IMPROVEMENT OF GRAIN RETENTIVITY OF ELECTROPLATED DIAMOND TOOLS BY NI-BASED CNT COMPOSITE COATINGS

Tsunehisa Suzuki*, Takashi Konno, Hikaru Ibukuro****
***Yamagata Research Institute of Technology, **Just Corporation**

Keywords: **CNT, Electroplated Diamond Tool, Grain Retentivity**

Abstract

Grain retentivity of Ni-based coatings of electroplated diamond tools were improved by codepositing Carbon nanotubes (CNTs) into Ni-based coatings for developing electroplated diamond tools having a long tool life for machining hard, brittle materials such as fused silica into microstructures. Ni-based CNT composite coatings were electroplated in a nickel sulphamate plating bath containing CNTs. Surface roughness of the coatings was extremely improved by ultrasonic vibration of the bath during electroplating, and the minimum value of the roughness was 0.28 μm Ra. When more than 1 g/l of CNTs was added to the bath, the coatings had a Vickers hardness of more than 500 and about twice the grain retentivity than normal nickel coatings. Developed tools composed of the above Ni-based CNT composite coatings had about eight times longer tool life than normal tools.

1 Introduction

In recent years, the microfabrication of hard, brittle materials such as fused silica has received considerable attention from the standpoint of applications to biochips and optical components. Micro electroplated diamond tools are one of the attractive tools for microfabrication because the tool diameter can be easily downsized to under 100 μm. However, the weak point where the tool life is short should be solved for utilization of the tools. For a long tool life, the grain retentivity of Ni-based coatings, which bury diamond grains on the shank, must be improved because grains that are removed from the coatings significantly reduce the tool life.

Carbon nanotubes (CNTs) as reinforcements are increasingly attracting scientific and technological interest because of their unique chemical and physical properties for producing composites of metallic and non-metallic constituents. Some recent researches have investigated Ni-based CNT composite coating methods by using electroless codeposition [1] and electrocodeposition [2] and have reported that friction and wear properties are improved by codepositing CNTs in the matrix. These properties of CNT composite coatings seem to be effective in improving grain retentivity; however, there are no experimental data in the literature on the grain retentivity of CNT composite coatings.

In the present work, electroplated CNT composite coatings were applied for improving grain retentivity. It is very important that the coatings have good surface roughness for micro electroplated diamond tools. The effects of agitation methods on surface roughness were investigated. Ultrasonic vibration stirring was used for improving the surface roughness of coatings. The effect of the amount of CNTs in the bath on the hardness and grain retentivity of Ni-based CNT composite coatings was evaluated. Furthermore, the tool life of electroplated diamond tools, which was composed of Ni-based CNT composite coatings as topcoat layers, was evaluated by side machining and hole drilling.

2 Experimental Procedure

2.1 Electroplating Method and Characterization

Table 1 lists the plating bath and operation conditions for Ni-based CNT composite coatings. Ni-based CNT composite coatings were deposited

Table 1 The plating bath and the operation conditions for CNT composite electroplatings.

Bath	Nickel Sulphamate plating bath
CNT size	ϕ60-100 nm, length 1-2 μm
CNT in bath	0 - 10 g/l
Ultrasonic agitation	42 kHz, 30 W
Bath temperature	50 °C
Current density	5 A/dm^2

on substrates by electroplating using a nickel sulphamate plating bath under galvanostatic conditions. The plating bath was composed of 500 g/l $NiHSO_3 \cdot NH_2$, 4 g/l $NiCl \cdot 6H_2O$, 33 g/l H_3BO_3, and 0-10 g/l CNTs with cation surfactants. CNTs used in this study were multi-walled carbon nanotubes (NTP Co., Ltd.) and were typically 60 to 100 nm in diameter and 1 to 2 μm in length. CNTs were added to plating baths after being dispersed by an ultrasonic homogenizer and by cation surfactants to improve dispersibility into aqueous solutions. Substrates were prepared by polishing, solvent cleaning, and acid cleaning in this order and were used as anodes. A pure Ni plate was used as the cathode. Current density was controlled at 5 A/dm^2 by a galvanostat. The plating bath was controlled at 50 °C using a water bath and was agitated during plating. Rotational stirring and ultrasonic vibration stirring were the agitation methods for improving surface roughness of the coatings. The stirring rate was 200 min^{-1} or 500 min^{-1} and the vibration frequency was 42 kHz.

Hardness and surface roughness of composite coatings, which were deposited on SK steel shanks, were measured by a micro Vickers hardness tester (HM-124, Akashi Co., Ltd.) and a 3D optical profiler (Newview200, Zygo Co., Ltd.), respectively.

2.2 Evaluation of Grain Retentivity

Grain retentivity was evaluated by the maximum shear strength measured by bondtesters (Series-4000, Dage Co., Ltd). Methods of measuring shear strength are illustrated in Fig. 1. A shear tool, which was made of a monocrystalline diamond with a 150 μm test width, was located 10 μm vertically from the coating surface and was moved horizontally, such as in the shear direction in Fig. 1. The tool speed was 100 μm/s. The tool was made to approach near exposed parts of grains, which were buried in coatings and hit them. When grains were dug up by pushing of the shear tool, the maximum shear strength reflecting grain retentivity of coatings was measured by a load cell. The load cell was attached to the bottom of the shear tool and can provide a maximum force of 20 N.

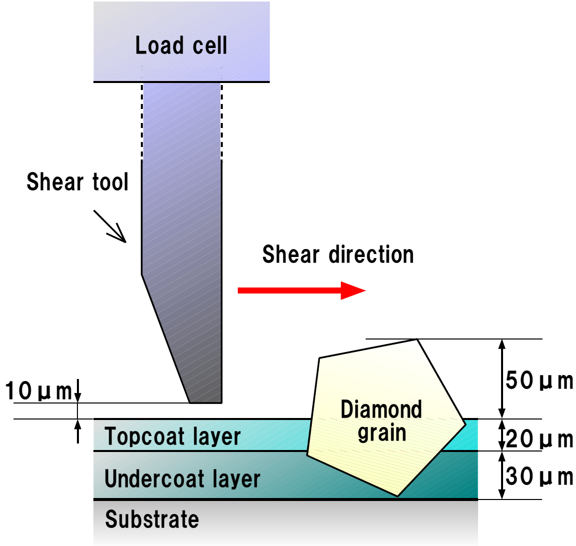


Fig. 1 Schematic illustration of shear test for grain retentivity evaluation using a bondtester.

Table 2 lists the conditions of shear test pieces. Well defined crystal diamond grains, which were about 100 μm in diameter, were buried in the coatings composed of two layers on S45C plates. Normal 30 μm-thick Ni coatings were arranged on substrates as undercoat layer. Ni-based CNT composite coatings or normal Ni coatings, which were 20 μm in thickness, were arranged on the undercoat layers as topcoat layers for evaluation. topcoat layers were electroplated using baths containing 0, 0.5 and 1.0 g/l CNTs. The bath was continuously vibrated ultrasonically during electroplating. Figure 1 depicts other electroplating conditions.

2.3 Evaluation of Tool Life

Ni-based CNT composite coatings were applied to electroplated diamond tools to improve tool life. The tool life of electroplated diamond tools was evaluated by side machining and hole drilling using the diamond tools. Testing tool life is schematically illustrated in Fig. 2. NC milling machines (Hyper5, Makino Co., Ltd.) were used for those tests.

Table 2 Conditions of Electroplated diamond tool for shear tests and tool life tests.

Test piece	For share test	For life test
Substrates or shanks	S45C plate	cemented tungsten carbide (ϕ0.4 mm or ϕ3 mm)
Diamond grains	well-defined crystals	friable irregular shapes
Diamond size	100 μm (#200)	50 μm (#400)
Thickness of all coatings	50 μm (50 % for diamond)	30 μm (60 % for diamond)
Thickness of composite coatings	20 μm from surface	15 μm from surface

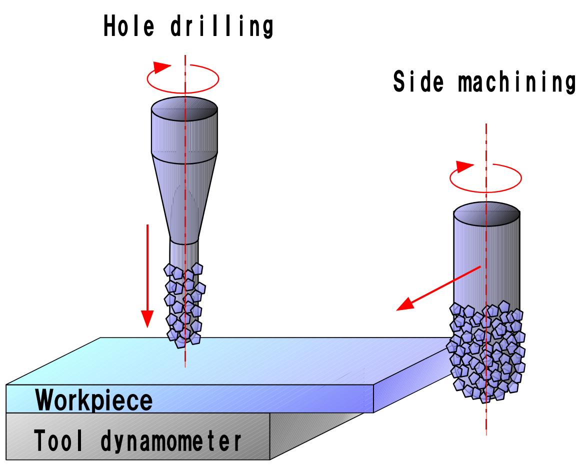


Fig. 2 Schematic illustration of side machining and hole drilling for testing tool life.

Table 3 presents the machining conditions for testing tool life. Side machining tool life was determined by the machining distance until the coatings were peeled from substrates. Side machining was performed to one side of workpieces which were approximately 75 mm in length, repeatedly until tool life. Workpieces were 1.0 mm-thick white plate glass. Machining methods were down cut to a depth of 50 μm. Rotation speed was 5,000 min^{-1}, and feed speed, 500 mm/min.

Hole drilling tool life was estimated by the grinding force and SEM observations of coatings after drilling 10 holes 4.0 mm in depth. Workpieces were 6.0 mm-thick fused silica. Workpieces were fixed on a tool dynamometer (Kistler Co., Ltd.), and thrust components of the cutting force were measured during drilling. Drilling was stepwise with 60 μm steps. Rotation speed was 15,000 min^{-1}, and feed speed, 1.5 mm/min.

Table 2 lists the characteristics of the

Table 3 The machining conditions for testing tool life.

	Side machinig	Hole drilling
Processing method	Down cut	Step processing
Rotation speed	5,000 min^{-1}	15,000 min^{-1}
Feed speed	500 mm/min	1.5 mm/min
Depth of cut	50 μm	---
Stepping amount	---	60 μm/time
Grinding oil	EDF-K2 (Nisseki Mitsubishi)	
NC milling machine	HYPER5 (Makino Co., Ltd)	
Work piece	White plate glass	Fused silica

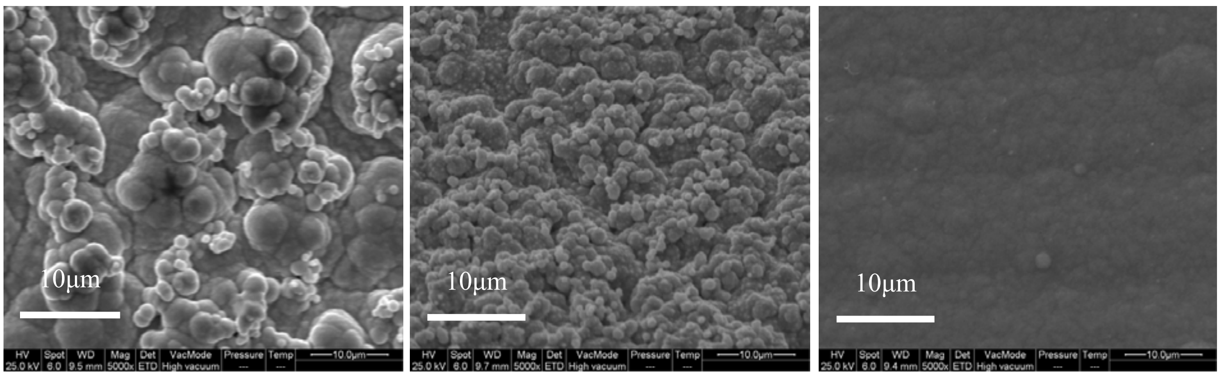
electroplated diamond tool. Friable irregular diamond grains about 50 μm in diameter were buried in two layers on WC-Co Cemented Carbide shanks.

Normal 15 μm-thick Ni coatings were arranged on shank surfaces as undercoat layer. Ni-based CNT composite coatings or normal Ni coatings, which were 15 μm in thickness, were arranged on the undercoat layers as topcoat layers for evaluation. topcoat layers were electroplated using baths containing 0 and 1.0 g/l CNTs. The bath was continuously vibrated ultrasonically during electroplating. Figure 1 depicts other electroplating conditions. The 0.5 mm in diameter electroplated diamond tools were used for hole drilling. Tools about 3.0 mm in diameter were used for side machining.

3 Results and Discussion

3.1 Characterization of Coatings

Figures 3 (a), (b) and (c) depict surfaces of Ni-based CNT composite coatings electrodeposited with rotational stirring or ultrasonic vibration stirring. The coatings were electroplated on 3.0 mm-diameter SK steel shanks in a plating bath



(a) Rotation speed 200 min^{-1} (b) Rotation speed 500 min^{-1} (c) Ultrasonic vibration 42 kHz

Fig. 3 SEM images of the surface of Ni-based CNT composite coatings electrodeposited with rotational stirring or ultrasonic vibration stirring.

containing 0.4 g/l CNTs. Rotational stirring at a rotation speed of 200 min^{-1} produced some large nodular deposits that can be observed on the surface (Fig. 3 (a)). At a rotation speed of 500 min^{-1}, nodular deposits, depicted in Fig. 3 (b), seem to become smaller. The surface state for ultrasonic vibration stirring completely differs from that for rotational stirring. No nodular deposits can be observed on the surface (Fig. 3 (c)), and surface roughness is minimum (0.28 μm Ra). Subsequent examinations were performed under ultrasonic vibration stirring. Some nodular deposits are due to large bundled CNTs in the plating bath. Rotational stirring can't disperse CNTs in the plating bath, and large bundled CNTs were codeposited in coatings. In contrast, ultrasonic vibration stirring could disperse CNTs in the plating bath, and discrete CNTs were codeposited in coatings.

Figure 4 presents an SEM image of a CNT composite coating surface etched by acid. CNTs appear well dispersed and embedded in the nickel matrix, indicating that ultrasonic vibration stirring is an effective method of obtaining well dispersed CNTs in coatings.

Figure 5 plots the Vickers hardness of coatings versus the amount of CNTs in the bath. For CNT amounts from 0 to 1 g/l, the hardness of composite coatings steadily increases with increasing amounts of CNTs and exceeded 500 HV. The hardness is considered to increase due to the increasing volume fraction of CNTs in the nickel matrix. However, increasing the amount of CNTs beyond 1 g/l resulted in a slower increase in hardness. We thought some kind of CNT interaction changed the mechanisms of codeposition and made it difficult to codeposite CNTs in coatings.

3.2 Grain Retentivity of Coatings

Figure 6 plots the measured grain retentivity of electroplated composite coatings versus the amount of CNTs in a bath. For the bath containing 0.5 g/l CNTs, the mean grain retentivity of Ni-based CNT composite coatings was almost the same as that of normal Ni coatings. For a bath containing 1.0 g/l CNTs, the mean grain retentivity of Ni-based CNT composite coatings was twice that of normal Ni coatings. The results indicated that grain retentivity is effectively improved by Ni-based CNT composite coatings electroplated in a bath containing 1.0 g/l CNTs. The high grain retentivity of coatings is considered to be due to the mechanical strength of CNT composite coatings. The dispersion in

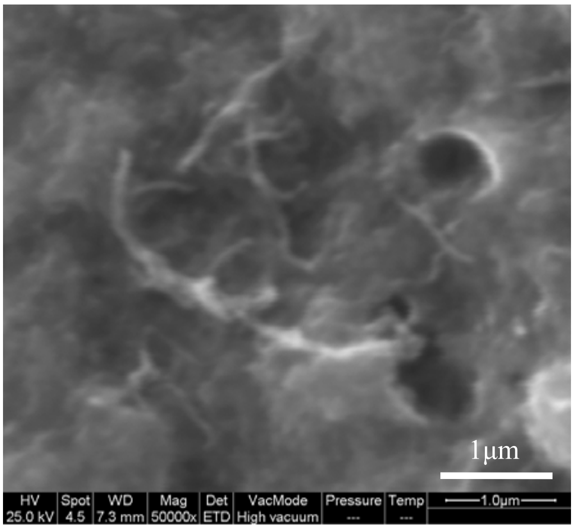


Fig. 4. SEM image of CNT composite coating surface etched by acid.

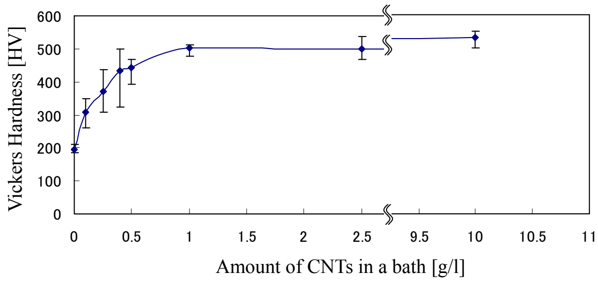


Fig. 5. Vickers hardness of coatings versus amount of CNTs in bath.

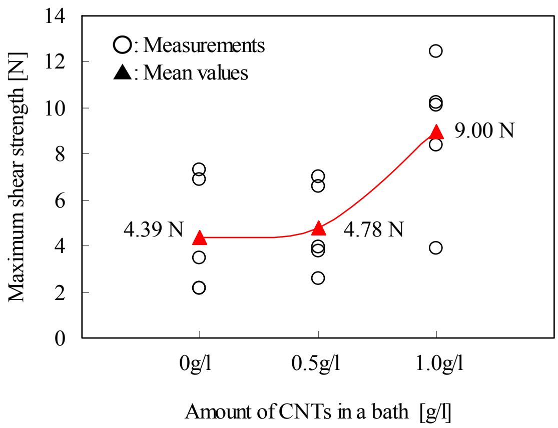


Fig. 6. Grain retentivity of coatings versus amounts of CNTs in bath.

measured values is due to the different shape of grains.

3.3 Tool Life of Electroplated Diamond Tools

3.3.1 Side machining

Side machining of white plate glass was performed with developed tools 3.0 mm in diameter under the conditions presented in Table 3. Figure 7 plots the life of electroplated diamond tools versus the amount of CNTs in the bath. The results indicate that CNT composite coatings electroplated in a bath containing 1 g/l CNTs have an eight times longer mean tool life than normal nickel coatings. However, results in Fig. 7 also indicate bad repeatability, probably due to irregular shapes of diamond grains or differences of partial characteristic in coatings.

SEM images of electroplated diamond tools

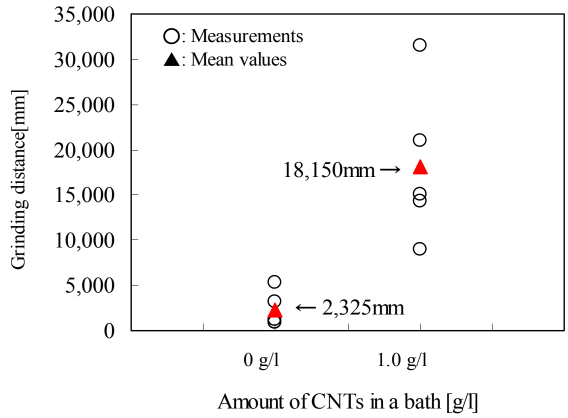
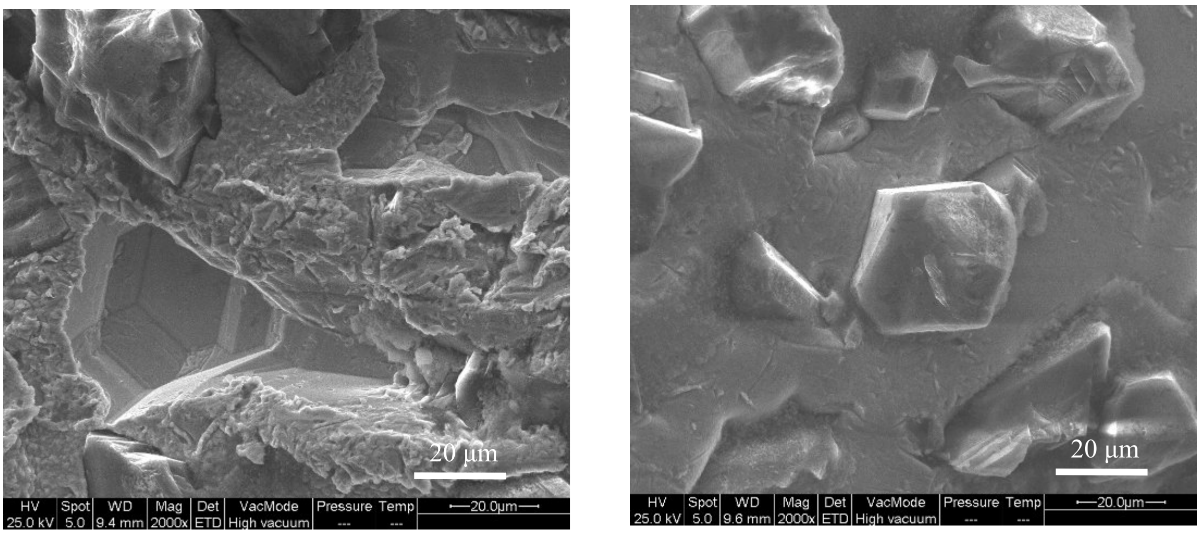


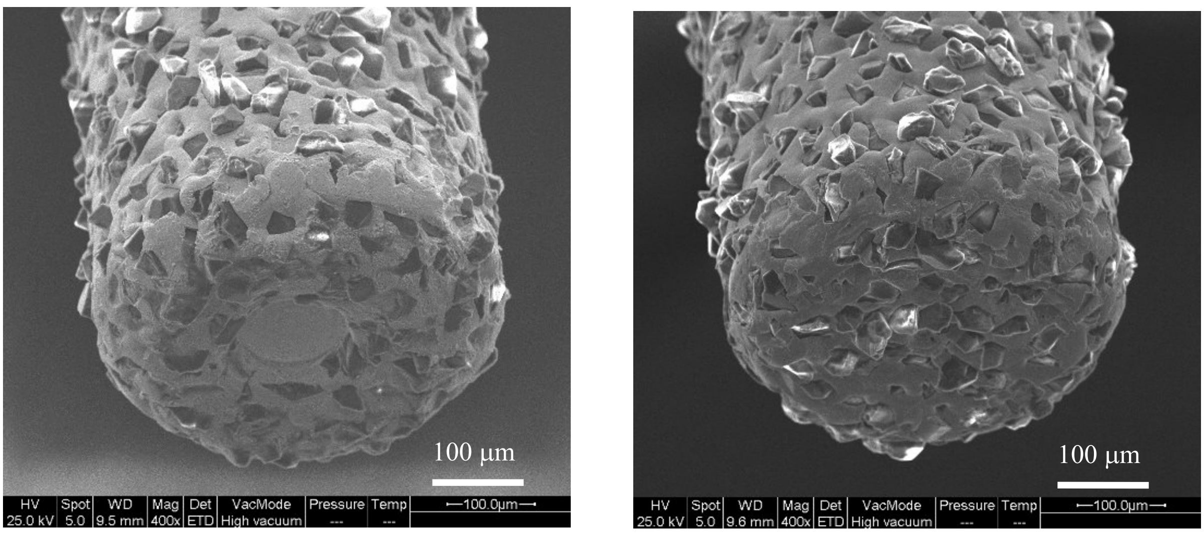
Fig. 7. The tool life of electroplated diamond tool versus amounts of CNTs in bath.



(a) For the topcoat layer composed of Ni coating

(b) For the topcoat layer composed of Ni-based CNT composite coating

Fig. 8 SEM images of ϕ3.0mm electroplated diamond after side machining



(a) For the topcoat layer composed of Ni coating

(b) For the topcoat layer composed of Ni-based CNT composite coating

Fig. 9 SEM images of ϕ0.5 mm electroplated diamond tool after processing ten holes.

after side machining are presented in Figs. 8 (a) and (b). For normal Ni coatings, holes and scratch marks formed by grain dropping can be observed on the tool surface. In contrast, for normal Ni-based CNT coatings electroplated in a bath containing 1.0 g/l CNTs, no such holes and scratch marks are observed. The results indicate that Ni-based CNT composite coatings improved grain retentivity of tools and decreased dropping grains.

3.3.2 Hole drilling

Hole drilling of fused silica was performed with developed tools 0.5 mm in diameter under the conditions presented in Table 3. Figure 9 depicts SEM images of electroplated diamond tools after drilling 10 holes to a 4.0 mm depth. Figure 9 (a) presents a tool with normal Ni coatings, and Fig. 9 (b), a tool with Ni-based CNT composite coatings. For the tool with normal Ni coating, a large hole was formed in the bottom of the tool near the center of the axis of rotation, probably formed by polishing of dropped grains retained between the tool and workpiece. In contrast, for Ni-based CNT composite coatings, no hole formed by grain dropping can be observed. Figure 10 plots the transition of the maximum grinding force for drilling up to ten 0.5 mm-diameter holes to a 4.0 mm depth. For the normal Ni coating, the grinding force increased gradually with the number of holes up to six holes, above which it increased rapidly due to the reduced machining ability because of dropping grains. In contrast, for Ni-based CNT composite coating, grinding force increased gradually with the number of holes up to 10 holes and then became stable.

The results (Figs. 6 to 10) indicate that the increased tool life is due to increased grain retentivity and improved wear resistance related to high hardness. Ni-based CNT composite coatings are extremely effective for improving grain retentivity of electroplated diamond tools.

4 Conclusions

The following conclusions were derived from the results and discussions.

1. Ultrasonic vibration stirring during plating effectively disperses CNTs in the plating bath, decreasing nodular deposits and improving the surface roughness of Ni-based CNT composite coatings. A surface roughness of 0.28 mm Ra was obtained.

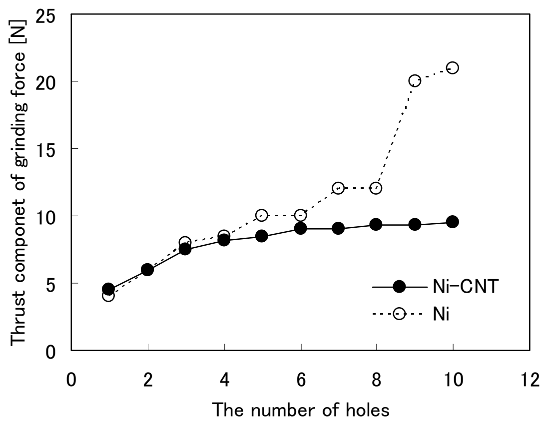


Fig. 10 Transition of the maximum grinding force (thrust component) for ϕ0.5 mm hole drilling.

2. The Vickers hardness of Ni-based CNT composite coatings electroplated in a bath containing more than 1 g/l of CNTs exceeds 500 HV
3. Grain retentivity is improved by codepositing CNTs in Ni coatings. Grain retentivity of the above Ni-based CNT composite coatings is about twice that of normal Ni coatings.
4. Electroplated diamond tools composed of the above Ni-based CNT composite coatings have a tool life about eight times longer than that of normal tools for side machining of glass plates. Micro electroplated diamond tools also have longer tool life for drilling holes in fused quartz glass.

Acknowledgement

This research was performed as a joint project with JUST Corporation and as an ultra-precision machining technology project at Yamagata Research Institute of Technology. We thank JUST Corporation for supporting the present work.

References

[1] L. Y. Wang et al., “Friction and wear behavior of electroless Ni-based CNT composite coatings”, *Wear*, Vol. 254, Issue 12, pp 1289-1293, 2003.

[2] X. H. Chen, F. Q. Cheng, S. L. Li, L. P. Zhou, D. Y. Li. “Electrodeposied nickel composites containing carbon nanotubes”. *Surface and Coatings Technology*, Vol. 155, pp 274-278, 2002.