



The microstructure and mechanical properties of TiN–Ni nanocomposite thin films

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ABSTRACT

The effect of varying substrate bias and temperature on the microstructure and properties of TiN–Ni nanocomposite thin films, fabricated using a filtered cathodic vacuum arc, was investigated. Films deposited at room temperature with no applied bias had low surface roughness and exhibited fine microstructure. An increase in bias or temperature resulted in significant changes in microstructure, including an increase in the crystallinity of the Ni phase and an increase in TiN and Ni crystallite sizes. At a substrate bias of -500 V, separation of the TiN and Ni phases was observed resulting from the high-energy ion bombardment. The intrinsic stress and hardness of the films were found to decrease with increasing Ni crystallite size. The TiN–Ni nanocomposite films were found to exhibit lower hardness than a TiN film deposited using similar conditions and were more ductile due to the presence of metallic Ni. The film deposited at room temperature and low bias was found to be highly elastic, exhibit reasonable hardness (~ 18 GPa) and low intrinsic stress. This combination of properties has the potential for exploitation in a range of tribological applications.

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1. Introduction

Over the past 40 years, hard ceramic thin film coatings such as titanium nitride (TiN) have significantly improved the performance of industrial cutting tools [1]. The materials science underpinning these productivity gains has to-date focussed largely on improving the hardness of physically vapour deposited (PVD) coatings. An inevitable consequence of this trend to ever-harder, even super-hard coatings is that coatings have become increasingly brittle often containing high intrinsic stress [2,3]. Such factors can adversely affect tool life performance, particularly under high impact load conditions, such those encountered during milling. Recognition of such problems is evident in the development of multilayer coatings, in which an attempt is made to arrest crack propagation by introducing inter-phase boundaries [4–6]. Also of interest are hard nanocomposite coatings which resist cracking, have high elastic recovery and low intrinsic stress [7,8]. Recent work has shown that hard nanocomposite coatings with enhanced resistance to cracking are characterised by a ratio of hardness (H) to effective elastic modulus (E^*) of greater than 0.1 and an elastic recovery of greater than 60% [7].

One method of producing such nanocomposite films is to reactively vapour deposit both nitride and non-nitride forming elements [9–14]. One such non-nitride forming element is Ni, which has been used in the fabrication of nanocomposites [15–17]. For example, TiN–Ni nanocomposite thin films have been produced by several methods including

magnetron sputtering [18], dual ion beam assisted deposition [19] and cathodic arc [20,21]. The thin films exhibited a range of microstructures depending on the deposition conditions and promising mechanical properties, which have the potential for improved tribological performance in metal cutting applications. However, the above studies did not undertake detailed microstructural analysis using cross sectional transmission electron microscopy (TEM), which limits the fundamental understanding of structure, hence properties and application. This is critically important, since in the production of nanostructured films, by physical vapour deposition (PVD), holds the possibility of producing unique, non-equilibrium compositions and novel structures [22].

In this study, TiN–Ni nanocomposite thin films were fabricated using a filtered cathodic vacuum arc (FCVA) system fitted with a single cathode composed of 50:50 at.% Ti:Ni. The motivation was to produce coatings suitable for exploitation as layers in functionally graded coatings [23] where the property mismatch between substrate and hard coating is reduced by using one or more interlayers with intermediate or graded properties such as, ductility, residual stress and hardness.

FCVA is a vapour deposition method in which the average energy of the highly ionised depositing flux is controllable by varying the substrate bias voltage. This deposition energy is known to influence the type of microstructure produced [24], which in turn, determines the resulting physical properties. Previous work on TiN–Ni nanocomposite thin films using cathodic arc [20,21] employed a dual cathode system (one Ti and the other Ni doped with 10 at. % Cr) to produce thin film coatings and concentrated mainly on the effect of post deposition annealing on grain size. Here, the results of varying the substrate bias and deposition temperature on the microstructure and mechanical properties of TiN–Ni thin films, are presented. This is supported with detailed microstructural characterisation using a range of techniques

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Table 1
Summary of the deposition parameters and several characteristics of the films deposited.

Film type	Bias (V)	Temperature (°C)	Thickness (nm)	Deposition rate (nm/min)	Roughness (nm)
TiNi	Floating	Room temp.	300	34	0.9
TiN–Ni	Floating	Room temp.	200	100	0.9
TiN–Ni	–200	Room temp.	330	85	4.4
TiN–Ni	–500	Room temp.	70	14	4.3
TiN–Ni	Floating	250	240	80	3.3
TiN–Ni	Floating	500	150	50	8.9
TiN	Floating	Room temp.	270	50	0.9

including direct observation of the cross sectional microstructure of the thin films using transmission electron microscopy (TEM). Also, structure–property relationships were determined using nanoindentation.

2. Experimental

Films were prepared using an FCVA deposition system, which is described elsewhere [25], using a rotating cathode operating at an arc current of 100 A. The plasma emanating from the cathode was directed through a magnetic double bend filter, designed to minimise the deposition of macroparticles and neutral species. Films were deposited onto polished silicon wafer substrates that were cleaned with acetone and ethanol prior to deposition. Stationary substrates were either biased by mounting on a steel holder connected to a variable DC voltage supply (<1 kV) or heated by mounting on a heater stage with the temperature monitored using a thermocouple. The temperature to which the substrates are initially heated before deposition is referred to as the

deposition temperature. The system was pumped to a base pressure of less than 10^{-3} Pa before each deposition. A $\text{Ti}_{50}\text{Ni}_{50}$ at.% cathode was used and a series of thin films was produced using the conditions shown in Table 1. All films were prepared at an operating pressure of 0.2 Pa. TiN–Ni films were prepared in an N_2 processing gas. A TiNi film was also prepared at floating potential and room temperature in Ar processing gas. For comparison purposes, a TiN thin film was produced from a Ti cathode in N_2 atmosphere at the same arc current and operating pressure.

The intrinsic stress of the thin films was obtained using Stoney's equation [26] after measuring the radii of curvature of the substrates before and after deposition using a Tencor surface profiler. The thickness of the thin films was also determined using surface profilometry.

Scanning electron microscopy (SEM) and atomic force microscopy (AFM) were used to study the surface morphology of the thin film coatings. A FEI Nova Nano SEM with field emission gun source was used in high vacuum mode with a working distance of 5 mm and accelerating voltage of 15 kV were used to obtain the SEM micrographs of the thin film surfaces. Surface roughness was measured using a Veeco Dimension 3100 AFM operated in tapping mode. AFM scans were performed over several different sized areas ranging from $3 \times 3 \mu\text{m}$ square to $10 \times 10 \mu\text{m}$ square to obtain an average RMS roughness value for each sample.

The composition of the thin films, as a function of depth, was determined by x-ray photoelectron spectroscopy (XPS) depth profiling using a Thermo Scientific K-Alpha Spectrometer with an Al anode x-ray source. The XPS sputter depth profiles were performed using an Ar ion beam operating at 1–2 keV etching approximately a 1 mm^2 area and the x-ray spot size used for analysis was $200 \mu\text{m}$.

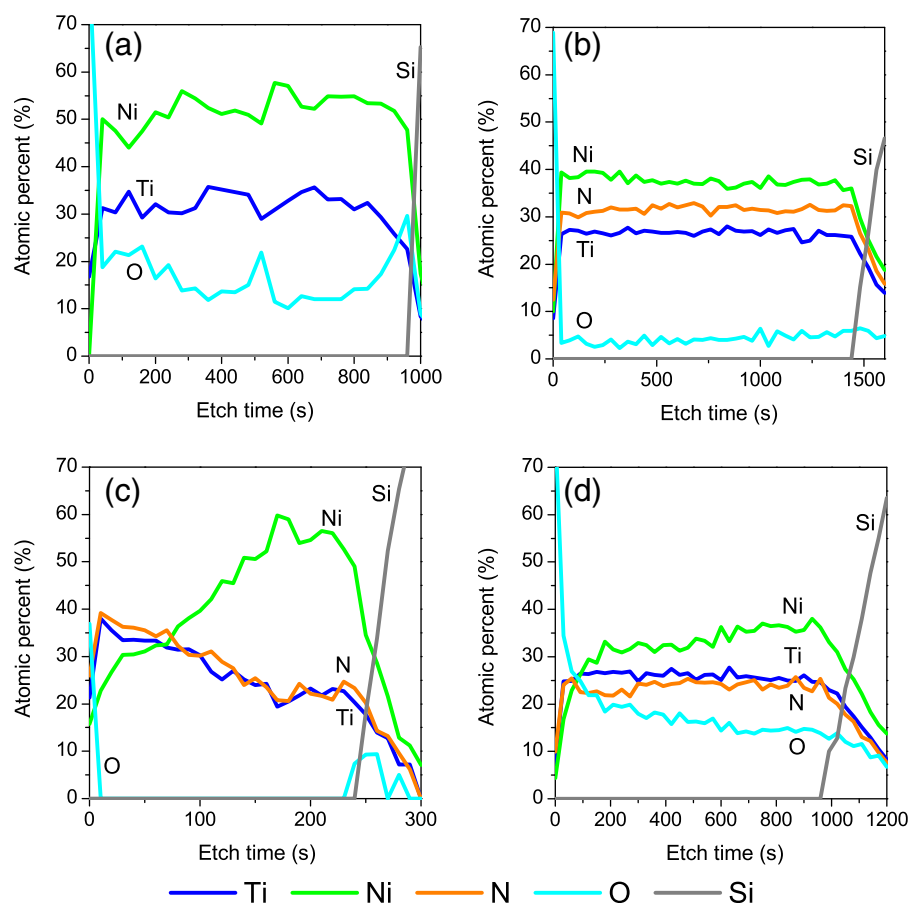


Fig. 1. XPS depth profiles of the films deposited at (a) floating potential-room temperature in Ar and the films prepared in N_2 at (b) floating potential-room temperature, (c) –500 V bias-room temperature and (d) floating potential-500 °C.

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