Characterization of superabrasive composite coatings made of Ni alloys and cubic boron nitride (cBN) particles

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Abstract

In recent years, the superabrasive cubic boron nitride (cBN) grinding wheels have been widely used in the automotive and aerospace industries. Many studies have been performed to increase the efficiency of the grinding tools. These studies include investigating the bonding force between the cBN grits and the bond coat. Although the cBN grits have an outstanding performance in the grinding applications, their performance inside different bond coats may vary. Therefore, in this thesis, the Ni-based cBN composite coatings, including NiP-cBN, NiP/Ni10wt%W-cBN and NiP/Ni20wt%W-cBN were studied.

The grit adhesion study on NiP-cBN, NiP/Ni10wt%W-cBN and NiP/Ni20wt%W-cBN coatings has been conducted. A new testing technique has been proposed by scratching the single grit embedded inside coating away using a flat-bottom stylus. Different grit behaviors were observed. The grits were either dislodged, partially dislodged, fractured or fully bonded inside the bond coat. Grits that were completely dislodged were used for the adhesion study. The tangential force and energy for all the dislodged grits were reported. It was found out that the bond force between the grit and the bond coat is majorly influenced by the bond depth of the grit. Although there was no significant difference in the tangential force and energy among different types of coatings, evidence of plastic deformation was observed on the NiP/Ni20wt%W coatings.

Mechanical properties, including hardness, reduced modulus and the time-dependent strain rate sensitivity (SRS) on NiP, Ni10wt%W and Ni20wt%W coatings were studied. The grain sizes of all the coating were also reported. The hardness and reduced modulus of Ni-W coatings were

higher than those of the Ni-P coating. Between the two Ni-W coatings, sample with higher W content has higher hardness and lower reduced modulus values. Two different nanoindentation techniques, constant load (CL) method and constant strain rate (CSR) method, were performed to evaluate the strain rate sensitivity of the coatings. The CSR SRS results on all the coatings were higher than the CL SRS results. The Ni-P coating has a higher SRS value compared with the Ni-W coating. The two Ni-W coatings with different W contents have similar SRS results. Tungsten segregation near the grain boundaries is assumed to lead to the lower SRS value. The small grain size of the Ni-P sample is the major reason for its high SRS value.

Résumé

Ces dernières années, les meules en nitrure de bore cubique superabrasif (cBN) ont été largement utilisées dans les industries automobile et aérospatiale. De nombreuses études ont été réalisées pour augmenter l'efficacité des outils de rectification. Ces études comprennent l'étude de la force de liaison entre les grains de nitrure de bore cubique et la couche de liaison. Bien que les grains de CBN aient une performance exceptionnelle dans les applications de rectification, leur performance à l'intérieur des différentes couches de liant peut varier. Par conséquent, dans cette thèse, les revêtements composites à base de Ni, y compris NiP-cBN, NiP/Ni10wt%W-cBN et NiP/Ni20wt%W-cBN ont été étudiés.

L'étude de l'adhérence des grains sur les revêtements NiP-cBN, NiP/Ni10wt%W-cBN et NiP/Ni20wt%W-cBN a été utilisé. Une nouvelle technique d'essai a été proposée en grattant le grain unique incorporé dans le revêtement à l'aide d'un stylet à fond plat. Différents comportements des grains ont été observés. Les grains étaient soit délogés, soit partiellement délogés, soit fracturés, soit entièrement liés à l'intérieur de la couche de liaison. Les grains qui ont été complètement délogés ont été utilisés pour l'étude d'adhérence. La force et l'énergie tangentielles de tous les grains délogés ont été rapportées. Il a été découvert que la force d'adhérence entre le grain et la couche d'accrochage est principalement influencée par la profondeur d'adhérence du grain. Bien qu'il n'y ait pas de différence significative dans la force tangentielle et l'énergie entre les différents types de revêtements, des preuves de déformation plastique ont été observées sur le revêtement NiP/Ni20wt%W.

Les propriétés mécaniques, notamment la dureté, le module réduit et la sensibilité à la vitesse de déformation en fonction du temps (SRS) du revêtement NiP, Ni10wt% W et Ni20wt %W ont été étudiées. La taille des grains de tous les revêtements a également été rapportée. La dureté et le module réduit des revêtements Ni-W étaient plus élevés que ceux du revêtement Ni-P. Entre les deux revêtements Ni-W, l'échantillon ayant une teneur plus élevée en W présente une dureté plus élevée et des valeurs de module réduit plus faibles. Deux techniques de nanoindentation différentes, la méthode à charge constante (CL) et la méthode à taux de déformation constant (CSR), ont été utilisées pour évaluer la sensibilité des revêtements au taux de déformation. Les résultats de la méthode CSR SRS sur tous les revêtements ont été plus élevés que ceux de la méthode CL SRS. Le revêtement Ni-P a une valeur SRS plus élevée que le revêtement Ni-W. Les deux revêtements Ni-W avec des teneurs en W différentes ont des résultats SRS similaires. On suppose que la ségrégation du tungstène près des limites des grains conduit à un résultat SRS plus faible. La petite taille des grains de l'échantillon Ni-P est la principale raison de son résultat SRS élevée.

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Contributions of authors

The author of this thesis was responsible for most of the experimental work and all the writing. The contribution of experimental work and data analysis that was not contributed by the author is listed below:

Chapter 4: Experimental results and discussion of single grit adhesion tests

- Prof. Richard Chromik supervised me for the work in this chapter and reviewed the thesis.
- Sima Ahmad Alidokht helped plan experimental, data analysis and also thesis review.

Chapter 5: Experimental results and discussion of hardness, reduced modulus and strain rate sensitivity tests

- Prof. Richard Chromik supervised me for the work in this chapter and reviewed the thesis.
- Sima Ahmad Alidokht helped for planning experimental, analyzing data and also reviewing this chapter.
- Yinyin Zhang measured the thickness, hardness and reduced modulus of the NiP coating.
- Lisa Lee measured the thickness, hardness and reduced modulus of Ni10wt%W and Ni20wt%W coatings. She also imaged the surface morphology of NiP, Ni10wt%W and Ni20wt%W coatings. In addition, she measured the grain size of NiP, Ni10wt%W and Ni20wt%W coatings.

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Chapter 1: Introduction

1.1 Background

Nowadays, high-performance grinding is becoming more and more concerned in the automotive and aerospace applications.^{1, 2} Prolonging the life span of the grinding tools is now a major consideration in order to improve the product quality and reduce the production costs. The grinding wheels, **Fig. 1**¹, are usually coated with a plating material with abrasive particles embedded inside. The grinding wheel would rotate with a linear speed and contact the workpiece. As a result, materials, which are also called chips, would be removed from the workpiece to reach a final surface finish. During the grinding process, abrasive grains might be detached from the bond coat, which will cause the grinding process to be less effective.¹ Thus, the grinding wheel with high grit retention properties is preferred to increase the grinding efficiency. It is assumed that increasing the bonding force between the bond coat and the particles would help to prolong the life span of the grinding tools.



Figure 1: Schematic of monolayer electroplated (a) and brazed (b) CBN wheels.¹

Therefore, increasing studies on the high-speed grinding wheels have be performed, including studying different abrasives³⁻⁵, bond coats⁵⁻⁸ and the adhesion between the abrasive and bond coat⁹⁻¹¹. Compared with conventional abrasives, superabrasives with higher hardness, better wear resistance and better thermal durability are favorable for the high-speed grinding. Furthermore, the bond coat with good mechanical properties, such as high hardness, good thermal and chemical stability, is preferred in the grinding applications.⁸ From previous studies, the cBN grinding wheels have attracted a lot of attentions these days. In this project, the cBN composite coatings will be specifically studied. Two types of bond coat were tested, including Ni-P and Ni-W. In addition, for Ni-W coating, samples with different W contents were used: Ni10wt%W and Ni20wt%W.

Many techniques have been proposed to measure the bonding force between the bond coat and the abrasives.^{10, 12-15} Most of the studies measured the bonding force by pulling out single grits using a single grit grinding machine. These studies can further allow to monitor the grit pull-out mechanisms. The grit behaviors and bond coat behaviors during and/or after grinding can also be studied. In this project, a new single grit testing technique is proposed to study the bonding force between the cubic boron nitride (cBN) superabrasives and the Ni-based bond coat.

The mechanical property of the coating is also being suggested to have an effect on the general grinding performance. Nanoindentation technique can study the mechanical properties of the materials on the local scale. In this thesis, the hardness, reduced modulus and the time-dependent properties, strain rate sensitivity, will be studied for Ni-P and Ni-W coatings. Different

nanoindentation techniques were performed to study the strain rate sensitivity, including constant load method and constant strain rate method.

1.2 Thesis objective and structure

1.2.1 Objective of thesis

This thesis is focused on the following parts:

- (a) Analyzing the adhesion force between the Ni alloy coatings (NiP, NiP/Ni10wt%W, NiP/Ni20wt%W) and the cBN grits.
- (b) Measuring hardness, reduced modulus and strain rate sensitivity of NiP, Ni10wt%W and Ni20wt%W coatings.

1.2.2 Structure of thesis

The thesis is divided into 6 chapters. Descriptions of each chapter are given in the following.

Chapter 1 presents a general background of this project and introduces the objective of this thesis.

Chapter 2 is a literature review including 5 main sections. The first section discusses about the selection of superabrasives. The second section reviews the influencing factors of the bonding force between the bond coat and the superabrasives. The third section will present the selection of bond coat and also a general review of bond coat preparation. The fourth section is a general review of previous bonding force testing. The last section discusses about the time-dependent strain rate

sensitivity. A general review of creep deformation will be presented, followed by different strain rate sensitivity testing techniques.

Chapter 3 presents the sample information, sample characterizations and experimental procedures. The working parameters of SEM and optical profilometry for sample characterizations will be presented. Detailed experimental procedures and analysis of grit adhesion testing and strain rate sensitivity testing will be shown separately.

Chapter 4 discusses the experimental results of grit adhesion tests. NiP-cBN, NiP/Ni10wt%WcBN and NiP/Ni20wt%W-cBN samples were used. Different grit behaviors after the test will be presented, including grit dislodgement, grit fracture, partial-grit dislodgement and fully-bonded grit behavior. The protrusion height and bond depth of the dislodged cBN grits were measured. The tangential force and energy for each dislodged grit were also reported. Results among all the samples will be discussed and compared. Possible grit failure mechanism will also be discussed.

Chapter 5 discusses the strain rate sensitivity (SRS) of NiP, Ni10wt%W and Ni20wt%W coatings. The hardness and reduced modulus of all the coatings were also presented. Two different nanoindentation testing techniques were used for the SRS testing: constant strain rate (CSR) method and constant load (CL) method. Results obtained from the two methods will be presented separately. SRS result comparison among all the coating materials obtained by different methods will be discussed. Different influencing factors on the SRS results will also be discussed.

Chapter 6 is a general conclusion of this thesis including the suggested future work.

Chapter 2: Literature review

2.1 Selection of superabrasives

Different grinding wheels have been studied these years, including conventional grinding wheels and superabrasive grinding wheels. Typical conventional abrasives for conventional grinding wheels are aluminum oxide (Al_2O_3) and silicon carbide (SiC). Superabrasives grinding wheels are made from diamond or cubic boron nitride (cBN).³ The hardness and wear resistance of superabrasives are much higher than those of conventional abrasives, which allows superabraisves to have a longer life span compared with conventional abrasives. Since 1930's, natural diamond wheels and artificial diamond wheels were introduced in the grinding applications consecutively. Decades later, cubic boron nitride (cBN) was synthesized to become another important superabrasive in the grinding applications.⁵ Similar to diamond, cBN has a crystalline sphalerite structure. Although cBN (62 GPa) has lower hardness compared to diamond (100 GPa), its higher thermal durability make itself to be superior to diamond. The cBN grains can work under a temperature up to 3500 °F. Although diamond shows an excellent performance on cutting nonferrous materials, it is not very effective in cutting ferrous materials. Due to its chemical affinity, while interacting with ferrous materials, such as iron or steel, diamond would carbonize during the grinding process.^{3, 16, 17} On the contrary, cBN does an excellent job in grinding materials like iron or steels due to its chemical inertness.¹⁸ The inherent sharpness of cBN can provide the workpiece with higher surface integrity and better surface finish.⁵ Due to the enumerated advantages, cBN is becoming more and more popular in the market place. A typical electroplated cBN wheel surface¹⁹ is shown in **Fig. 2**.



Figure 2: A SEM image of an electroplated cBN wheel surface.¹⁹

2.2 Influencing factors on the bonding force

In order to prolong the life span of the grinding wheel, a sufficient bonding force between the grain and the bond coat is required. Many factors in the grinding wheel system may influence the working performance. Li *et al.*^{10, 20} established a finite element model (FEM) to analyze the bonding force of single layered electroplated cBN bond and also study the failure mechanism of the bonding layer. The influencing factors of the bonding force are divided into 3 groups: grain properties, bond properties and the process condition¹⁰. In this review, the grain properties and bond properties would be discussed. Overall, for the grain properties, the bonding force could be influenced by the grain geometry, the grain size and the grain distribution. And for the bond properties, the bonding force could be influenced by the bond thickness and different bond mechanical properties. Thus, a schematic of the influencing factors of the grain properties and bond properties was modified and displayed in **Fig. 3**.



Figure 3: The influencing factors of the bonding force: grain properties and bond properties.¹⁰

The grain properties on the grinding applications can be influenced by grit sizes, grain shape, hardness, toughness, friability, chemical and thermal properties.^{3, 4} Linke⁴ did a comprehensive review on the grit properties that are considered for the grinding tool manufacturing. First of all, the grit size and the grit size distribution would determine how many cutting edges that the tool has. Smaller surface roughness could be achieved with smaller grit size. However, smaller grit size is usually associated with higher machining forces so that the lifespan of the grinding tool would be shorter. Moreover, oversized grits would lead to poor surface quality. Thus, appropriate selection of the grit size and grit size distribution is very important. Another important influencing factor is grit morphology because the grit shape would influence the number of the cutting edge. The grit fracture behavior also varies with different grit shapes. For grit hardness, the value is normally measured by a Knoop or Vicker hardness test. The grit hardness would affect the cuttingedge stability and also the grit sharpness. The hardness of abrasives must be harder than the workpiece in order to achieve the abrasive wear. Grit toughness is the measurement of the ability of a grit to resist the breakage and crack propagation. Friability is an inverse term of fracture toughness, which indicates the tendency for a grain to fracture under certain load.^{3, 4} During the

grinding process, the grits might break which would result into either a smooth surface or a rough surface.⁴ Tougher grit would lead to lower tool wear. However, thermal damage and process vibration might occur due to higher friction. Moreover, the grits that are more friable tend to expose more new cutting edges during grinding. But shorter tool life and form errors might be resulted. The grit embedding depth would also affect the grinding efficiency.²¹ The grit embedding depth is the depth of the grit that is embedded inside the bond from the bond surface. In most cases, efficient grinding would be achieved if the grit embedding depth is below 50% of the grit height.

The bond properties could be affected by the bond thickness, material mechanical properties (hardness, toughness, etc.) and also some time-dependent properties (creep or strain rate sensitivity).^{8, 10} Possible failure patterns (**Fig. 4**) for a electroplated Ni-matrix coating were proposed by Ghosh and Chattopadhyay²². Due to the ambient plating process, there was no residual stress inside the bond so that the grains could be easily dislodged because of the weak mechanical anchorage between the grain and bond. Thus, the thickness of the bond plays a more important role in the grain failures. It was found out that the grains are more easily pulled out at lower bond thickness but tend to stay inside the bond with higher bond thickness. At high level of bond, grains would undergo fracture beyond the bond surface which could lead to new possible cutting edges.



Figure 4: Possible grain failure patterns in electroplated CBN wheels.²²

2.3 Selection and preparation of bond coat

Since both superabrasives and bond would influence the grinding behavior, not only the selection of the superabrasives is important, the bond selection is also noteworthy. As part of the grinding wheel, the bond coat plays few important roles. ^{3, 9} First of all, most importantly, the bond helps to retain the grains inside. If the bonding strength is not strong enough, some of the grains even in good shapes would be pulled out prematurely, which will lead to ineffective grinding. Not only retaining the grains during high-speed grinding process, the bond would also help to resist the centrifugal force. The bond will also allow the grains to be exposed gradually leading to new cutting points with adequate erosion rate. It can also transfer heat resulted from the high-speed operation.^{3, 9} Thus, in order to have an optimized grinding performance, selection of the bond coat need to be considered thoroughly.

Numerous bonding materials have been considered for grinding wheels: nickel alloys, cobalt alloys, titanium, copper, chromium, iron, zirconium, niobium, molybdenum and tantalum. Among all the considerations, nickel, nickel phosphorus, cobalt, cobalt phosphorus and titanium were reported to have a better grain retention ability in the cBN wheel applications due to the irregularity of the coated surface. Nickel coating is preferred by industries because of its outstanding corrosion resistance compared with the other materials.⁷ Nickel alloy coatings are also known for their high resistance in thermal fatigue, thermal shock and creep.¹⁵ The bond coats of grinding tools are designed in both multi-layer and monolayer. Compared with multi-layered superabrasive coatings, single layer superabrasive coating required less superabrasives.^{3, 18} Monolayer cBN tools also have other distinct advantages, including the ability to run at high cutting speed, less complexity in the preparation steps and possibility of reapplication by stripping and recoating.¹

Monolayer coating can be prepared either by brazing or electroplating. One advantage of fabricating the electroplated cBN wheel is that there is no thermally induced residual stress in the bond layer due to its ambient fabricating temperature (60 °C or room temperature).^{1, 15} However, the performance of the electroplated cBN is limited due to the weak mechanical anchorage between the grain and bond. The grains tend to be dislodged during the high grinding process under heavy load.¹ Few researches showed that the brazed mono-layer cBN wheels presented an outstanding result compared with the electroplated coating. Using the brazing technique, a strong chemical bond will be created between the cBN grains and the wheel substrate with the aid of the brazed alloy. The grains in the resulted coating will have higher protrusion height and stronger bonding to the wheel. Surface roughness and residual stress can be optimized by adjusting the grinding parameters.^{1, 23}

Electroless plating is another technique that is used recently in industries for metal bond preparation. The first discovery of electroless plating started from 1940's using nickel. It was found out that the plating process was driven by chemical reduction using specific reducing agents. The reaction would occur consecutively without any applied electricity once the reaction started.²⁴ For this reason, electroless plating is known an autocatalytic technique without the introduction of electricity. The electroless NiP coating exhibits good properties in high hardness. The hardness of the NiP coatings would vary with the P content and also be affected with the heating temperature. It was found out that when the phosphorus content decreases, the hardness would increase.^{25, 26} Research showed that the hardness would reach to maximum for electroless NiP coating if the treated temperature is around 400 °C.²⁷ For as-plated electroless NiP coating, the hardness

increased from 520 HV for high P content (10-12%) to 650 HV for low P content (2-3%).²⁵ When the NiP coating was annealed at 400 °C for one hour, the hardness of NiP coating would then dramatically increased to 1050 HV for high P content and 1200 HV for low phosphorus content. Taheri *et al.*²⁶ also examined the hardness of electroless NiP coatings under 300 °C. For high P content, 11.3%, the hardness of the electroless heat-treated NiP coating ranged from 500 to 550 HV. When the P content decreased to 7.5%, the hardness of the coating increased to a range of 820-900 HV. The prominent advantage of electroless nickel plating is that this technique can offer a coating with uniform thickness no matter the shape or the size of the substrate because each point of the surface has a constant driving potential.^{24, 28} Electroless nickel plating can not only provide the coating with a shiny outlook but also have a good control on the thickness and the volume of the coating.²⁸ Compared with the conventional plating techniques, the metal bonds made from electroless plating have better corrosion properties and wear resistances.^{25, 28} Thus, electroless nickel plating technique attracts more interest these days in the engineering applications.

2.3.1 Conventional electroless plating technique for NiP coatings

Electroless plating is an auto-catalytic process without using external electricity. A basic electroless plating apparatus with temperature control is presented in the **Fig. 5**.²⁹ A basic electroless bath includes metals ions with appropriate concentration, reducing agents, complexing agents, accelerator and inhibitor.²⁵ The pH and temperature of the bath have to be well adjusted to have an optimized reaction.



Figure 2.4

Figure 5: A typical electroless Ni plating apparatus.²⁹

A traditional electroless deposition mechanism of NiP was summaried in a review by Loto²⁸:

$$Na_{2}H_{2}PO_{2} + H_{2}O \rightarrow NaH_{2}PO_{3} + H_{2}$$
$$NiCl_{2} + NaH_{2}PO_{2} + H_{2}O \rightarrow Ni + NaH_{2}PO_{3} + 2HCl$$
$$3H_{3}PO \rightarrow H_{3}PO_{3} + 3H_{2}O + 2P$$

Thickness of the coating could be controlled with the plating time. Longer bath time would result into more deposition material. Moreover, the P content is mainly controlled by the bath acidity. Lower pH would lead to higher P content.²⁸

2.3.2 The pulsed electroplating technique for NiW coatings deposition

NiW coatings have attract a lot of attentions due to their excellent tribological, electrical and electro-corrosion properties.^{30, 31} NiW alloys also present high tensile strength, high hardness, high thermal resistance and high corrosion resistance. The hardness of as-plated NiW is between 650 HV and 750 HV.³¹ After the heat treatment, the hardness of NiW would be around 1200-1400 HV. NiW alloy is also an environmentally friendly material that has been used to substitute hard chromium plating in the aerospace applications. Tungsten (W) cannot be electrodeposited in the aqueous solution, sodium tungstate (Na₂WO₄·2H₂O).^{30, 31} However, NiW can be co-deposited

when iron group-based metal compound is added, such as nickel sulphate (NiSO₄). The W content produced from conventional electroplating technique usually ranges from 13 to 50 wt.%. In order to improve the mechanical properties of the NiW coatings, higher W contents are usually required. Thus, pulse plating is introduced to plate NiW coatings. The major difference between the pulse plating and conventional electrodeposition plating technique is that pulse plating technique uses periodic current. Coatings produced from the pulse plating techniques have lower residual stress, less porosity, higher wear and corrosion resistance. The reaction mechanisms³⁰ were proposed:

, where the citrate $(C_6H_5O_7)^{3-}$ was added as a ligand to form a ternary compound with the tungstate/citrate complex. The ternary complex, $[(Ni)(HWO_4)(Cit)]^{2-}$, would then help to deposit NiW alloy. With appropriate control, the W content could be added up to 76 wt.%. Studies also showed that higher W concentration in the bath and higher absolute value of current density would lead to higher W content in the coating.³² Because of the excellent tribological, electro-corrosion and mechanical properties, NiW alloys have been suggested to replace NiP alloys used on the grinding wheel for the high performance grinding applications.

2.4 Bonding force testing

In order to study the bonding force between the superabrasive grits and the bond coating and to better improve the high-speed grinding performance, single grit high-speed grinding testing has been studied these years.^{10, 12-15} A novel single-grit pull-out technique (**Fig. 6**) was developed by Zhi *et al.* to find the maximum bonding force using the grit-turning tool.¹³ The cBN grain was used

as a tool to cut the rotating workpiece material under high speed. The force curve was generated through the force dynamometer as the grain was removed away from the bond. The sudden drop of the curve refers to the grain dislodgement. The maximum point of the curve refers to the maximum bonding force for the grain. Four types of bond failure mechanisms were observed for the electroplated cBN layer: no wear, grain chipping, grain dislodgement and bond erosion. It was found out that using shaper grains and allocating shaper corner towards the cutting direction can allow the grains to have more possibilities to retain in its original position.¹² The finite element model (FEM) was also developed to support the experimental results.^{10, 13}



Figure 6: Cutting process design to determining the bonding force. (a) The inclined micro-thread turning process. (b) The expected bonding force curve.¹³

Ding *et al.*¹⁴ examined the grinding force and force ratio on the binderless cBN grains with the single layer brazed coating. The critical normal force and tangential force were determined for the grains with different protrusion heights. FEM models were developed later for cBN grits to analyze the grit stress.²¹ It was found out that the major cause of the macro-fracture for the brazed cBN grits was the large brazing-induced tensile strength in the grit-bond junction region.

Yu *et al.*¹⁵ examined the strength of the electroplated nickel alloy coating using a novel grit pullout experimental setup (**Fig.** 7)¹⁵. For this instrument, a piece of grinding wheel was placed on the stage. And the diamond anvil was then brought to the selected single grit on the surface of the

grinding wheel using the 20x optical microscope, meanwhile the normal force and tangential force would be measured. The grit pullout sequence using this instrument was shown in **Fig. 8**.¹⁵ As the tool came to the side of the grit, the horizontal stage would move in order to load the grit for the pullout process. Grit rotation would be observed by seeing the color change. The grit would then be continued extracted from the surface.



Figure 7: grit pullout experimental setup.¹⁵



Figure 8: The grit pullout sequence¹⁵

Interfacial normal and shear strength, which were calculated from the maximum force and the grit impression area, were studied before and after the grinding process. It was found that the average bond strength decreased around 35% at the end of the grinding process. The research also showed that larger grits were dislodged more quickly than smaller grits. During the grit dislodgement process, load redistribution to the surrounding grits were observed, accompanied with attritious and grit fracture, which was presented in **Fig. 9**.¹⁵



Figure 9: Representation of the evolution of the damage process, wherein an initial grit pullout commences, followed by a load redistribution to the surrounding grits within the highlighted damage zone. λ is the average spacing between grit pullout and *r* is the average size of the damage zone.¹⁵

In this thesis, a micromechanical test was used to study the bonding force and energy between the single superabrasive grit and the bond coat. This technique was inspired by the adhesion strength study between the cold sprayed splats and the substrate by Goldbaum *et al.*³³ The adhesion strength testing was performed with a Micro-Combi Scratch Tester with a designed flat-bottom stylus. A schematic of the splat adhesion test is shown in **Fig. 10**.³³ During the test, a normal force was applied to the stylus that was placed around 30 μ m before the splat. And the substrate was then moved below the stylus with a certain speed rate. Using the ideal of this micromechanical test technique, the bonding force testing may be able to perform on the monolayer cBN tool. In the current study, instead of moving away the splats, the stylus would pull out the single cBN grit.



Figure 10: A schematic of splat adhesion test.³³

The adhesion strength can be calculated in the following equation:

$$Adhesion strength(MPa) = \frac{F_T - F_B}{A}$$
(2.1)

where F_T is the tangential force exerted on the stylus, F_B is the baseline force, A is the splat area.

The results exhibited clear trends for F_T versus deposition velocity or temperature. The effect of the particle size on the adhesion strength can be tracked using the micromechanical test. However, if the substrate surface is very rough, the adhesion test cannot be performed since the results would be affected by the noises. Moreover, if the embedded depth of the splats was deeper, the adhesion test cannot be performed as well because the stylus may not be able to shear the splats away.³³

Thus, in order to perform the single grit test, the roughness of the sample surface and the grit population have to be low. The protrusion height, the penetration depth, the grit size and the orientation of the grits may be the limitation of the application of the suggested test. Thus, before testing, characterization of the grits has to be done. In addition, appropriate applied load and the movement speed rate have to be adjusted in order to dislodge the grit.

2.5 Strain rate sensitivity

Materials would have different content of dynamic plastic behavior when they are exerted with dynamic loading. The time-dependent strain rate sensitivity or viscoplasticity is used to define the material sensitivity to the plastic flow.³⁴⁻³⁶ Materials exhibited time dependent plasticity when deforming at an increased temperature.³⁷ Three typical stages (**Fig. 11**)³⁸ would occur when the material is under creep deformation.³⁷ In the primary stage (Stage I) the strain increased with decreased strain rate. The strain starts to increase at a constant stain at the secondary stage (Stage II), which is also named as steady-state creep. In the end, in State III, the materials start to have cavitation and/or cracking, which resulted into an increased strain and strain rate so that rupture occurs. The strain rate sensitivity of materials can be further expressed into a power-law relation.



Figure 11: A schematic plot of power-law creep.³⁸

Chowdhury *et al.*^{39, 40} did several studies on the metal viscoplasticity using modeling. Crystallographic slip resulted from dislocation was found out to be the primary cause of the viscoplastic deformation. Mechanisms, such as twinning, grain boundary sliding, void growth etc., might also be associated with the viscoplastic deformation behavior.^{39, 40} As the material is elastoviscoplastically deformed, a thermodynamic system would accompany. The thermodynamic system would then split into two weakly subsystems that could interact with each other, including and a configurational subsystem and a kinetic-vibrational subsystem. The configurational subsystem indicates the defect motion, rearrangement and also lattice distortion with slower degrees of freedom. The kinetic-vibrational system has fast degrees of freedom and is an indication of the ordinary thermal vibration of atoms. These two processes have their own time scale. As the plastic deformation continues, heat from the configurational subsystem would transfer to the kinetic-vibrational subsystem due to the temperature difference between the two subsystems. Furthermore, Armstrong and Malley⁴¹ did an comprehensive review on the material behaviors at high strain rate. It was found out that as the strain rate becomes very high, the plasticity is mainly controlled by slip or twinning defect generations instead of the dislocation or twinning defect movements which occurs at relatively lower strain rate.

Many models to calculate the SRS of the materials have been proposed.^{36, 42, 43} From previous models, Larour⁴³ summarized a basic equation from the Hollomon equation:

$$\sigma = k\varepsilon^n \dot{\varepsilon}^m \tag{2.2}$$

where σ is the true stress, k is the extended Hollomon parameter, ε is the true plastic strain, $\dot{\varepsilon}$ is the strain rate, n is the strain hardening Hollomon coefficient, m is the strain rate exponent. The strain rate sensitivity could be further derived from Equation (2.2) in a logarithmic form m_{10} or a nature logarithmic form m_e :

$$m_{10} = \frac{d(\log(\sigma))}{d(\log(\hat{\varepsilon}))}$$
(2.3)

$$m_e = \frac{d(\ln(\sigma))}{d(\ln(\dot{\varepsilon}))} \tag{2.4}$$

where $m_{10} = m_e$

In order to better understand the SRS of the materials, different techniques were proposed.^{35, 36, 44} Uniaxial macroscopic testing have been widely used to study the SRS of the materials, such as compression and tensile tests.^{35, 45, 46} Stout and Fallansbee⁴⁵ did the uniaxial tensile and compression tests on the 304L stainless steel with the strain rate ranged from 10^{-4} to 10^4 s⁻¹. Studies showed that different strain rate ranges have different material behaviors. Dislocation/obstacle interaction is the main cause of the SRS when the strain rate is relatively low whereas the material structure change became more important when the strain rate was higher than 100 s⁻¹. Lin and Chen⁴⁶ did the straining hardening ($d\sigma/d\varepsilon$) and SRS study on a Mg alloy using tensile tests with constant strain rates ranged from 10^{-5} to 10^{-2} s⁻¹. It was found out that the SRS of the material decreased with increasing true strain. Moreover, thinner specimens with smaller grain sizes has higher strain-hardening exponent and lower ductility. Higher SRS values was found for thinner specimens. In addition, when the strain rate increased, both yield strength and the ultimate tensile strength increased, and the materials became more brittle fracture.

For the SRS studies, submicron-scale testing have also been proposed.⁴⁴ Due to the small-scale ability and precision, nanoindentation has become an ideal technique to access the mechanical properties of the materials and also study the local deformation mechanism. The first well-known submicron-scale technique was developed by Mayo and Nix: the constant rate of loading (CRL) test.⁴⁴ This method applied constant loading rate during nanoindentation on Pb and Sn, with controlled final displacements. The displacement to time curves were further analyzed to obtain the SRS. The results obtained from the CRL method showed a similarity with the results obtained from the conventional tensile tests. The SRS obtained from nanoindentation method can be define using the following equation^{35, 44, 47}:

$$m_{nanoindentation} = \frac{d(\ln H)}{d(\ln \dot{\varepsilon}_{nanoindentation})}$$
(2.5)

where *m* is the SRS exponent, H is the hardness and $\dot{\varepsilon}_{nanoindentation}$ is strain rate during the nanoindentation.

Another nanoindentation method was also proposed, which was known as constant load (CL) method.^{48, 49} This method involved a long holding period at the maximum peak load. The maximum peak load would be reached rapidly in one second. Information is extracted from the long holding segment. Strain rate and hardness during the long holding segment were studied in pairs to calculate the SRS.

Constant strain rate (CSR) method, another popular nanoindentation technique, was introduced by Lucas and Oliver.⁴⁸ A constant ratio of loading rate to load (\dot{P}/P) was used to keep the strain rate steady during the loading process. They also performed \dot{P}/P change experiment to show that the presence of the steady-state path-independent hardness was due to the strain accumulation.

Both CRL and CSR methods requires large area of the samples to perform indentation because several strain rate-hardness pairs are required to find the relation.⁵⁰ Thus, only samples with uniform surfaces are suitable for these techniques. If the microstructure of the surface varies a lot between different regions, these techniques are not ideal to be used. On the contrary, CL method does not require a large testing area because each indent would give its own SRS information.

Thus, compared with CRL and CSR methods, CL method is more suitable to analyze the SRS for the samples with inhomogeneous microstructures.

Two main disadvantages need to be concerned when performing creep-indentation test, especially in nano-scale size: indentation size effect and thermal drift.⁵¹ Several studies found the SRS changes with different indentation sizes.^{37, 52, 53} Li *et al.*^{52, 53} presented several indentation sizedependent studies on different metal samples. Studies showed that larger indent sizes tend to have higher stress exponent.^{52, 53} The smaller indent size would lead to higher SRS. Moreover, the indentation size effect was also found to have interaction with indentation creep which could cause an SRS error up to 30% if the indentation is shallow.⁵¹ Thus, different indentation sizes would lead to the SRS results of a material to become uncertain. Another important influencing factor is the thermal drift because it would limit the test resolution.⁵¹ The thermal drift rate is normally in the order of 10^{-2} nm/s. A typical drift rate correction model was proposed by Alkorta *et al*⁵¹:

$$1 - 2m^{-1} = (1 - 2\bar{m}^{-1}) \frac{\left(1 - \frac{\lambda t}{\bar{h}}\right)}{\left(1 - \frac{\lambda}{\bar{h}}\right)^2}$$
(2.6)

where \overline{m} is the SRS exponent without thermal drift correction, λ is the constant thermal drift, t is the time, \overline{h} is the uncorrected indentation depth and $\dot{\overline{h}}$ is the uncorrected penetration rate. An alternative nanoindentation method was then developed by Alkorta *et al*, which was known as the strain rate jump test.⁵¹ Specimen were tested at several strain rate levels. This test not only revealed the importance of drift rate correction on the accuracy of the result, but also showed its capacity to minimize the thermal drift and the indentation-size effect.
Many SRS studies have been conducted using different submicron techniques. However, most of the studies examined the materials based on one technique. Only few papers reported the direct comparison between the SRS values obtained from different nanoindentation methods using exactly same specimen.^{50, 51, 54} In this thesis, CSR and CL method were selected to study the strain rate sensitivity of Ni-based alloys. The SRS results obtained by CSR and CL would be discussed in Chapter 5.

Chapter 3: Experimental procedures

3.1 General description of the samples

The single layered NiP and NiP-cBN coatings were prepared using the conventional electroless plating technique. Single layered Ni-W coatings and bi-layered NiP/NiW-cBN coatings with different W contents were prepared using the pulse electroplating technique. No heat treatment was performed. The samples were prepared at Saint Gobain in Northborough, MA. The grit sizes and the grit size distribution on the bond coat were selected by Saint Gobain. All the detailed sample preparation procedures would not be revealed due to confidentiality agreement. The single layered NiP and single layered Ni-W coatings were used for strain rate sensitivity testing (Chapter 5). Single layered NiP-cBN and bi-layered NiP/NiW-cBN coatings were used for adhesion force testing (Chapter 4). A summary of the sample used in this thesis was shown in **Table 1**. A brief literature review for different types of plating techniques was presented in Chapter 2.

| Sample name | Coating material | Surface coating material | Thickness of NiP layer (μm) | Thickness of NiW layer (µm) | Chapters |
|--------------|------------------|--------------------------------|-----------------------------------|-----------------------------------|----------|
| NiP | NiP | NiP | 23.8±2.9 | - | 5 |
| NiP-1 | NiP-cBN | NiP-cBN | 51.1±0.7 | - | 4 |
| NiP-2 | NiP-cBN | NiP-cBN | 45.4±0.2 | - | 4 |
| Ni10wt%W | Ni10wt%W | Ni10wt%W | - | 10.3±1.0 | 5 |
| NiP/Ni10wt%W | NiP/Ni10wt%W - | Ni10wt%W- | | 9.8±0.2 | 4 |
| | cBN | cBN | 42.1±0.3 | | |
| Ni20wt%W | Ni20wt%W | Ni20wt%W | - | 12.9±1.0 | 5 |
| NiP/Ni20wt%W | NiP/Ni20wt%W- | Ni20wt%W- | | 7.9±0.1 | 4 |
| | cBN | cBN | 42.8±0.1 | | |

| | Table 1: | The same | ples used | in | this | thesis |
|--|----------|----------|-----------|----|------|--------|
|--|----------|----------|-----------|----|------|--------|

3.2 Characterization of electro-deposited coatings

3.2.1 Coatings thickness and surface morphologies

To study the surface morphologies of the coatings, the scanning electron microscope (SU-3500, Hitachi, Japan) was used. This scanning electron microscope (SEM) is equipped with a back scattered electron (BSE) detector and an energy dispersive X-ray spectroscope (EDS) detector. With the aid of BSE, grit residuals could be observed inside the coatings. In the BSE images, the coating showed a grey color. The darker color represented the cBN grits or their residuals. Accelerating voltage and working distance were varied from 5 to 20 keV and from 5 to 10 mm, respectively.

For the coating thickness measurement, the samples were cross-sectioned using a low-speed abrasive cutter. The cut samples were cold mounted by the conductive copper filler. Sample grinding was performed using 240, 320, 400, 600 grit abrasive grinding papers followed by using 9, 3 and 1 µm polycrystalline diamond suspension solution. The polished cross-sections of the sample were then imaged using SEM. Five random positions were taken. The thickness of the coatings was then measured using an open source software ImageJ.

3.2.2 Surface and grit profiles

The roughness of the coatings for both Chapter 4 and Chapter 5 were analyzed using a white light interferometric surface profiler (NewView 8000, Zygo instruments, USA). Surface profiles and images were taken at 50x objective, 2x FOV to avoid the effect of waviness. For the grit adhesion test in Chapter 4, samples with grits were analyzed using the optical surface profiler (NewView 8000, Zygo instruments, USA). Using the coating surface as the baseline, surface profiles for all the grits were taken at 10x objective, 1x FOV. In addition, signal oversampling was used in Zygo

scanning in order to extract very weak signal from challenging surface, such as steep slopes. Line profiles were extracted to see whether the grits were vertical or sloped with respect to the matrix. Grits that were vertical or slightly sloped with respect to the matrix were selected for the test. The grits that were selected to be tested also have to be very well spaced from the other grits. Grits that form agglomerates are not appropriate for testing. An example of testable grits and agglomerate is shown in **Fig. 12**.



Figure 12: SEM image of NiP/Ni20wt%W. The grits that can be tested were pointed with the red arrows. Agglomerates that are not appropriate for testing were squared with the blue box.

A standard interference objective consists of a beam splitter and a reference mirror.^{55, 56} When the optic is focused on the sample surface, as the white light pass through the interferometer, the light would split by the beam splitter to the reference mirror and the sample. Interference pattens, known as fringes, would be observed as the light reflected back from the reference mirror and the sample. The interference signals were then analyzed into frequency domain by Fourier analysis. The fringe contrast for each image pixel would be calculated by the system to obtain the surface height information. Thus, before the test, the optic was brought down to focus on the cBN grits to obtain the grit information, such as the grain top-view geometry and the grain height above the surface.

After the scratch test, for the dislodged grits, the optic was also brought down to focus on the matrix to obtain the depth of the cavity.

3.3 Grit adhesion testing

3.3.1 Sample descriptions

Two single layered NiP-cBN coating samples, one double layered NiP/Ni10wt%W-cBN coating sample and one double layered NiP/Ni20wt%W-cBN coating sample (**Table 1**) were used in Chapter 4. The two NiP-cBN samples were analyzed separately, which were named as NiP-1 and NiP-2 in the following sections. A schematic single layler cBN coating was presented in **Fig. 13**. The SEM images of the cross section of the single layer NiP coating and double layer NiP/NiW coating was presented in **Fig. 14**.



Figure 13: A schematic single layer cBN coating.



Figure 14: SEM images of the cross-section of the single layer NiP coating (left) and the double layer NiP/NiW coating (right).

3.3.2 MicroCombi scratch tester and the tip geometry

Grit adhesion testing were carried out using scratch tester (Surface Testing Platform STeP⁴, Anton Parr, Switzerland) (**Fig. 15**), is located in the Nano-Mechanics and Tribology Laboratory (NMaTL), in Materials Engineering Department at McGill University. The STeP⁴ is equipped with three measurement systems: Micro-indentation and scratch test (MCT) system or Micro-Combi Tester (MCT), Nano-Hardness Tester indentation (NHT) system and optical microscope with a rotating turret. The scratch mode on the MCT system was used to push on cBN grains in an attempt to remove them. Under scratch mode, the instrument records the tangential force and surface profile of different samples. The moving stage can move in any direction with an accuracy of 1 µm. The normal load for scratch testing ranges from 25 mN to 30N.



Figure 15: STeP⁴ instrument setup.

A custom-designed wedge-shaped manufactured tungsten carbide (WC) indenter (**Fig. 16**) was used to perform the scratch tests. The designed tip is a flat-bottomed cone with the flat leading edge. The leading edges of the tip are designed to be 0.100 ± 0.005 mm (100 ± 5 µm) and 0.500 ± 0.005 mm (500 ± 5 µm), respectively. The conical angles are manufactured to be 20° for tip with a 100 µm leading edge and 30° for tip with a 500 µm leading edge.



Figure 16: The design of the tungsten carbide tip: (a) The tip with a 100 μ m leading edge and a 20° conical angle and (b) the tip with a 500 μ m leading edge and a 30° conical angle.

3.3.3 Experimental procedures

Scratches were performed over a distance of 0.4-1 mm, with the cBN grit at the center of the track. A rate of 0.15 mm/s and a normal load of 0.1 N were used to test the samples. Some tests were performed with larger tips and/or higher forces. However, tip was found out to be highly damaged. Moreover, the number of grits that can be tested was highly limited by the higher cross-sectional tip because grits were required to be more widely spaced. Thus, a 100 μ m tip was used. The schematic of the scratch test is displayed in **Fig. 17**.



Figure 17: A schematic of the scratch test

Before the test, each grit was imaged by Zygo. The testing side of the grit was found out to have either a flat side or a corner side (**Fig. 18**). Grits that tested from different sides were categorized and discussed in Chapter 4.



Figure 18: A schematic of top view of the grain orientation with respect to the scratch tip

Protrusion height was also taken for each tested grit using Zygo before the test. The protrusion height (Fig. 21(a)) is the grit height that is above the matrix surface. After testing, different grit behaviors (Fig. 19) would occur. The grits were either completely dislodged, partially dislodged, fractured or remained intact.



Figure 19: A schematic of testing possibilities. Grits could be completely dislodged, partially dislodged, fractured or remain intact.

A tangential force-displacement curve (**Fig. 20**) is generated during the test. Peak force started to rise once the tip is in contact with the grit. If the grit is dislodged, a single peak would be observed. The highest point of the peak which is called maximum tangential force (max. Ft), and the area under the peak, which is the energy required to dislodge the grit, were noted in order to study the effect with the protrusion height and bond depth of the grits.



Figure 20: An example of the generated data (A plot of tangential force (Ft) versus position (X)

Grits that were completely dislodged left a clean cavity after the test. Thus, the depth that the grit was embedded inside the matrix for completely dislodged grits, referred as bond depth (**Fig. 21(b**)), was measured using Zygo. Grits after testing were imaged using SEM-SU3500. Using the SEM images, the cavity sizes for completely dislodged grits were measured using ImageJ.



Figure 21: Schematic of the protrusion height (a) and bond depth (b) of a grit.

3.3.4 Calculations

In order to see the grit behavior after tests for each sample, the fractions of the dislodged grits, partially dislodged grits and intact grit for each sample were calculated using the following equations:

$$Dislodged ratio = \frac{No.of \ dislodged \ grits}{Total \ number \ of \ tested \ grits}}$$
(3.1)

$$Partially \ dislodged \ ratio = \frac{No.of \ partially \ dislodged \ grits}{Total \ number \ of \ tested \ grits}}$$
(3.2)

$$Intact \ ratio = \frac{No.of \ intact \ grits}{Total \ number \ of \ tested \ grits}}$$
(3.3)

During the tests, grits can be scratched from corner side or the flat side. Thus, the grit behavior from different testing side for each sample was also interested. Corner dislodged ratio and flat dislodged ratio for each sample were obtained from the following equations:

$$Corner dislodged ratio = \frac{No.of grits that were dislodged from the corner side}{Total number of the grits tested from the corner side}$$
(3.4)
Flat side dislodged ratio = $\frac{No.of grits that were dislodged from the flat side}{Total number of the grits tested from the flat side}$ (3.5)

size, protrusion height, bond depth, Max Ft and energy between different types of samples. For the 2 tailed student t test, two samples are assumed to have unequal variance. The 0.05 probability \mathbf{p} was used as the critical value to determine whether the samples had significant values between each other. The null hypothesis would suggest that there was no significant difference between the two samples. When the calculated \mathbf{p} was higher than 0.05, the null hypothesis would be accepted. On the other hand, when the calculated \mathbf{p} was lower than 0.05, the null hypothesis would be rejected, which indicated that there was significant difference between the sample. If there is significant difference between two samples, an asterisk symbol, *, would show on the statistical plot. If no significant difference is found between the two samples, no asterisk symbol would be shown.

3.4 Hardness, reduced modulus and strain rate sensitivity (SRS) testing

3.4.1 Sample descriptions

Single layered NiP, Ni10wt%W and Ni20wt%W samples with no cBN grits embedded were used to conduct the strain rate sensitivity study. Hardness and reduced modulus for each coating were also measured. The result discussion was demonstrated in Chapter 5. The sample details were listed in **Table 1**.

3.4.2 Nanoindentation testing

A Hysitron Ubi 3 (Hysitron Incorporated, Minneapolis, MN) system was used for both constant strain rate (CSR) method and constant load (CL) method. A diamond Berkovich indenter was used. All the tests were performed directly on the sample surface. The nanoindentation system contains a base, including a moving stage, the optics and the TriboScanner.⁵⁷ The optics on the Z stage allow to focus on the sample and to choose the testing position on the sample. The TriboScanner on the Z stage allows sample imaging before and after the tests. Samples were mounted to the XY stage through the magnetic attraction. The most important component of the nanoindentation system a linear force or displacement signal with high sensitivity and large dynamic range. A schematic of the capacitive transducer was presented in **Fig. 22**. The indenter is mounted at the middle plate by

a spring. A force is applied by applying a large DC bias (up to 600 V) to the bottom plate. In the meanwhile, the middle plate would be pulled down due to the electrostatic attraction between the middle and the bottom plate. The system will determine the applied force using the magnitude of the voltage, and the displacement using the position of the plate, respectively. The maximum normal load force of the used transducer is around 11 mN.



Figure 22: A schematic of the capacitive transducer.⁵⁷

A typical load-displacement curve is displayed in Fig. 23.⁵⁸ Hardness H and reduced modulus E_r are calculated using Oliver-Pharr method:



DISPLACEMENT, h Figure 23: A schematic of a typical load-displacement curve.⁵⁸

$$H = \frac{P_{max}}{A} \tag{3.6}$$

$$S = \frac{2}{\sqrt{\pi}} E_r \sqrt{A} \tag{3.7}$$

where P_{max} is the peak load, A is the projected area and S is the stiffness. The stiffness is the maximum tangent of the initial part of the unloading curve. Samples were cross-sectioned and polished for the hardness and reduced modulus measurement. During nanoindentation, the following parameters were used: 5-s loading, 2-s holding at peak load and 5-s unloading.

For a perfect Berkovich indenter, the projected contact area A is calculated using $A(h_c) = C_0 h_c^2$, where h_c is the contact depth of the indenter and C_0 is the coefficient, which is 24.5 for a perfect Berkovich tip geometry.⁵⁸ However, after a number of test, the indenter might have different degree of defect. Thus, in order to get reliable nanoindentation results, area function calibration was carried on the Berkovich indenter. The calibration was carried on fused quartz with the elastic modulus of 72 GPa. Ten indents were performed at each set load (10,000 µm, 9500 µm, 9000 µm...5000 µm).⁵⁷ With all set of data, the incorporated software fit the A vs hc curve up to 4 polynomial orders. The recalibrated projected area of an indent with a Berkovich indenter was then calculated using the following equations:

$$A(h_c) = 24.5h_c^{2} + C_1h_c + C_2h_c^{1/2} + C_3h_c^{1/4}$$
(3.8)
$$h_c = h_{max} - 0.75\frac{P_{max}}{s}$$
(3.9)

where h_c is the contact depth of the indent, h_{max} is the maximum depth of the indent and C_n were coefficients calculated from the area function calibration.

For the strain rate sensitivity testing, all the nanoindentations were performed on the sample surface. Before indenting, the indenter was set to contact sample surface with a 2 μ N load for over an hour to stabilize the instrument. Thus, the drift rates would be kept in consistent to below than

0.1 nm/s. During unloading, the load dropped to a load of 20% of the peak load for a 40-s drift assessment. Tests with any large changes (>0.05 nm/s) in the drift rate were discarded.

3.4.3 Calculations of strain rate sensitivity (SRS)

The strain rate $(\dot{\varepsilon})$ is calculated by dividing the displacement rate (\dot{h}) to displacement (h), Equation (3.10). The hardness (H) was obtained by dividing the applied load (P) to the indent project area (A), Equation (3.11). In this study, instead of contact depth, the instantaneous total depth was used to calculate hardness.

$$\dot{\varepsilon} = \frac{\dot{h}}{h}$$
 (3.10)
 $H = \frac{P}{A}$ (3.11)

The equation used to calculate the SRS was listed in the following⁵⁰:

$$m = \left(\frac{\partial \ln H}{\partial \ln \dot{\varepsilon}}\right)_T \tag{3.12}$$

3.4.4 Constant strain sensitivity (CSR) method

The following ratios of loading rate (\dot{P}) to load (P) were used: 0.02, 0.03, 0.05, 0.1, 0.2, 0.3, 0.5, 1, 2 and 3 s⁻¹. The load was set to reach to a 500 µN preload in 2 s and hold at the preload for 5 s. The indenter would hold at the peak load, which was 10 mN for 5 s. An example of load-displacement curve of Ni20wt%W at a strain rate of 0.1 s⁻¹ was shown in **Fig. 24**.



Figure 24: An example of load-displacement curve of Ni20wt%W at a strain rate of 0.1 s⁻¹. The 5-s 500 μN preload holding period plotted in red curve was magnified for a better view.

The experimental \dot{P}/P ratio was calculated from the exponential fit of the loading curve after the 500 μ N preload:

$$P = P_0 e^{kt} \qquad (3.13)$$

where P_0 is the load when t = 0 and k is the experimental \dot{P}/P ratio.

The strain rate was calculated from the time derivative of the exponential fit of the displacementtime loading curve. The averaged strain rate of the second half of the test was taken due to the instability in the early stage. The hardness at the peak load was used in this CSR method. At least 12 indents were performed at each \dot{P}/P ratio for each sample. Each indent was at least 10 µm away from each other.

3.4.5 Constant load (CL) method

In the CL study, a peak load of 10 mN was used for each test. The indenter was set to reach the peak load in 1 s. The hold period was 100s. An example of displacement-time curve was presented in **Fig. 25**.



Figure 25: An example of CL displacement-time curve of NiP sample. Loading, holding, drift assessment and unloading segments were marked separately. The long holding period used for the analysis was marked in red.

After testing, the following equation was used to fit the displacement-time curve of the hold segment using Origin 2016 (origin labs, USA)⁵⁰:

$$h(t) = h_0 + a(t - t_0)^P + kt \qquad (3.14)$$

The instantaneous displacement rates were the derivative of Equation (3.14). Using Equation (3.10), the instantaneous strain rate could then be derived. The hardness was calculated using Equation (3.11), where the load is the average load during the hold. For each specimen, 30 indents with at least 10 µm indent spacing were performed.

Chapter 4: Experimental results and discussion of single grit adhesion tests

Single grit adhesion tests were performed for different types of superabrasive coatings. The superabrasive coatings were composed of the bond coat and cubic boron nitride grits. In this chapter, bond coats with different materials were tested: NiP, NiP/Ni10wt%W and NiP/Ni20wt%W. The surface morphology and the grit height distribution of the coatings will be presented. As discussed in **Chapter 2**, the adhesion between the grit and the bond has many influencing factors. In this chapter, the effect of grit size, protrusion height, bond depth, grit orientation with respect to the adhesion test direction on the bonding force for different types of coatings will be discussed separately. Furthermore, for each sample, grits showed a wide range of maximum tangential force (Max. Ft) and energy (E) values. Grits on the two NiP coatings would be discussed and compared in terms of bond depth, protrusion height, Max. Ft and E. SEM images of the grit cavities after grit dislodgement for NiP and Ni20wt%W coatings were presented.

4.1 Surface morphologies of the coatings

The surface morphologies of different coatings under different magnifications were shown in **Fig. 26** and **Fig. 27**. The surface roughness of each coating measured by the white light interferometric surface profiler was showed in **Table 2**. All the coatings showed nodular morphology. Compared with the NiP/Ni10wt%W coatings, the NiP coating had a much smoother surface, with a lower measured surface roughness. Larger agglomerates were observed on the NiP/Ni10wt%W surface.

Moreover, when comparing the surface morphology between NiP/Ni10wt%W coating and NiP/Ni20wt%W coating, NiP/Ni10wt%W coating showed a smoother surface than NiP/Ni20wt%W coating, which further indicates that the roughness of NiP/Ni20wt%W is higher than NiP/Ni10wt%W coating. Bulges (**Fig. 28**) were observed on the surface of most of coatings. Surface of the bulges displayed a nodular morphology that was similar to matrix.



Figure 26: Surface morphology: (a) NiP and (b) NiP/Ni10wt%W coatings.



Figure 27: Surface morphology: (a) NiP/Ni10wt%W and (b) NiP/Ni20wt%W coatings.



Figure 28: Surface morphology of the bulge: (a) NiP/Ni10wt%W and (b) NiP/Ni20wt%W coatings. Bulges were observed on both NiP/Ni10wt%W and NiP/Ni20wt%W samples. The bulges on both materials showed nodular morphology (presented in the black frame), which were similar to the morphology of the matrix (presented in the red frame).

Table 2: Surface roughness of NiP, NiP/Ni10wt%W and NiP/Ni20wt%W coatings

| | NiP | NiP/Ni10wt%W | NiP/Ni20wt%W |
|------------------------|------|--------------|--------------|
| Surface roughness (nm) | 12±3 | 22±7 | 32±21 |

4.2 Summary of grit testing behaviors

As mentioned in the experimental section, a tip scanning speed of 0.15 mm/s and at a normal load of 0.1 N were used for the single grit test. During the scratching tests, grits were either completely dislodged, partially dislodged, fractured or remained intact and fully bonded. Examples of different testing results were presented in **Fig. 29**. When a grit is completely dislodged after a test, that resulted in a cavity clean from grit residue. For a grit that is partially dislodged, part of the resulted cavity would be clean. However, residues of the grit would still remain inside the cavity. When

the grit is fractured, part of the grit would remain inside the bond. But the top or the side of the grit would be fractured. For the intact grit, the whole grit will remain inside the bond with mild damage on the surface.





Figure 29: SEM SE and BSE images of different testing result possibilities: grits are (a) completely grit dislodged, (b) partially dislodged, (c) fractured and (d) remained intact.

Two single layered NiP-cBN samples (named as NiP-1 and NiP-2), one double layered NiP/Ni10wt%W-cBN (named as NiP/Ni10wt%W) sample and one double layered NiP/Ni20wt%W-cBN (named as NiP/Ni20wt%W) sample were tested. The sample coating details were summarized in Chapter 3, **Table 1**. The grit behaviors for NiP-1, NiP-2, NiP/Ni10wt%W and the NiP/Ni20wt%W bond coats were summarized in **Table 3**.

| | NiP-1 | NiP-2 | NiP/Ni10wt%W | NiP/Ni20wt%W |
|---------------------|-------|-------|--------------|--------------|
| Total tests done | 37 | 57 | 63 | 58 |
| No. of dislodged | 17 | 16 | 17 | 18 |
| grits | | | | |
| No. of partially | 11 | 5 | 14 | 12 |
| dislodged and | | | | |
| fractured grits | | | | |
| No. of intact grits | 9 | 36 | 32 | 28 |

 Table 3: Sample behaviors after single grit tests.

Table 4: Dislodged ratio, Intact ratio and partially dislodged/fracture ratio summary for tested samples.

| | NiP-1 | NiP-2 | NiP/Ni10wt%W | NiP/Ni20wt%W |
|--------------------------|-------|-------|--------------|--------------|
| Dislodged ratio | 0.46 | 0.28 | 0.27 | 0.31 |
| Intact ratio | 0.24 | 0.63 | 0.51 | 0.48 |
| Partially | 0.30 | 0.09 | 0.22 | 0.21 |
| dislodged/fracture ratio | | | | |

Dislodged ratio, intact ratio, partially dislodged/fracture ratio summarized in Table 4 were calculated for each sample. Among the 4 types of the samples, grits are more easily dislodged from the NiP-1. The dislodged ratio of NiP-1 sample is approximately 1.6 times of that for NiP-2 sample. The intact ratio of NiP-2 sample is around 2.6 times of that for NiP-1 sample. For NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples, the dislodged ratios were similar to one another. The intact ratios of NiP/Ni10wt%W sample and NiP/Ni20wt%W sample were similar as well, whereas NiP-2 sample showed a slightly higher intact ratio compared to the two NiP/NiW samples. During the grit adhesion testing, the grit itself might crack or fracture. The NiP-2 sample displayed lowest partially dislodged/fractured ratio with highest intact ratio among all samples and a low dislodged ratio, following by NiP/NiW samples. The grit dislodged ratio were similar among NiP-2 coating and NiP/NiW samples. In fact, the two Ni-W samples exhibit similar dislodged, partially dislodged/fractured, and intact ratio values. Overall, grits are more easily dislodged from the NiP-1 sample. In order to measure the bonding force between the grit and bond coating, grits that were completely dislodged were studied. Maximum tangential force (Max. Ft) and energy (see Chapter 3 for the details of the measurements and calculations) for completely dislodged grits were analyzed from the plot of tangential force versus displacement.

4.3 Effect of the grit size

Grit size is considered to be one of the influencing factors on the adhesion force. The size of the grits for different samples were summarized in **Table 5**. The statistical plot of the grit size for different samples were shown in **Fig. 30**.

| | NiP-1 | NiP-2 | NiP/Ni10wt%W | NiP/Ni20wt%W |
|-------------------------|----------|----------|--------------|--------------|
| Grit size | 15.8±6.3 | 18.3±7.9 | 15.0±5.9 | 14.7±6.1 |
| $(\times 10^3 \mu m^2)$ | | | | |

Table 5: Grit size of NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples



Figure 30: Statistical comparison of grit size among NiP -1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. No significant difference was observed among the samples.

The cBN grits on all the received samples were from the same batch. Thus, it is expected that the grit sizes on all the received samples are similar to one another. The measured grit sizes on the NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples were 15777±6269, 18254±7928, 15002±5860 and 14735±6095 µm², respectively. Based on the statistical analysis, no significant difference in grit size was found among NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples, which showed that all the sizes of the completely dislodged grits on the tested samples were in the same range. Thus, the grain size in this study played a minor role.

4.4 Effect of the protrusion height on Max Ft and energy

Grits protrusion height is another possible influencing factor for the bonding strength. The grit protrusion heights for different samples were measured using optical profilometry before the single grit testing. The influence of grit protrusion height on the adhesion force between the grit and bond coat was tested. **Table 6** summarized the values of averaged protrusion height, Max Ft and energy for each sample. **Fig. 31** showed Max Ft and energy versus protrusion height for completely dislodged grits in different samples. The statistical comparison results for the samples were presented in **Fig. 32, 33, 34**.



Figure 31: Effect of the protrusion height on Max Ft and energy for NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples: (a) Max Ft versus protrusion height and (b) Energy versus protrusion height. Grits on NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W coatings showed a wider range of Max Ft and energy values compared with NiP-1 coating.

| | NiP-1 | NiP-2 | NiP/Ni10wt%W | NiP/Ni20wt%W |
|----------------------------|-----------------|-----------------|-------------------|-----------------|
| Averaged protrusion height | 127±26 | 117±30 | 116±25 | 108±28 |
| (μm) | | | | |
| Average Max Ft (N) | $0.8{\pm}0.7$ | 1.9±1.6 | 1.9±1.2 | $1.5{\pm}1.0$ |
| Average energy (mJ) | 0.02 ± 0.02 | 0.05 ± 0.05 | $0.05 {\pm} 0.04$ | $0.04{\pm}0.03$ |

Table 6: Data summary of the effect of the protrusion height to the Max Ft and energy.



Figure 32: Protrusion height comparison among NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. The asterisk indicates the significant difference in protrusion height between NiP-1 and NiP/Ni20wt%W samples.



Figure 33: Max Ft comparison among NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. The asterisk indicates the significant difference between NiP-1 and NiP-2; NiP-1 and NiP/Ni10wt%W samples, and NiP-1 and NiP/Ni20wt%W samples.



Figure 34: Energy comparison among NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. The asterisk indicates the significant difference between NiP-1 and NiP-2; NiP-1 and NiP/Ni10wt%W samples.

The comparison of the grit protrusion height between each sample was presented in **Fig. 32**. The grit protrusion height on NiP-1 sample is significantly higher than that on NiP/Ni20wt%W samples. No significant difference was found among the other samples. Thus, except the pair of NiP-1 and NiP/Ni20wt%W samples, grits on all the other samples have similar protrusion heights. From **Table 6**, NiP-1 has the smallest values of the average Max Ft and energy among all samples. Significant differences in Max Ft (**Fig. 33**) were found between the NiP-1 and NiP-2; the NiP-1 and the NiP/Ni10wt%W samples, and NiP-1 and NiP/Ni20wt%W samples, respectively. Moreover, significant differences on energy (**Fig. 34**) were found between the NiP-1 and NiP-2, and NiP-1 and NiP/Ni10wt%W samples. There is no significant difference on energy between NiP-1 and NiP/Ni20wt%W samples.

Although the significant difference on protrusion height between the NiP-1 and NiP/Ni20wt%W samples might result into the significant difference on Max Ft, there was no significant difference on energy between the NiP-1 and NiP/Ni20wt%W sample. Some of the samples that has no significant difference to each other on protrusion height were still observed to have significant difference on Max Ft and energy. For example, no significant difference on protrusion height were found between NiP-1 and NiP/Ni10wt%W sample, but significant differences on Max Ft and energy were found between these 2 samples. If grits have the same range of protrusion height but show different Max Ft and energy on different materials, the sample material might be the influencing factor. However, same trend was observed between sample NiP-1 and sample NiP-2. Thus, it is hard to conclude that the coating material has influence on the adhesion strength. Furthermore, the protrusion height of the grits on the NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples were in the same range. No significant differences were found on the Max Ft and energy among these 3 types of samples. Overall, the protrusion height did not present a clear effect on the adhesion force. It is also ambiguous to define whether the coating material has influence on the strength force between the grit and the bond. Thus, more effects were studied in the following sections.

4.5 Effect of the bond depth on Max Ft and energy

The bond depth is also assumed to be one of the influencing factors. The effects of the bond depth to the Max Ft and energy for completely dislodged grits were shown in **Table 7** and **Fig. 35**. The 2-tailed student t-tests (**Fig. 36**) were performed to see the significant difference.



Figure 35: Effect of the bond depth to the Max Ft and energy for NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples: (a) Max Ft versus bond depth and (b) Energy versus bond depth.

| Table 7: Data summary of the effect of the bond dep | oth to the Max Ft and energy. |
|---|-------------------------------|
|---|-------------------------------|

| | NiP-1 | NiP-2 | NiP/Ni10wt%W | NiP/Ni20wt%W |
|-------------------------|-----------------|-------------------|-----------------|-----------------|
| Averaged bond depth(µm) | 27±8 | 38±13 | 43±6 | 43±6 |
| Average max Ft (N) | $0.8{\pm}0.7$ | $1.9{\pm}1.6$ | 1.9±1.2 | $1.5{\pm}1.0$ |
| Average energy (mJ) | $0.02{\pm}0.02$ | $0.05 {\pm} 0.05$ | 0.05 ± 0.04 | $0.04{\pm}0.03$ |



Figure 36: Bond depth comparison among NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W sample. The asterisk indicates the significant difference between NiP-1 and NiP-2; NiP-1 and NiP/Ni10wt%W; and NiP-1 and NiP/Ni20wt%W.

Grits on the NiP-1 has the lowest bond depth (27±8 µm) among the 4 types of samples. Bond depth of NiP-2, NiP/Ni10wt%W samples and NiP/Ni20wt%W samples were 38±13 µm, 43±6 µm and 43±6 µm, respectively, which were significantly higher than the bond depth of the NiP-1 based on statistical analysis. No significant differences on bond depth were found among NiP-2, NiP/Ni10wt%W samples and NiP/Ni20wt%W samples. As shown in **Section 4.4 (Fig. 33)**, the averaged max Ft of NiP-1 is significantly lower than NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. No significant difference on Max Ft was found among NiP-2, NiP/Ni10wt%W samples and NiP/Ni20wt%W samples. For energy comparisons, the averaged energy of NiP-1 is significantly lower than that of NiP-1, there is no significant difference between NiP-1 and NiP/Ni20wt%W samples. Moreover, no significant difference was found in energy among NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. Combined the statistical result of bond depth, Max Ft and energy, it can be seen that the bond depth plays an important role

on the adhesion force. Grits with lower bond depth would require less force and energy to be dislodged. In order to see the material effect, max Ft and energy for each grit were normalized to the bond depth. The normalization summary was shown in **Table 8**.

| Table 8: Bond depth normalization result summa | ary for all the tested samples. |
|--|---------------------------------|
|--|---------------------------------|

| | NiP baseline | NiP Ref-1 | NiP/Ni10wt%W | NiP/Ni20wt%W |
|-------------------|---------------|-----------------|--------------|-----------------|
| Max Ft/bond depth | 0.03±0.03 | 0.05 ± 0.04 | 0.04±0.03 | $0.04{\pm}0.03$ |
| (N/µm) | | | | |
| Energy/bond depth | $0.8{\pm}0.8$ | 1.5±1.4 | 1.2±1.0 | 0.8±0.7 |
| (J/µm) | | | | |



Figure 37: Max Ft/bond depth comparison among NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. No significant difference was found among the 4 samples.



Figure 38: Energy/bond depth comparison among NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. No significant difference was found among the 4 samples.

When the Max Ft and energy were normalized to bond depth, it was found that there was no any significant difference among all the tested samples, which further indicate that the coating materials barely influence the force or the energy.

4.6 The combined effect of protrusion height and bond depth on Max Ft and

energy

As mentioned in the literature review section, efficient grinding would be achieved if the grit embedding depth is below 50% of the grit height.²¹ Thus, the effect of the ratio of the embedding depth to the total grit height on the adhesion strength is also interested to be studied. In this section, the ratio of protrusion height to bond depth was studied. Higher the value of protrusion height to bond depth, less part of the grit will be embedded inside the bond. The result summary was shown in **Table 9**. Statistical results were presented in **Fig. 39**.

| | NiP-1 | NiP-2 | NiP/Ni10wt%W | NiP/Ni20wt%W |
|------------------------|-----------------|-----------|-----------------|---------------|
| Protrusion height/bond | 5.1±1.9 | 3.7±2.3 | $2.8{\pm}0.8$ | 2.6±0.8 |
| depth | | | | |
| Average max Ft (N) | $0.8{\pm}0.7$ | 1.9±1.6 | 1.9±1.2 | $1.5{\pm}1.0$ |
| Average energy (mJ) | 0.02 ± 0.02 | 0.05±0.05 | 0.05 ± 0.04 | 0.04±0.03 |

Table 9: Protrusion height to bond depth result summary for all the tested samples.



Figure 39: protrusion height/bond depth comparison among NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W sample. Significant difference was found between NiP-1 and NiP/Ni10wt%W samples; and NiP-1 and NiP/Ni20wt%W samples.

It can be seen that the embedding ratio of the grits on the NiP-1 is the lowest among the 4 type of samples. More parts of the grits were embedded inside the NiP/NiW samples. The result was also consistent with the bond depth comparison. For the embedding ratio, no significant difference was found between the 2 NiP samples. Significant difference was found between NiP-1 and the NiP/Ni10wt%W samples and NiP-1 and NiP/Ni20wt%W samples. No significant difference was found among the NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. As discussed previously, it was found out that the material did not have a major influence on the adhesion force. Thus, when looking into the ratio of protrusion height to bond depth, grits with higher embedding ratio tend to be dislodged with higher force and energy.

4.7 Tested side effect on Max Ft and energy

Due to the irregular shapes of cBN, it is inevitable that tests were performed either on the flat side or the corner side. Thus, it is also interested to see whether the testing side have influence on Max Ft and energy. A summary of the number of dislodged grits tested from different sides for all the samples were shown in **Table 10**. The dislodged grits on NiP-2 sample were all tested from the corner side. In this section, NiP-1, NiP/Ni10wt%W and NiP/Ni20wt%W samples will be discussed. The 2-tailed student t-tests were performed to see if there is any significant difference between the 2 testing conditions for each type of the sample.

Table 10: Summary of the number of dislodged grits tested from flat side and corner side for all the tested samples

| Sample name | Number of grits tested from | Number of grits tested from |
|--------------|-----------------------------|-----------------------------|
| | flat side | corner side |
| NiP-1 | 4 | 13 |
| NiP-2 | 0 | 16 |
| NiP/Ni10wt%W | 6 | 11 |
| NiP/Ni20wt%W | 6 | 12 |

4.7.1 NiP-1 testing side comparisons

Table 11: Flat side and corner side data comparison: NiP-1

| Parameter | Flat side | Corner |
|------------------------|------------------|-----------------|
| Protrusion height (µm) | 146±26 | 122±24 |
| Bond depth (µm) | 26±7 | 28±8 |
| Max Ft (N) | $0.39{\pm}0.03$ | $0.92{\pm}0.72$ |
| Energy (mJ) | $0.01{\pm}0.007$ | $0.02{\pm}0.02$ |


Figure 40: NiP-1 sample: protrusion height and bond depth comparisons for grit tested from corner side and flat side.



Figure 41: NiP-1 sample: Max Ft (Left) and energy (Right) comparisons for grit tested from corner side and flat side. Grits that tested from corner side showed significantly higher Max Ft than grits tested from flat side.

No significant difference was found in protrusion height and bond depth between the 2 testing side conditions, which mean the grits tested through different side were tested in the similar ranges of protrusion height and depth. When comparing the Max Ft between the grits tested from 2 sides, the Max Ft of grits that were dislodged from the corner side $(0.92\pm0.72 \text{ N})$ was significantly higher than the grits dislodged from the flat side $(0.39\pm0.03 \text{ N})$. The energy of the grits dislodged from the

corner side was higher than the energy of the grits dislodged from the flat side. However, no significant difference was found in energy between the 2 testing side conditions.

4.7.2 NiP/Ni10wt%W samples testing side comparisons

| Parameter | Flat side | Corner |
|------------------------|-------------------|-----------------|
| Protrusion height (µm) | 114±32 | 117±22 |
| Bond depth (µm) | 43±4 | 43±7 |
| Max Ft (N) | 1.58 ± 1.31 | 2.00±1.25 |
| Energy (mJ) | $0.05 {\pm} 0.04$ | $0.06{\pm}0.05$ |

Table 12: Flat side and corner side data comparison: NiP/Ni10wt%W



Figure 42: NiP/Ni10wt%W samples: protrusion height and bond depth comparisons for grit tested from corner side and flat side.



Figure 43: NiP/Ni10wt%W samples: Max Ft (Left) and energy (Right) comparisons for grit tested from corner side and flat side.

For NiP/Ni10wt%W samples, no significant difference was found between bond depth and protrusion height for different testing side conditions. Higher force and energy were required to dislodge the grit from corner side for NiP/Ni10wt%W sample. However, no significant difference was found in Max Ft and energy for grits testing from different sides.

4.7.3 NiP/Ni20wt%W samples testing side comparisons

| Parameter | Flat side | Corner |
|------------------------|-------------------|-----------|
| Protrusion height (µm) | 89±36 | 117±18 |
| Bond depth (µm) | 44±5 | 43±7 |
| Max Ft (N) | 1.01±0.16 | 1.76±1.18 |
| Energy (mJ) | $0.020{\pm}0.007$ | 0.04±0.03 |

Table 13: Flat side and corner side data comparison: NiP/Ni20wt%W samples.



Figure 44: NiP/Ni20wt%W samples: protrusion height and bond depth comparisons for grit tested from corner side and flat side.



Figure 45: NiP/Ni20wt%W samples: Max Ft (Left) and energy (Right) comparisons for grit tested from corner side and flat side. Significant difference was found in energy between grits tested from flat side and corner side.

For NiP/Ni20wt%W samples, no significant difference was found between bond depth and protrusion height for different testing side conditions. Both Max Ft and energy of grits tested from the corner side showed higher values than those of grits tested from the flat side. No significant

difference was found in Max Ft. However, significant difference was found in energy, which indicates that the grits need more energy to be dislodged from the corner side.

Overall, the protrusion height and bond depth for grits that were tested from flat side and corner side for all the samples did not have any significant difference. When doing the average comparisons, the max Ft and energy for grits that were tested from corner were always higher than the grits that were tested from flat side. However, combined with the student t-test results, significant difference was only found in NiP-1 for max Ft effect and in NiP/Ni20wt%W samples for energy effect.

4.8 Comparisons between NiP coatings

When comparing the Max Ft and energy, it is noticed that NiP-2 samples, NiP/Ni10wt%W and NiP/Ni20wt%W samples exhibited a higher range of max Ft and energy values (**Fig. 31**) compared with NiP-1 sample. Thus, it is curious to study the difference between the grits with higher max Ft and energy and the grits with lower max Ft and energy. In this section, the two NiP samples would be discussed.

From the student t-test results (**Fig. 32**), it is found that the protrusion height for the 2 NiP samples are in the same range. Moreover, there was significant difference on bond depth between the NiP-1 and NiP-2 sample. All the grits that dislodged from NiP-2 samples were tested from corner side. As discussed in **Section 4.7**, the testing side might affect the bonding force between the grit and the bond. Thus, in order to keep the comparisons in consistent, for NiP-1, only the grits that were tested from corner side was considered for this comparison analysis. Thus, detailed comparisons between the 2 Ni-P samples were showed in the following.

| Parameter | NiP-1 | NiP-2 |
|------------------------|-----------------|-----------------|
| Protrusion height (µm) | 122±24 | 117±30 |
| Bond depth (µm) | 28±8 | 38±13 |
| Max Ft (N) | $0.92{\pm}0.72$ | $1.93{\pm}1.56$ |
| Energy (mJ) | $0.02{\pm}0.02$ | $0.05{\pm}0.05$ |

Table 14: NiP-1 and NiP-2 sample comparisons. All the grits were tested from corner side.



Figure 46: Bond depth and protrusion height comparisons between NiP-1 and NiP-2. All the grits were dislodged from the corner side. The bond depths between NiP-1 and NiP-2 have significant difference. No significant difference was found in protrusion height.



Figure 47: Max Ft (Left) and energy (Right) comparisons between NiP-1 and NiP-2 samples. All the grits were dislodged from the corner side. The asterisk indicates the significant difference between NiP-1 and NiP-2 on both Max Ft and energy.

From student t-test, there are no significant difference between the protrusion height of the 2 NiP samples. The bond depth of NiP-2 is significantly higher than that of the NiP-1. Both max Ft and energy of NiP-2 showed significantly higher value than NiP-1. Thus, it is suggested that the bond depth have influence on the energy and max. Ft. Moreover, compared with NiP-1, NiP-2 showed wider ranges in max. Ft. The wider range of max. Ft was then divided into 2 different ranges (**Fig. 48**): the higher max Ft range ranged from 2.5N to 5N and the lower max Ft range ranged from 0 to 2.5 N. The max Ft range of NiP-1 lied in the same range as the lower max Ft range of NiP-2. Thus, the 2 max Ft ranges of NiP-2 were used to make comparisons with NiP-1.



Figure 48: NiP-1 and NiP-2 comparison: max Ft versus protrusion height. All the grits were tested from corner side. A higher max Ft range and lower max Ft range fond in NiP-2 was marked in red.

Table 15: NiP-1 and 2 ranges of NiP-2 sample comparisons. All the grits were tested from corner side.

| Parameter | NiP-1 | NiP-2 | NiP-2 |
|------------------------|-----------------|-----------------|----------------|
| | | (Lower range) | (Higher range) |
| Protrusion height (µm) | 122±24 | 119±34 | 107±23 |
| Bond depth (µm) | 28±8 | 35±13 | 43±12 |
| Max Ft (N) | $0.92{\pm}0.72$ | $1.00{\pm}0.60$ | 4.0±0.8 |
| Energy (mJ) | $0.02{\pm}0.02$ | 0.02±0.01 | 0.13±0.03 |

The NiP-2 sample in higher range showed a much higher value in both energy and max Ft when comparing with NiP-1 and NiP-2 in the lower range. By doing the mean comparisons, the NiP-2 results in the lower range were similar to the results of NiP-1. Thus, statistical analysis was done to see any significant difference among the three situations.



Figure 49: Bond depth (Left) and protrusion height (Right) comparisons between NiP-1, NiP-2 grits with lower Max Ft range and NiP-2 grits with higher Max Ft range. The asterisk indicates the significant difference between NiP-1 and NiP-2 in higher Max Ft range on bond depth.

The protrusion height of the dislodged grits on the 2 NiP samples were in the same range. The bond depth of NiP-2 sample in higher Max Ft range (43 ± 12 um) is higher than the bond depth of the grits in lower Max Ft range (35 ± 13 um). No significant difference was found on the bond depth between the two ranges. However, there is significant difference in the 2 ranges of NiP-2 sample when comparing the max Ft and energy. Moreover, the bond depth of grits of NiP-2 sample (43 ± 12 um) in higher Max Ft range was significantly higher than that of NiP-1 (28 ± 8 um). The Max Ft and energy of the NiP-1 sample in higher Max Ft range were significantly higher than those of the NiP-1 sample. As the protrusion height and the testing side were controlled, the results here indicate that the bond depth of the grit has a major influence on the force and energy.

4.9 Post-analysis of the grits

In order to better understand the failure meachnism, SEM images of the dislodged grits were studied to see if there are any brittle or plastic evidence. In this section, grit cavities after the test for NiP-2 and NiP/Ni20wt%W samples were presented. The grits on NiP-2 and NiP/Ni20wt%W coatings have similar protrusion height and bond depth ranges. Since the grits for all types of coatings were found to have wide ranges of Max Ft values, the cavities of the grits with higher max Ft values ranged from 2.5 to 5 N and lower max Ft ranged from 0 - 2.5 N were all presented.

In **Fig. 50**, few examples of SEM images of the grit cavities after the tests for NiP-2 sample were shown. The coating surface around the cavity showed no evidence of any plastic deformation. Grits embedded inside the NiP coatings seem to be dislodged easily from the matrix without causing any damage to the surrounding. Previous studies showed that any defect near the bond level is caused by the sudden change of the stress at or within the bond level.¹ Thus, this might be an indication that the change in stress on the NiP coating around the grit is not high enough to cause deformation after dislogement.

In **Fig. 51**, few examples of SEM images of the grit cavities after the tests for NiP/Ni20wt%W were shown. From the SEM images, evdiences of plastic deformation were observed on the matrix around the cavities for the grits with both higher and lower max. Ft. It is assumed that the dislodgement of the grit might cause a dramatic change in stress on the coating, which further leads to the plastic deformation on the bond coat.



Figure 50: SEM images of some grits tested from corner side on NiP-2 sample: (a) Grits with higher max Ft value and (b) grits with lower max Ft values.



Figure 51: SEM images of some grits tested from corner side on NiP/Ni20wt%W sample: (a) grits with higher max Ft values and (b) grit with lower max Ft values

Overall, the SEM images of NiP-2 and NiP/Ni20wt%W samples were presented to see any evidence of the brittle behavior and the plastic behavior. From the SEM images, no evidence of any deformation was observed for grits on NiP samples. For NiP/Ni20wt%W samples, evidences of plastic deformation were observed on the grits within both max Ft ranges. It is assumed that NiP/Ni20wt%W coating had a dramatic stress change after the grit dislogement so that defect was observed near the grit cavity. Looking back to the statistical data in Table 4, the dislodged ratio of NiP-2 and NiP/Ni20wt%W samples were similar to each other. Thus, it is suggested that, compared with NiP-2, grits have higher adhesion force with NiP/Ni20wt%W coating. In addition, it is observed that grits tend to be more easily fractured on NiP/Ni20wt%W coating. This observation is in agreement with previous reported studies.¹ Ding et al.¹ did a comprehensive review on the monolayer cBN grinding wheel. It is reported that grits breakage/fracture occured more easily on the materials that would have defect during grinding. The grit breakage can either lead to new cutting sides or to total grit break out. Li et al.²⁰ once reported cBN-bond couple failure. Bond erosion, one of the failure behaviors, was observed after grinding. It was pointed out that grain would become unstable if the bond coat is removed so that grit displacement would occur inside the bonding material. As a consequence, the grit might be broke out. The grits on all tested samples were in low population. In the real applications, the population of superabrasives on the bond coat is much higher so that one grit failure behavior might influence the other neighbouring grits. Yu et al.¹⁵ demonstrated an evolution of the damage process during grinding. Their study also showed that only 40% or less grits participated in the grinding process. It is suggested that once the grit is pulled out under an excessive load, the local load would be redistributed to the surroundings. Thus, other grits within the load redistribution range would be affected. Grit fracture and attritious wear would then occur continuously. As the wear and damage zone increase, more

grits would engage into grinding process. From currrent observation in this study, with similar grit bond depth and protrusion height, the NiP/Ni20wt%W-cBN coating seemed to have a better grinding performance compared with NiP-cBN coating, by potentially providing more new cutting sides and more grit engagement during the grinding process.

4.10 Summary and future work

Three major type of bonding materials were tested using the single particle tests: NiP, NiP/Ni10wt%W and NiP/Ni20wt%W. For NiP samples, 2 samples were tested and analyzed separately due to the significant difference of their bond depths. Two NiP were name as NiP-1 and NiP-2, respectively.

The grit size and protrusion height for the tested grits on all the samples did not have any significant difference. Grits on NiP-1 coating have a lower bond depth compared with grits on NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W coatings. Lower energy and max Ft were observed on the NiP-1 coating, which indicate that the bond depth could have influence on the energy and max Ft. Thus, normalization of energy and max Ft to bond depth was done in order to see whether the material played any role. However, no significant difference was found among all the samples, which further showed that the material of the bond coating does not affect the energy and max. Ft.

Due to the irregular shapes of cBN, it is inevitable that tests were performed either on the flat side of the grit or the corner side of the grit. Thus, the dislodged grits with different tested side were studied. The protrusion height and bond depth for grits that were tested from flat side and corner side for all the samples did not have any significant difference. When doing the average comparisons, the max Ft and energy for grits that were tested from corner were always higher than the grits that were tested from flat side. However, combined with the statistical results, significant difference was only found in NiP-1 for max Ft effect and in NiP/Ni20wt%W for energy effect.

For NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples, higher ranges of max Ft and energy results were observed compared with NiP-1 sample. Grits of NiP-2 sample were divided into 2 groups with a higher max Ft range and a lower max Ft range. The max. Ft range of grits on NiP-1 is similar to the lower max. Ft range of NiP-2. Thus, the two NiP samples were compared in terms of bond depth, protrusion height, max. Ft and E. The comparisons showed that grits with higher bond depth needed more energy and force to be dislodged.

In addition, in order to better understand the failure meachnism, SEM images of the dislodged grits of NiP-2 and NiP/Ni20wt%W samples were studied to see if there are any brittle or plastic evidence. No evidence of any deformation was observed for grits on NiP sample. But for NiP/Ni20wt%W samples, evidence of plastic deformation was observed. It is suggested that the NiP/Ni20wt%W coating has a dramatic stress change after the grit dislodgement so that defect occurred. Moreover, based on previous studies, the NiP/Ni20wt%W coating is more potential than NiP coating to have a better grinding performance.

In the future, a FEM model could be developed to simulate the single grit-bond interactions during grinding. However, the single grit test only probe grit adhesion for a first interaction. For long term interactions of the grit with a workpiece, the mechanical property differences of the bond coat may provide some improvements. In the real applications, the wheel would continuously grind the

workpiece, which would eventually lead to the fatigue failure of the bond coating. Thus, it is also worth to study the mechanical properties of the bond coating.

Chapter 5: Experimental results and discussion of hardness, reduced modulus and strain rate sensitivity tests

The single grit test only probe grit adhesion for a first interaction. For long term interactions of the grit with a workpiece, the mechanical property differences of the bond coat may provide some improvements. In the real applications, the wheel would continuously grind the workpiece, which would eventually lead to the fatigue failure of the bond coating. Thus, it is worth to study the mechanical properties of the bond coatings, such as hardness, reduced modulus, creep and strain rate sensitivity.

In this chapter, hardness, reduced modulus and strain rate sensitivity (SRS) of Ni-P, Ni10wt%W and Ni20wt%W coatings will be presented and discussed. Two different nanoindentation testing techniques were used: constant strain rate (CSR) method and constant load (CL) method. Results obtained from the two methods were presented separately. SRS result comparison among all the coating materials obtained by different methods was discussed. Different influencing factors on the SRS results would also be discussed.

5.1 Microstructure

The surface morphologies of Ni-P, Ni10wt%W and Ni20wt%W were presented in **Fig. 52**. The measured roughness for all the coatings were summarized in **Fig. 53**. The Ni-P coating showed a much flatter and more uniform surface compared with Ni-W coatings, which further indicate that the roughness of Ni-P coating is lower than that of Ni-W coatings. The measured roughness also showed an agreement. Both Ni-W coatings has nodular morphologies. Between the 2 Ni-W

coatings, Ni10wt%W coating has larger agglomerates, which would further lead to a higher roughness.



Figure 52: SEM images of the surface morphology of Ni-P, Ni10wt%W and Ni20wt%W coatings.



Figure 53: Roughness of Ni-P, Ni10wt%W and Ni20wt%W coatings.

Grain sizes of different coatings measured from X-ray diffraction (XRD) were displayed in Fig. 55. The typical corresponding XRD patterns for Ni-P and Ni-W coatings were presented in Fig. 54. The Ni-P coating has a broad peak at plane (111). The Ni-W coating shows peaks at planes (111), (200), (220), (311) and (222). The grain size of each coating was calculated using the

Scherrer's equation. The grain sizes of Ni-P, Ni10wt%W and Ni20wt%W coatings were 1.8 nm, 9.2 nm and 8.6 nm, respectively. Compared with the 2 Ni-W coatings, the Ni-P coating has a much smaller grain size. For the two Ni-W coatings, sample with higher W content has smaller grain size.



Figure 54: Diffraction pattern of Ni-P (left) and Ni-W (right) samples.



Figure 55: Grain size of Ni-P, Ni10wt%W and Ni20wt%W

5.2 Hardness and reduced modulus

The hardness and reduced modulus for Ni-P, Ni10wt%W and Ni20wt%W coatings were presented in **Fig. 56**. All the reported hardness and reduced modulus values were tested on the cross sections of each sample. The hardness and reduced modulus of Ni-P is 7.1±0.3 GPa and 155±8 GPa, respectively. For Ni10wt%W, the hardness and reduced modulus are 7.4±0.2 GPa and 202±6 GPa, respectively. For Ni20wt%W, the hardness and reduced modulus are 8.3±0.3 GPa, and 192±21 GPa, respectively. Both Ni-W samples have higher hardness and reduced modulus values than the Ni-P sample. For the two Ni-W samples, sample with higher W content have higher hardness value and lower reduced modulus value.



Figure 56: Hardness (Left) and reduced modulus (Right) of Ni-P, Ni10wt%W and Ni20wt%W samples.

5.3 Strain rate sensitivity (SRS)

Two different nanoindentation testing techniques were used: constant strain rate method and constant load method. The SRS testing was performed directly on the sample surface. The SRS results obtained using each technique will be presented separately in the following.

5.3.1 Constant strain rate (CSR) method results

In this method, different loading rate (\dot{P}) to load (P) ratios were used: 0.02, 0.03, 0.05, 0.1, 0.2, 0.3, 0.5, 1, 2, 3 s⁻¹. Lucas and Oliver⁴⁸ showed that the indentation strain rate could be controlled by the loading rate to load ratio. In this study, the strain rate at each \dot{P}/P ratio was obtained from the time derivative of the exponential fit of the displacement-time loading curve. Thus, the experimental strain rate at each \dot{P}/P ratio obtained from the loading segment of the displacement-time curve was presented in **Table 16**.

| \dot{P}/P ratio (s ⁻¹) | Strain rate (s ⁻¹) |
|--------------------------------------|--------------------------------|
| 0.02 | 0.012 |
| 0.03 | 0.019 |
| 0.05 | 0.030 |
| 0.1 | 0.060 |
| 0.2 | 0.12 |
| 0.3 | 0.19 |
| 0.5 | 0.32 |
| 1 | 0.64 |
| 2 | 1.3 |
| 3 | 2.1 |

Table 16: Experimental strain rate at each \dot{P}/P ratio

Some experimental load-displacement curves for CSR method on NiP, Ni10wt%W and Ni20wt%W coatings at different strain rate were presented in **Fig. 57**. The instantaneous total depth at the peak load was used to calculate the hardness. Thus, at each \dot{P}/P ratio, a hardness value was reported. From **Fig. 57**, it can be seen that the instantaneous total depth at peak load for NiP is higher than that for Ni-W coatings. For the two Ni-W coatings, the instantaneous total depth of Ni10wt%W coating was lower than that of Ni20wt%W coating. Thus, compared with the Ni-W samples, the higher instantaneous total depth of NiP sample at various strain rates would result

into lower hardness at peak load. Between the two Ni-W samples, the Ni20wt%W sample has higher hardness at peak load than Ni10wt%W at different strain rates.



Figure 57: Load-displacement curves for CSR method on NiP, Ni10wt%W and Ni20wt%W at different strain rates (SR = 0.012, 0.06, 0.32 and 0.64 s⁻¹). The load-displacement curves at the other strain rates were presented in **Appendix**.

The double logarithmic plot of hardness-strain rate for each sample was then plotted in Fig. 58. The hardness comparisons at peak load among all the samples at each strain rate can also be seen in Fig. 58. The data with the strain rate ranged from 0.012 to 0.19 s⁻¹ were used to calculate the strain rate sensitivity, *m*. Ni-P coating has a higher *m* value of 0.063 ± 0.0055 than the Ni-W

coatings. The two Ni-W coatings showed similar *m* values, which was 0.031 ± 0.0042 for Ni10wt%W coating and 0.031 ± 0.0015 for Ni20wt%W coating. Guo *et al.*⁵⁹ reported the *m* value of magnetron sputtered nc Ni20wt%W obtained by CSR method, which was 0.043. This result is comparable with the *m* value of Ni20wt%W in this study. The *m* values of Ni-P coatings and other Ni-W coatings obtained by nanoindentation methods were barely reported.



Figure 58: CSR results of Ni-P, Ni10wt%W and Ni20wt%W samples on a log-log Hardness to strain rate plot.

The gradient of the trend became negative when the strain rate was above 0.19 s⁻¹ and 0.32 s⁻¹ for Ni-W coatings and the Ni-P coating, respectively. One previous study by Muller⁶⁰ showed that nickel is very sensitive to high strain rate which further implied that the deformation mechanism for nickel at high strain rate was different from that at static strain rate. There are also some previous studies which reported the influence of the strain rate on the microstructures. ⁶¹⁻⁶³ For another FCC metal, Cu, Andrade *et al.*⁶¹ reported on microstructure changes for samples deformed

at high strain rate. Their study proposed that the dynamic recrystallization was responsible for the microstructure alternation. Molinari and Ravichandran⁶² also did a microstructural evolution study for metals which deformed at high strain rate. Different microstructural lengths (δ) at each deformation stage were discussed. At the beginning deformation stage, δ reduced accompanied with strain hardening. As the strain became high, the reduction of δ is inhibited where strain softening occurs. It is mentioned that cell size is associated with strain rate and temperature. It is concluded that the cell size would decrease with increasing strain rate. Moreover, Dalla Torre *et al.*⁶³ studied the microstructure change on nc electrodeposited Ni with a range of strain rate. When the strain rate was highest, shear band with local grain growth was observed on the surface. Thus, the transition observed in **Fig. 58** is suggested to be caused by the microstructural change with increasing strain rate. A transition from strain hardening to strain softening might occur.

5.3.2 Constant load (CL) method results

Strain rate sensitivity study using constant load methods on different materials have also been widely used.^{50, 52, 64-67} The constant load method has a rapid loading rate and a long holding time. The experimental load-displacement curves for CL method on NiP, Ni10wt%W and Ni20wt%W samples were shown in **Fig. 59**. The maximum depth at the peak load of NiP was higher than that of the Ni-W samples, which further indicated its lower hardness at the peak load. For the CL method, SRS of the material was analyzed from the holding segment of the displacement-time curve. From previous studies, during the holding segment, the displacement-time curves of some materials were observed to reveal 2 stages. In this study, 2 stages on the displacement-time curves were observed for all the tested samples. One example of the displacement-time curve and strain rate-time curve for Ni-P was presented in **Fig. 60**. The first stage and the second stage were marked.

Stage I is known as the transient creep, where the penetration depth would increase rapidly.^{50, 67} In this stage, the strain rate and the stress were maximum.⁵⁴ Stage II is called steady-state creep, where the penetration depth would gradually increase linearly and the strain rate tends to be saturated. Thus, two *m* values would be obtained. The *m* value for each stage is the gradient of the logarithmic hardness-strain rate (**Fig. 61**) plot for each stage.



Figure 59: Load-displacement plot for CL testing on NiP, Ni10wt%W and Ni20wt%W coatings.



Figure 60: An example of displacement-time and strain rate-time curve for Ni-P at 10 mN. Stage I and II were marked.



Figure 61: An example of a nature logarithmic plot for Ni-P at 10 mN. Stage I and II were marked for both plots.

Since each stage has its own *m* value, the *m* value for Stage I will be referred as m_1 . And for Stage II, the value would be named as m_2 . For Ni-P, m_1 and m_2 were 0.0036 ± 0.0011 and 0.018 ± 0.0010 , respectively. For Ni10wt%W, the *m* value was 0.0070 ± 0.0012 for Stage I and 0.017 ± 0.0021 for Stage II. Moreover, m_1 and m_2 of Ni20wt%W were 0.0058 ± 0.0010 and 0.017 ± 0.0021 , respectively. For Stage I, both NiW coatings showed higher *m* values than NiP coating. A different trend was observed for Stage II, where the *m* value of NiP coating was slightly higher than the two NiW coatings. And the two NiW coatings has the same m_2 values. Since the Stage I is less stable, its result is normally not considered. However, some studies showed that *m* value obtained from Stage I is closer to the *m* value obtained from CSR method and also other values reported in the literature.⁵⁰ In the following section, the SRS results obtained from CSR and CL methods would be discussed and compared.

5.3.3 SRS result comparisons

The comparison of the *m* values of Ni-P, Ni10wt%W and Ni20wt%W coatings obtained from CSR and CL method was shown in **Fig. 62**. The *m* values for all the coatings obtained from CSR method were higher than those obtained from CL method. The *m* value of Ni-P obtained from CL Stage II was only slightly higher than those of the two Ni-W coatings. The CSR values of Ni-P was much higher than those of the 2 Ni-W samples.



Figure 62: Comparison of the SRS results obtained by different testing techniques for Ni-P, Ni10wt%W and Ni20wt%W coatings

Only few papers reported the direct comparison between the *m* values obtained from different nanoindentation methods using exactly same specimen. Peykov *et al.*⁵⁰ compared the SRS results obtained from CSR and CL method. The tested materials were Zn, Al, Cu, Ti and fused quartz (FQ). They found out that the results obtained from CSR methods were more comparable with those obtained from CL Stage I. On the contrary, the CL Stage II values were not really correlate with the CSR values. It was also observed that the *m* values from CSR methods were generally higher than those from CL methods. In their study, Stage I was redefined as a stage where the displacement rate was much higher than the drift rate. Moreover, significant drift error was found

in Stage II. Thus, a shorter loading period was suggested to reduce the drift errors. Another comparative study was performed by Shen *et al* to compare the CSR values and the CL values.⁵⁴ The sample that they used were single crystal Al and polycrystalline Sn. Similar to the trend found in Peykov's paper, the CSR values were higher than both CL stage values. This study showed that the CSR value is more reliable than CL values because a steady state is more easily reached in a CSR test. As mentioned before, the experimental CSR values of Ni20wt%W sample was comparable with the values found in the literature. From the result comparison, CL values in both stages were not really correlate with CSR values. Thus, it is suggested that the lower CL values could be resulted from the absence of the steady state.

5.3.4 Influencing factors of SRS results

In **Fig. 62**, it can be seen that the *m* value of Ni-P was much higher than the *m* values of the Ni-W samples when looking into CSR results. The difference could be resulted from different grain sizes. In recent years, SRS studies on nanocrystalline Ni and Ni-based alloys have been studied using different techniques. ^{35, 59, 68-73} SRS values obtained by nanoindentation techniques from previous literature were listed in **Table 17**, along with the sample grain sizes.

The *m* value of the dual-phase (DP) Ni20wt%W exhibited a higher *m* value⁵⁹, 0.086, compared with the other pure nc Ni and magnetron sputtered nc Ni20wt%W sample with similar grain size. In general, higher grain boundary (GB) activity, such as GB sliding and GB diffusion, would cause higher *m* value. A schematic illustration of the GB for nanocrystalline, amorphous and dual-phase NiW plotted by Guo *et al.* was demonstrated in **Fig. 63**.⁵⁹ The microstructure of the electro-deposited DP NiW was special as most of the nanocrystalline was distributed by itself inside the

amorphous matrix. Hence, instead of GB, amorphous/crystalline interface (ACI) was observed. There was only limited number of GB existed inside the sample. Due to the lack of lattice restriction, the ACI would more easily result into dislocation by nucleation and annihilation. The nucleated dislocation travelled in the opposite direction to the annihilation process through the grain, which further cause a higher m value. In this study, all the samples were nanocrystalline. Thus, a smaller m value of electroplated Ni20wt%W than that of the DP Ni20wt%W sample is expected.

| | Testing | Material | Mean grain | m |
|----------------------------------|-------------|--------------|------------|-------|
| | technique | | size (nm) | |
| Guo et al. ⁵⁹ | CSR | nc Ni20wt%W | 12 | 0.043 |
| Gu et al. ⁶⁸ | CSR | nc Ni | 20 | 0.033 |
| Guo <i>et al</i> . ⁵⁹ | CSR | dual-phase | 24 | 0.086 |
| | | Ni20wt%W | | |
| Maier et al.35 | CSR | nc Ni | 25 | 0.052 |
| Maier et al.35 | strain rate | nc Ni | 25 | 0.019 |
| | jump test | | | |
| Shen et al. ⁶⁹ | CRL | nc Ni | 25 | 0.026 |
| Hu <i>et al</i> . ⁷¹ | CL | nc Ni20wt%Fe | 26 | 0.018 |
| Hu <i>et al</i> . ⁷¹ | CL | nc Ni | 30 | 0.035 |
| Liu et al. ⁷⁰ | CRL | nc Ni | 55 | 0.016 |

Table 17: Strain rate sensitivity of nc Ni and Ni-based alloys obtained by nanoindentation techniques with different grain sizes



Figure 63: A schematic illustration of the relationship between hardness and *m* for dual-phased, nanocrystalline and amorphous NiW. Dislocation nucleation and annihilation, amorphous/crystalline interfaces (ACIs) and grain boundary (GB) related mechanism were presented.⁵⁹ In our study, Ni-P and Ni-W samples were all nanocrystalline samples.

Hu *et al.* studied the creep behaviors between nc Cu, nc Ni and nc Ni20wt%Fe.⁷¹ Comparing the *m* value of nc Ni20wt%Fe with those of pure nc Ni in the other literatures with similar grain size, it was found out that the nc Ni20wt%Fe had a lower SRS value. This study demonstrated that the loading strain rate and stacking fault energy (SFE) would influence the creep behaviors. During the high strain loading process, grain boundary dislocation was activated and could be stored rapidly. New nucleated dislocation would be further generated during the holding process. When the strain loading rate is low, GB-mediated deformation was suggested to be the major reason of the creep behavior. Thus, the *m* values of nc Ni and Ni20wt%Fe was lower due to their lower GB sliding at low loading strain rate and their lower dislocation storage rate.

For the m values in Table 17, as the nanoindentation techniques varied, the relation between the m values and the grain size was not clear. However, some other literature reviews observed a clear

increasing trend in *m* values as the grain size decreases for nc Ni.^{68, 70} Chen *et al.*⁷⁴ did the grain size effect study on SRS on a nc Cu-Ni-P alloy using nanoindentation technique. Their observations showed that the grain size of the sample would play an important role on its plastic deformation mechanism. As the grain size ranged from 33 nm to 7 nm, the *m* values changed from 0.18 to 0.33. Their study also showed that the addition of the elements would result into a lower *m* value when comparing with the pure nc Cu under similar grain size conditions. They referred Fäber's study⁷⁵ to suggest that the P segregation at grain boundaries (GB) stabilize the GB activities so that adding P could provide more resistance of the plastic deformation. The amount of P content would decide whether the material is amorphous (16-22 wt%), nanocrystalline (<10 wt%) or mixed in both states (10-15 wt%).75,76 The grain size for nc NiP ranged from 2 to 9 nm as the P content changed from 13 to 3 wt%. In Fäber's study⁷⁵, electroless Ni3.6wt%P was studied using tomographic atom probe (TAP). Their study showed that the P element was distributed inhomogenously on the sample. Most of the P content was segregated at the grain boundaries while inside the grains, there was almost no P content. The P segregation was observed in the NiP samples with heat treatment as well, which further proved that the addition of P could improve the thermal stability of the sample. The *m* values of nc Ni in **Table 17** varied a lot because different techniques and grain sizes were used. Thus, the m values^{35, 68} of nc Ni obtained from CSR technique in Table 17 were used for the following comparisons. Both selected nc Ni from Table 17 have larger grain sizes compared with the two Ni-W samples (Ni10wt%W and Ni20wt%W) in this study. From previous observations, materials with smaller grain size tend to have higher m values. However, instead of having higher m values, it is found that the m values of the two Ni-W samples were lower than the reported *m* values of nc Ni. Based on the above discussions, it is assumed that the addition of W could lead to W segregation near GB to further stabilize the GB so

that the *m* values were lower. The *m* value of the Ni-P sample was much higher than most of the reported values. Although the addition of P element might stabilize the GB, its very small grain size could alternate the creep behavior, which will be discussed in the following.

A lot of models to calculate strain rate sensitivity were proposed.^{34, 68, 69} A classical model was proposed by Wei *et al.*³⁴ The SRS can be calculated from the following equation:

$$m = \frac{kT}{\tau v_*} \tag{5.1}$$

where k is the Boltzmann constant, T is the absolute temperature and v^* is the activation volume. Thus, it is commonly known that lower activation volume would result into higher m value. This model is proposed for the materials with grain sizes ranged from 250-300 nm. It is known that the grain size of the materials could influence the mechanism of deformation, especially when the grain size was fewer than ten nanometers. Dislocation near the GB is the major reason for the dislocation. Studies showed that the classical dislocation theories, such as Hall-Petch relationship, work for grain sizes down to 5-20 nm.⁷⁷ Based on different literature reviews, Morris et al.⁷⁷ summarized that for Ni-based alloys, the classical dislocation theory is not obeyed when the grain size is below 15nm, 20-40 nm and 120 nm, respectively. As the grain becomes much finer, the deformation mechanisms may change completely. One previous study by Wang et al.78 showed that the deformation mechanism changed when the grain size of nc Ni was below 10-20 nm. Softening effect was found for the grain size from 10 to 6 nm. And the creep rate was much higher than the expectation when the sample grain size was 6 nm. Thus, it was suggested that the diffusion creep mechanism is more reasonable for smaller grain size to explain the deformation mechanism. Gu et al.⁶⁸ proposed a new model to study the relationship between SRS and small grain sizes. The equation⁶⁸ for nc materials is presented below:

$$m = \frac{\sqrt{3}kT}{0.36Gb^3} \left[\ln\left(\frac{V}{b^3}\right) - 1.65 \right]^{-1}$$
(5.2)

where G is the shear modulus, b is the Burgers vector, V is the activation volume. The activation volume V of a material can usually reveal its deformation mechanism. It can be further defined as:

$$V = Lb^2$$
(5.3)
$$L = cd^{\frac{1}{2}}$$
(5.4)

where L is the dislocation line length, c is the a constant and d is the grain size. Thus, as the grain size decrease, the dislocation line length would decrease so that the nucleation behavior would be restrained, which would result into an increased m value. This assumption is suggested for the materials with grain size larger than 6 nm. The grain sizes of Ni10wt%W and Ni20wt%W were similar to each other. The experimental CSR values were also similar to each other. Thus, the W content seems to have no influence on the creep behavior. The CSR value of Ni-P sample was much higher than those of Ni-W samples. Grain size is suggested to be the major factor. Based on the proposed models, the much smaller grain size of Ni-P would have much smaller dislocation line length. Hence, it would possess a much lower activation volume compared to the 2 Ni-W samples. Thus, a higher m value would be expected for the Ni-P sample.

5.4 Summary

The hardness and reduced modulus of NiP, Ni10wt%W and Ni20wt%W coatings were presented. In general, Ni-W coatings have higher hardness and reduced modulus than Ni-P coating. Between the Ni-W coatings, samples with higher W content has higher hardness and lower grain size. A comparative SRS study on NiP, Ni10wt%W and Ni20wt%W coatings using CSR and CL method were also presented. CSR values were all higher than CL values. The CL values were found to not correlate with the CSR values. This could cause by the absence of the steady state in the CL method. In addition, the gradient of the double logarithmic CSR hardness-strain rate plot became negative when the strain rate reached to a critical point, which is 0.19 s^{-1} for Ni-W coatings and 0.32 s^{-1} for Ni-P coating. A change in microstructure would be the contribution. In the CSR method, the lower *m* values of Ni-W coatings compared with those of nc Ni coatings found in the literature could be explained by the W segregation near the grain boundaries. Addition of W element would help to stabilize the grain boundaries. In this study, the change in W content does not influence the SRS. The high *m* values of Ni-P coating could be explained by the small grain size of Ni-P, where dislocation line length was lower so that there would be less nucleation behaviors.

Appendix



Figure 64: Load-displacement curves for CSR method on NiP, Ni10wt%W and Ni20wt%W at strain rates of 0.019 s⁻¹.



Figure 65: Load-displacement curves for CSR method on NiP, Ni10wt%W and Ni20wt%W at strain rates of 0.030 s⁻¹.



Figure 66: Load-displacement curves for CSR method on NiP, Ni10wt%W and Ni20wt%W at strain rates of 0.12 s⁻¹.



Figure 67: Load-displacement curves for CSR method on NiP, Ni10wt%W and Ni20wt%W at strain rates of 0.19 s⁻¹.



Figure 68: Load-displacement curves for CSR method on NiP, Ni10wt%W and Ni20wt%W at strain rates of 1.3 s⁻¹.



Figure 69: Load-displacement curves for CSR method on NiP, Ni10wt%W and Ni20wt%W at strain rates of 2.1 s⁻¹.
Chapter 6: Conclusion

6.1 Conclusions

The two major objectives in this thesis are to measure the bonding force between the grit and the bond coat and to determine the hardness, reduced modulus and time-dependent strain rate sensitivity of Ni-based cBN composite coatings.

6.1.1 Grit adhesion test

For the grit adhesion study, two NiP-cBN, one NiP/Ni10wt%W-cBN and one NiP/Ni20wt%W-cBN coatings were studied. Samples were named as NiP-1, NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W, respectively in the following. The grit size and protrusion height for the tested grits on all the samples did not have any significant difference. Sample NiP-1 has grits with lower bond depth compared with NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W coatings. Lower energy and max Ft were observed for the grits on the NiP-1 coating, which further indicate that the bond depth could have influence on the energy and max Ft. Normalization of energy and max Ft to bond depth was done in order to see whether the material played any role. However, no significant difference was found among all the samples, which indicated that the material of the bond coating does not affect the energy and max. Ft.

Due to the irregular shapes of cBN, it is inevitable that tests were performed either on the flat side of the grit or the corner side of the grit. Thus, the dislodged grits with different tested side were studied. The protrusion height and bond depth for grits that were tested from flat side and corner side for all the samples did not have any significant difference. When doing the average comparisons, the max Ft and energy for grits that were tested from corner were always higher than the grits that were tested from flat side. However, combined with the statistical results, significant difference was only found in NiP-1 for max Ft effect and in NiP/Ni20wt%W for energy effect.

The NiP-1 sample showed a lower range of max. Ft and energy compared with NiP-2, NiP/Ni10wt%W and NiP/Ni20wt%W samples. Comparisons between the two Ni-P coatings were made. Grits of NiP-2 sample were divided into 2 groups with a higher max Ft range and a lower max Ft range. The max. Ft range of grits on NiP-1 is similar to the lower max. Ft range of NiP-2. Thus, the two NiP samples were compared in terms of bond depth, protrusion height, max. Ft and E. The comparisons showed that grits with higher bond depth needed more energy and force to be dislodged.

In addition, in order to better understand the failure meachnism, SEM images of the dislodged grits of NiP-2 and NiP/Ni20wt%W samples were studied to see if there are any brittle or plastic evidence. No evidence of any deformation was observed for grits on NiP sample. But for NiP/Ni20wt%W samples, evidence of plastic deformation was observed. It is suggested that the NiP/Ni20wt%W coating has a dramatic stress change after the grit dislodgement so that defect occurred. Moreover, based on previous studies, the NiP/Ni20wt%W coating seemed to have a better grinding performance compared with NiP-cBN coating, by potentially providing more new cutting sides and more grit engagement during the grinding process.

6.1.2 Hardness, reduced modulus and strain rate sensitivity

In this study, coatings without cBN grits were tested: Ni-P, Ni10wt%W and Ni20wt%W. The grain size measured from XRD was presented. The grain sizes of Ni-P, Ni10wt%W and Ni20wt%W coatings were 1.8 nm, 9.2 nm and 8.6 nm, respectively. The hardness of Ni-P, Ni10wt%W and Ni20wt%W coatings were 7.1±0.3, 7.4±0.2 and 8.3±0.3 GPa, respectively. Reduced modulus of each coating was 155±8, 202±6 and 192±21 GPa, respectively. In general, Ni-W coatings have higher hardness and reduced modulus than Ni-P coating. Between the Ni-W coatings, samples with higher W content has higher hardness and lower grain size.

A comparative SRS study on NiP, Ni10wt%W and Ni20wt%W coatings using CSR and CL method were presented. CSR values were all higher than CL values. The CL values were found to not correlate with the CSR values. This could cause by the absence of the steady state in the CL method. In addition, the gradient of the double logarithmic CSR hardness-strain rate plot became negative when the strain rate reached to a critical point, which is 0.19 s⁻¹ for Ni-W coatings and 0.32 s⁻¹ for Ni-P coating. The occurrence of strain softening might be the reason for the transition in the trend.

In the CSR method, the lower *m* values of Ni-W coatings compared with those of nc Ni coatings found in the literature could be explained by the W segregation near the grain boundaries. Addition of W element would help to stabilize the grain boundaries. In this study, the change in W content does not influence the SRS result. The high *m* values of Ni-P coating could be explained by the small grain size of Ni-P, where dislocation line length was lower so that there would be less nucleation behaviors.

6.2 Suggestions for future work

- A FEM model could be developed in the future to simulate the single grit-bond interactions during grinding. The change of stress exerted on the bond coat around the grit during grinding could also be investigated through the FEM model.
- 2. The single grit test only probe grit adhesion for a first interaction. For long term interactions of the grit with a workpiece, the mechanical property differences of the bond coat may provide some improvements. In the real applications, the wheel would continuously grind the workpiece, which would eventually lead to the fatigue failure of the bond coating. Thus, it is worth to study the other mechanical properties of the bond coat, such as fracture toughness, which can tell the ability of the bond coat to resist crack growth.
- 3. For the strain rate sensitivity study, TEM could be used to investigate the microstructural evolution for each coating in order to better understand the deformation mechanisms.

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