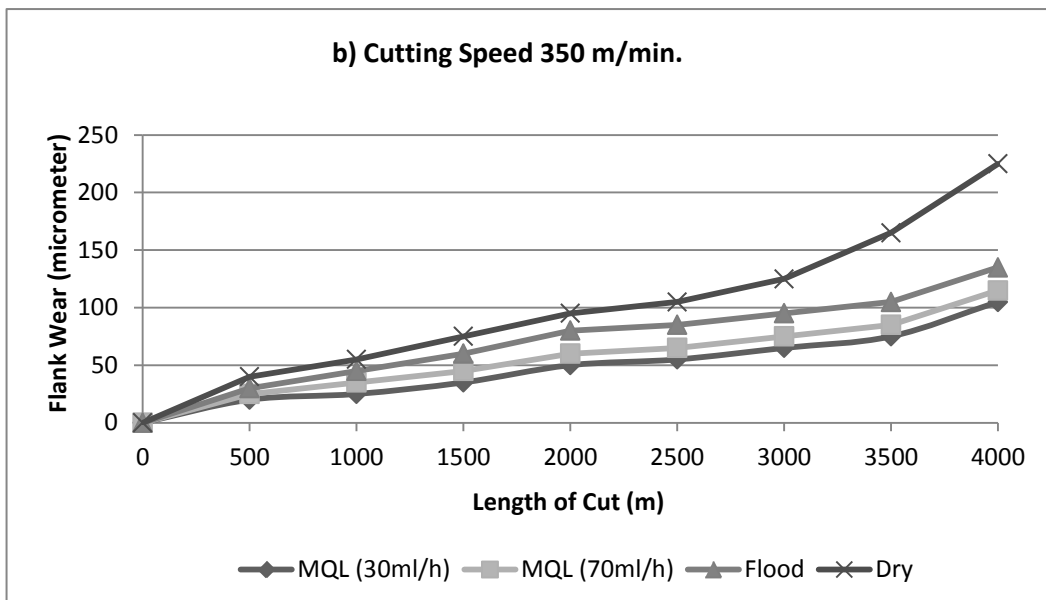


Figure 6. Change in flank wear VB with machining distance at two different speeds.

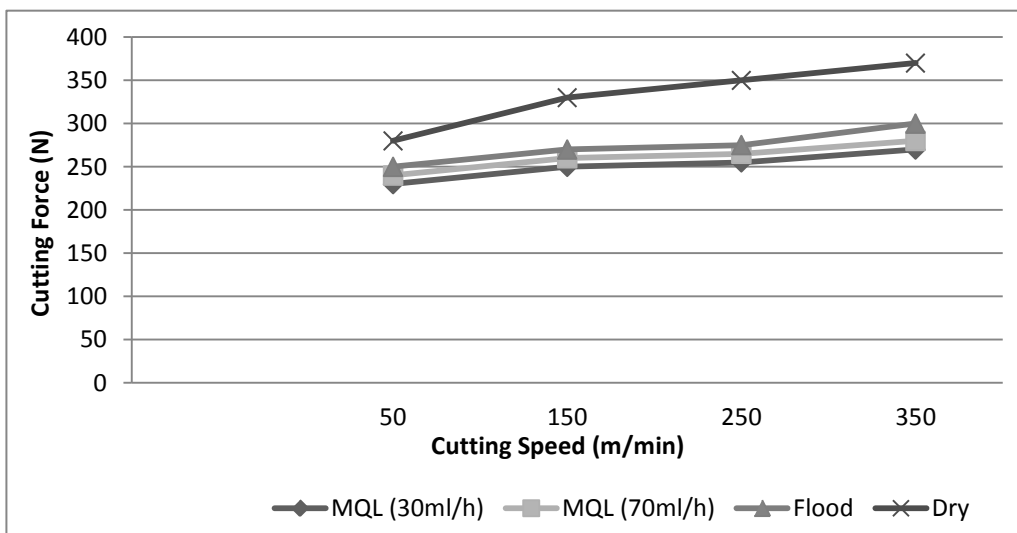
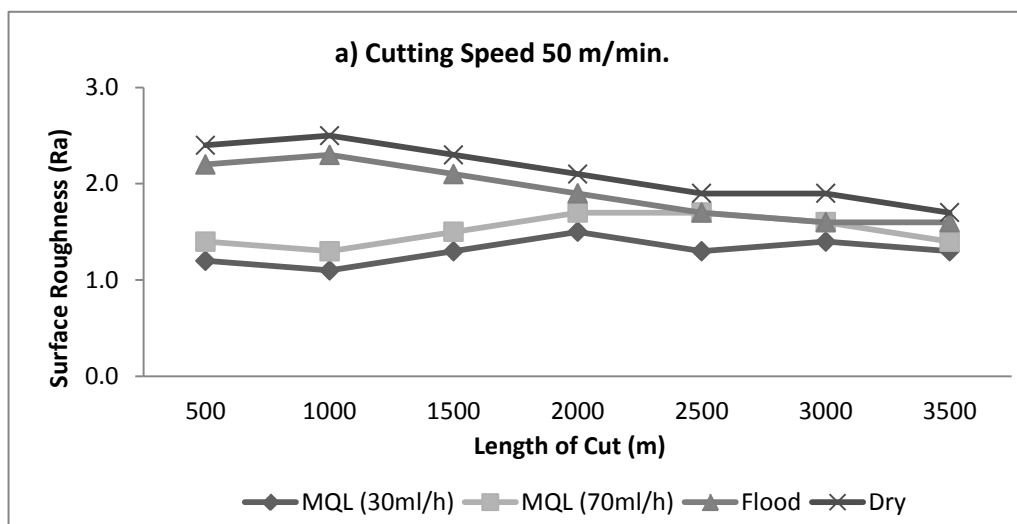
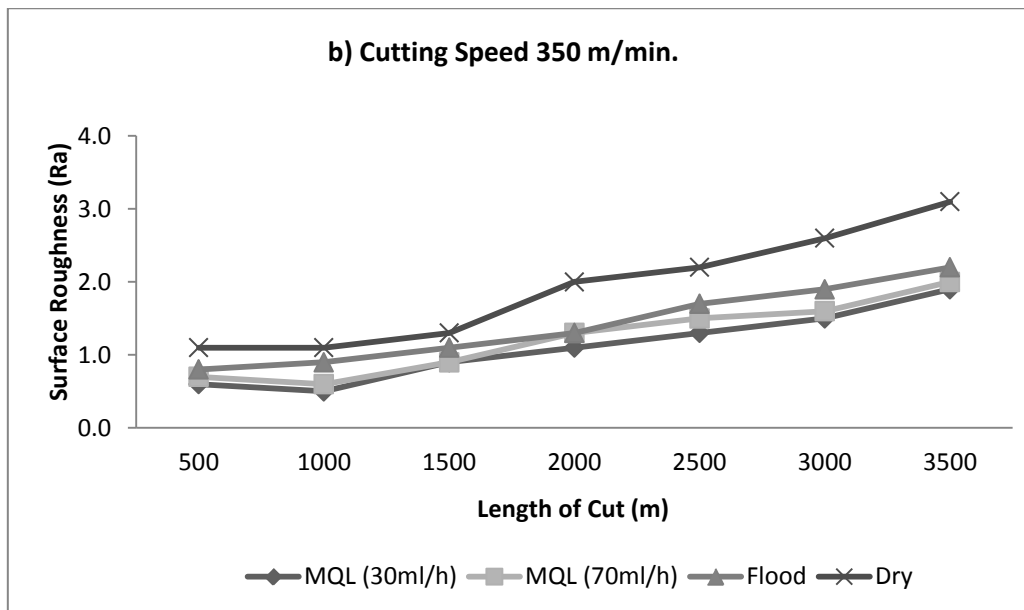


Figure 7. Relationship between the resultant cutting forces and cutting speeds measured under various machining environments.







**Figure 8.** The surface roughness ( $R_a$ ) values of the work-piece measured parallel to the feed direction.

Figure 8 compares the surface roughness for dry flood and nanofluid milling for seven different cutting conditions. Over a machined length of 500 m, the surface roughness was measured at 9-12 places and the mean value was taken. Finally, the mean value of all replicates at a cutting condition was taken and the same have been plotted in Figure 8. It is observed that in general the surface roughness is lower at higher cutting speeds compared to lower cutting speeds. However, at high speed provided a relatively poorer surface finish with nanofluid milling. It is observed that nanofluid does not improve the surface finish.

With reducing the nose radius of the tool and geometrical modifications, the amount of adhered material can be brought down. Investigations have to be carried out in this direction by changing the tool geometry for MQL conditions. The wear land width is seen to be almost same with MQL and flooded coolant application. This suggests that the coolant application has very little influence on flank tool wear. But the coolant has a significant effect on the amount of material adhesion on the tool. The resultant force was seen to be the lowest with flooded coolant system. This is because due to the flooded cooling, the adhesion on the tool is lowest. This lower adhesion produces lower frictional force.

#### 4. Conclusion

In summary, nano scale SiO<sub>2</sub> powders were successfully produced using flame spray prolysis for preparing of the nanofluid with SiO<sub>2</sub> nanoparticles. The produced powders were characterized in order to determine phase structure, particle size distributions and surface properties for cutting performances.

At all the cutting speeds, it was observed that the surface roughness could be improved by the application of coolant which contains SiO<sub>2</sub> nanoparticles. The improvement in surface finish can be attributed to the reduction in the material transfer onto the machined surface. At a higher speed of 350 m/min, it was clear from the graphs that the quantity of the coolant was not a deciding factor for surface roughness but there has to be MQL conditions which can reduce the surface roughness.

7075 aluminium alloy has been machined under different conditions of dry, MQL and flooded coolant/lubricant using uncoated carbide inserts. The process of machining was successful with using SiO<sub>2</sub> based nanofluids.

#### Acknowledgements

The authors are indebted to State Planning Foundation (DPT) and Dokuz Eylul University for financial and infrastructural support.

## References

- 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<sub>2</sub> nanofluid. *International Communications in Heat and Mass Transfer*, **37**, 687-694.
- Manna, I., 2009. Synthesis, Characterization and Application of Nanofluid-An Overview. *Journal of the Indian Institute of Science*, **89**, 21-33.
- 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.
- Saidura, R., Leong, K.N., Mohammad, H.A., 2011. A review on applications and challenges of nanofluids. *Renewable and Sustainable Energy Reviews*, **15**, 1646-1668.
- Singh, D., Kumar, R., Kumar, A., Rai, K.N., 2008, Synthesis and characterization of rice husk silica, silica-carbon composite and H<sub>3</sub>PO<sub>4</sub> activated silica. *Ceramica*, **54**, 203-212.
- Sreenivasa 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.
- Tok, A.I.Y., Boey, F.Y.C., Zhao, X.L., 2006, Novel synthesis of Al<sub>2</sub>O<sub>3</sub> nano-particles by flame spray pyrolysis, *Journal of Materials Processing Technology*, **178**, 270-273.
- 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.