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Research Article

Nano-Pyramid Structured Coatings for Demanding Tribological Applications

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Abstract

Present trends in metal cutting, high speed/feed machining, hard and dry cutting set more demanding characteristics for cutting tool materials. Machining leads to high friction between tool and work piece, and can result in high temperatures, impairing the dimensional accuracy and the surface quality of products. Applications of conventional cutting fluids are a major source of pollution. Solid lubricant assisted machining is an environmental friendly clean technology for desirable control of cutting temperature. The present work investigates the role of nano coated (water based sol-gel ceramics) tools of different tool geometry (milling tool: radial rake angle and nose radius) in machining on surface quality, wear rate, cutting forces and specific energy. Multilayer coatings (Al_2O_3) were applied by using dip coating method on CNC tools with help of water based sol-gel ceramic solution. These coatings act as solid lubricants. The performance of solid lubricant assisted machining was studied in comparison with uncoated tool. These coatings are characterized by High Resolution Scanning Electron Microscope (HRSEM), Scratch test analysis and nanoindentation.

Keywords: *Cutting tools, Dry machining, Solid lubricant, Water based sol-gel method.*

1. Introduction

Making improvements in machining process productivity along with machined part quality are two of the major goals associated with high performance manufacturing. These challenges can be addressed through the enhancement of the tribological compatibility with in the cutting tool/workpiece system. Tribological compatibility is the ability of the frictional bodies to adapt to each other and in so doing provide stable wear without undergoing intensive surface damage, for the longest period of time (Brown *et al*, 1995), (Bushe *et al*, 1981), (Fox-Rabinoyich *et al*, 2007), (Priest *et al*, 2000), (Neyille *et al*, 2007), (Ukonsaari *et al*, 2006), (Lu *et al*, 2007), (Eason *et al*, 1955). Indicators for tribological compatibility improvement within the tool/workpiece system are the following: (1) a critical and beneficial change in frictional properties (reduction in the coefficient to friction) at the tool/workpiece interface ;(2) tool life increase ;(3) wear behavior and chip formation improvement, as well as(4) simultaneous improvements in the surface integrity of the machined part (Brown *et al*, 1995), (Bushe *et al*, 1981).

In the 1980's hard ceramic TiN, TiC and Al_2O_3 coatings were commercially introduced as protective layers on cutting tools in the production industry. The BN coatings normally exhibit too much internal stress which causes premature failure of coatings.

It is essential to apply a protective coating on the cutting tools to improve the surface quality of tool components and service life of cutting tools. There are several materials which can be utilized as protective coatings including Cr plating, Diamond-like carbon (DLC), various nitrides, carbides, oxides and noble metal coatings, mostly deposited by use of the physical vapor deposition (PVD), chemical vapor deposition (CVD) process and sputtering deposition methods. The lifetime of the cutting tools with above mentioned coatings has been increases, but their performance is inconsistent (Dobrza´nski and Gołombek, 2003), (Dobrza´nski *et al*, 2004), (Dobrza´nski *et al*, 2003), (Sokovi´c *et al*, 2005), (Saoubi *et al*, 2009), (Muenz *et al*, 2008), (Paulitsch *et al*, 2008), (Quinto *et al*, 1988), (Rech *et al*, 2006), (Rech *et al*, 2007), (Ruppi *et al*, 2001), (Settineri *et al*, 2007), (Shin *et al*, 2004), (Staia *et al*, 2006), (Suzuki *et al*, 2006), (Tanoue *et al*, 2009).

The ceramic tool materials, and especially those Al_2O_3 based gain more and more importance over the last years in the high-speed dry-cutting processes. The oxide ceramics has characteristics of high hardness, high mechanical strength at room and high temperatures, and also of low grindability compared to other tool materials (Settineri *et al*, 2007), (Staia *et al*, 2006).

The Al_2O_3 based oxide tool materials feature the biggest and dynamically growing group of materials among the ceramic tool materials. The resistance of the Al_2O_3 based materials to chemical wear, their stability and abrasion wear resistance in the neutral and oxidizing atmospheres, and also at high temperature is, however, connected with their high brittleness, low strength (mostly for bending), and the low thermal shock resistance, which feature the significant limitations in using the corundum ceramics for cutting tools. Partial improvement of the

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disadvantageous properties of the pure Al_2O_3 and Al_2O_3 + ZrO_2 ceramics is possible by introducing the TiC additive to the sinter (Ruppi, 2005), (Ruppi, 2008), (Sarakinos *et al*, 2010), (Schuelke *et al*, 2010).

Alumina is uniquely suited for metal cutting tools due to its chemical inertness and high hot-hardness at the temperatures typically reached in these applications [33]. In addition to sintered alumina based ceramics, Al_2O_3 is also important as coating material. Alumina coated cemented carbide tools are used, for example, in turning and milling of steels and cast irons. The Al_2O_3 coatings are typically manufactured by CVD. CVD has been used for about 30 years for industrial deposition of wear resistant coatings and still dominates the market of Al2O3 coatings on cemented carbide tools Ruppi, 2005), (Ruppi, 2008), (Sarakinos *et al*, 2010), (Schuelke *et al*, 2010), (Schuetze and Quinto 2003), (Settineri and Faga 2006), (Settineri and Faga 2008).

Normally, the requirements of protective coating for tool and mold are as following: surface qualities--no scratch, no particles, surface roughness (Ra) less than 1 µm, and film thickness uniformity less than 5 percent.

Solvent acted as the coating carrier. The removal of solvent or drying of the coating proceeds simultaneously with condensation and solidification of the gel network. The origin of stress developed during drying of a solidified coating is due to the constrained shrinkage and low rate of solvent loss after solidification (Brinker and Hurd 1994), (Troczynski and Yang 2001), (Brinker and Scherer 2002), (pierre and Kluwerr 1998), (Sanchez and Ribot 1994), (Sharp 1999), (Barrow *et al*, 1996), (Wang *et al*, 2004), Zhang *et al*, 2001). The solvent content at solidification should be minimized in order to lower the stress in the coating (Brinker and Scherer 2002). It is very important to limit the condensation reaction rate during the removal of solvent upon drying, so that the volume fraction of solvent at solidification is kept small.

The drying rate plays a very important role in the development of stress and formation of cracks particularly in the late stages and depends on the rate at which solvent or volatile components diffuse to the free surface of the coating and the rate at which the vapor is transported away in the gas (Brinker and Hurd 1994), (Troczynski and Yang 2001).

Practically, there are several methods available for applying liquid coatings to substrates; the best choice depends on several factors including solution viscosity, coating speed and desired coating thickness. Most commonly used methods for sol-gel deposition are dip coating and spin coatings (Troczynski and Yang 2001), (Brinker and Scherer 2002), (pierre and Kluwerr 1998), (Sanchez and Ribot 1994). The film microstructure depends on the size and extent of branching (or aggregation) of the solution species prior to film deposition and relative rates of condensation and evaporation during film deposition. Physics of film formation examines the dipping and spinning processes with respect to such parameters as withdrawal rate, spin speed, viscosity, surface tension, and evaporation rate. The reactions which occur during this sol-gel process can be classified in two categories: hydrolysis and condensation reactions.

The present work investigates the role of nano coated (water based sol-gel ceramics) tools of different tool geometry (milling tool: radial rake angle and nose radius) in machining on surface quality, wear rate, cutting forces and specific energy. Single and multilayer coatings (i.e. Al_2O_3,) were applied by using dip coating method on CNC tools with help of water based sol-gel ceramic solution. The tool life can be extended by applying nanocrystalline protective coatings by using the sol-gel technique. With proper optimization of the Sol-gel process, single and multilayer Al_2O_3 thin films were applied as barrier layer for corrosion protection, wear resistance coatings for cutting tools and mold tools etc.

2. Experimental details: details of the sols and coating preparation

An alumina sol was prepared by adding 2272 grams of $AlCl_3$-$6H_2O$ to 10 liters of distilled water (for the hydrolysis), and the mixture is stirred vigorously at 300 rpm for 1hr until the suspension turned to a clear sol. Its pH value was adjusted to 4 by quickly adding 20% HNO_3 (so for called as peptization process) with a stirred speed of 600 rpm and followed by the slowly adding HNO_3 (2 ml) until the pH value to achieve required value (5.5~8.5). The solvent was filtered and dried by vacuum process until it turned into aggregated white powders (orthoaluminic acid).

The second step is to add orthoaluminic acid powders into 110 liters of distilled water and stir it for 1 hour with the speed of 300 rpm. Next, the NP (Nonyl phenol) was added into the mixture and kept stirring for 30 min at room temperature. The mixture was then gradually heated at 85~95°C and followed by adding 500 ml H_2O_2 into the mixture and kept stirring for 6 hours. Finally, the mixture solution was cooled down and turned to clear Al_2O_3 sol. Flow charts for Al_2O_3 sol preparation process as shown in Figure: 1

Overall shrinkage of Al_2O_3 sol during annealing of the gel material is negligible and does not imply crack formation.

The diluted water acts as solvent for solution. The gelation and condensation of the derive solution were carried out through suitable method. The pH value of water based Al_2O_3 sols is approximately 4~4.1. The Al_2O_3 sols were transparent at room temperature. No precipitate was formed even after storing such a long time.

The cleaned substrates were dipped into the water-based sol-gel solution for 20 seconds, and then withdrawn at a speed of 5 ~ 200mm/min and to be dried in air for 2 minutes inside the clean room.

Observations of surfaces and structures of deposited coatings were carried out on the transverse fractures on the Opton DSM 940 scanning electron microscope (SEM).

The pH of the sol-gel solution produced by alumina alkoxide was measured at 25°C using pH meter electrode. $50cm^3$ of the sol-gel solution was collected into a beaker and the pH value was determined by inserting the pH meter electrode into it. The pH value from the instrument was recorded. Depending on the pH value, chain-like or branched networks will be built. The morphology and the size of the particles of coated films can be tailored by pH value on the sol-gel process.

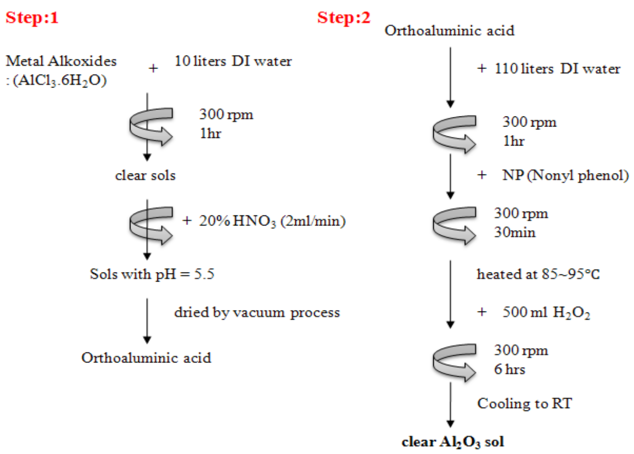

Fig. 1.Sequence steps for Al_2O_3 sol preparation process.

Differential Thermo-Gravimetry (DTG) analysis of a material at thermal treatment coupled with mass spectrometry analysis of the gaseous species which evolve during heating. DTG of the gel samples were carried out at a heating rate of 20 °C/min up to 900 °C, in air.

3. Results and discussions

Hydrolysis and polycondensation reactions of the alkoxy and hydroxyl group take place during the deposition and drying stages at the room temperature. The 2µm thickness sol-gel derived films can be achieved by dip coating process. A good uniformity and cracks free surface coatings were observed from the optical microscope (OM) analysis. These sol-gel derived Al_2O_3 coatings exhibit amorphous-crystalline phase transformation at 300°C and 350°C respectively. Heat treated at 650°C, water based sol-gel Al_2O_3 coatings become fully crystalline structure and the grain size increases with increasing the heating duration as shown in Figure 2. The grain size was increased to 500nm when the sintering temperature was up to 800°C; however, the surface becomes rough due to the crystal facet effect.. From the DTG curve at 100°C, weight loss peak appeared due to the evaporation of the solvent (water) from the Al_2O_3 coating surface as shown in Figure 2. In the temperature range from 150°C to 500°C, it has showed thermal effects. Moreover, during this range, polycrystallization processes associated inside the coating material. Above the 650°C temperature range, DTG curve manifested crystallization peaks. The pH value of water based Al_2O_3 sol-gel coating is approximately 4~4.1

The cross-sectional HRSEM image shows Al_2O_3 film with smooth and dense structure, as shown in Figure 3. The measured thickness of Al_2O_3 film is 34 nm at a drawing speed of 200 mm/min and the variation of film thickness is less than 10 %. The surface morphology of Al_2O_3 film after scratch test was shown in Figure 4. The detached Al_2O_3 film presents obvious plastic deformation and ductile failure appearance. It is believed that the Al_2O_3 film composed by aggregated nanoparticles, which are able to flow to accommodate a large amount of ploughing and associated shear stress through densification and shear deformation. There is no stress concentration at flaws in nanoparticle aggregated ultra-thin film. The critical applied stress for fracture of ultra-thin Al_2O_3 film may become infinite or failure approaches theoretical strength. In this case, the adhesion between the Al_2O_3 nano-particles may dominate fracture behavior of Al_2O_3 film.

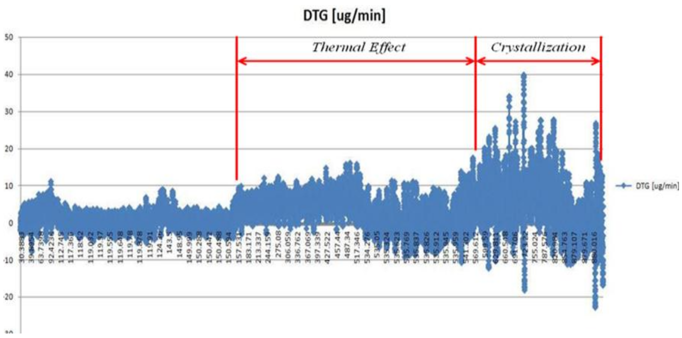

Fig.2 Differential Thermo-gravimetry (DTG) spectra of Al_2O_3 coated substrate.

Conclusions

This work presents, the importance and applications of water based Al_2O_3 coatings as a protective layer for cutting tools that has been gained in laboratory testing, in-plant trials, and modeling in an economic way. In-house developed water based sol-gel Al_2O_3 protective coating having good resistance to chemical attack. Single-coated and several superimposed sol–gel Al_2O_3 coated cutting tools and mold samples have been prepared and tested. The morphology and surface roughness of the coated tools under controlled and satisfy the criteria of the protective coating for cutting tools.

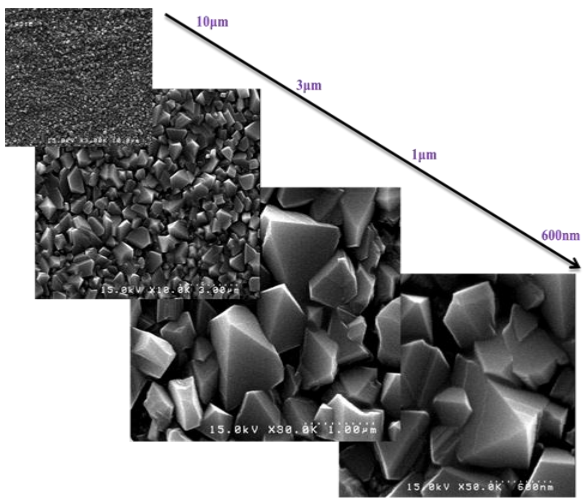

Fig. 3 HRSEM images of Al_2O_3 coated stainless steel heat treatment at 650°C (different magnification).

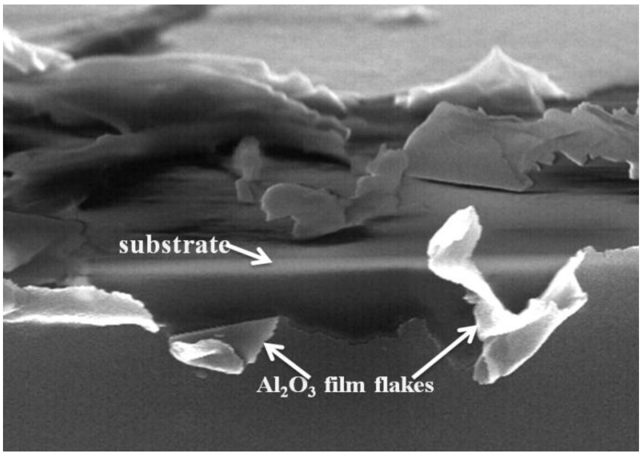

Fig. 4 Surface morphologies of Al_2O_3 film coated glass after scratch test.

Extend the life of the cutting tools by applied nanocrystalline protective coatings by using the water based sol-gel technique. With proper optimization of the Sol-gel process, Al_2O_3 thin films were applied as barrier layer for corrosion protection, wear resistance coatings for cutting tools and mold tools etc.

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