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# Tribological behavior of TiN and Ti (Si,C)N coatings on cold sprayed Ti substrates



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## ABSTRACT

The evolution of the cold spray technology in the aerospace part repair created a need to better understand the mechanical and tribological behavior of the protective coatings on the cold sprayed repair. For that purpose, a bilayer system consisting of titanium cold spray coating on a commercially available titanium substrate and TiN, TiSiN and TiSiCN HiPIMS protective coatings were produced. The mechanical and tribological behavior of the coatings was evaluated under reciprocating wear with a reciprocating tribometer equipped with an aluminum ball (4 mm in diameter) counterface. The wear tracks were analyzed with a light interferometer. A cross-sectional analysis of the wear track was performed with SEM following focused ion beam milling. Focused ion beam milling revealed the deformation induced damages to the coatings. Cold sprayed substrate porosity, grain refinement and hardness were shown to play a significant role on the wear characteristics of HiPIMS deposited TiN, TiSiN and TiSiCN coatings.

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## 1. Introduction

Repair engineers for the aerospace industry are increasingly challenged to develop new routes for repairing expensive components, such as compressor blade in turbine engines [1–5]. Laser remanufacturing process is one of the commonly used techniques for repairing blades [6,7]. In recent times cold spray technique gained importance in repairing engine components and to re-coat oxygen sensitive materials [8,9]. The prospect of repairing a component becomes even more challenging when the component has a coating that must be reapplied [10]. The coating reapplication process involves the removal of any remnant coating surrounding the damage that is followed by welding or thermal spray repair and coating reapplication [7,11,12]. A duplex structure is created of a repaired material with coating. Matching of the properties of the repaired material with the coatings is critical for the success of any of these types of repairs.

The cold spray is, currently, a candidate technology to repair the engine components due to the applicability of this process as a near net shape production technology [13–17]. The process consists of metallic powder deposition at supersonic velocities with preheated and pressurized nitrogen or helium gas passing through de Laval type nozzle [18, 19]. Upon impact with a substrate material, the powder undergoes plastic deformation and adiabatic shear, which contribute to the powder adhesion and metallic coating build-up. The low process temperature of the cold spray process does not significantly alter the chemistry and microstructure of the metals like titanium and, therefore, provides a great alternative to more traditional thermal spray techniques currently used. Cold spray deposited materials, typically, demonstrate high coating density, extensive grain refinement and an increase in the material hardness in addition to the residual compressive stresses. These aspects of the cold spray materials can be beneficial to the strain tolerance of protective coatings (like TiN, TiSiN and TiSiCN) during sliding wear applications. These sorts of coatings were studied extensively on traditional substrates (non-cold sprayed), including steel [20,21] and titanium [22–24].

TiN coatings are used in the aerospace industry as an erosion resistant coatings to protect compressor blades of the gas turbine engine, which are made of titanium alloys. The components of the engines operated in harsh environments are prone to erosion or surface damage. These coatings are also useful for their wear resistance in sliding wear applications.

Ionized physical vapor deposition (IPVD) techniques have gained pivotal importance in large scale industrial production of nitride, carbide and oxide based coatings. This is due to the fact that the IPVD techniques provide greater tools to control sputtered material flux, thereby reducing deposition temperature with increased atom mobility and chemical reactivity on a wide variety of component surfaces [25]. High power impulse magnetron (HiPIMS) sputtering is one of the classified pulsed IPVD technologies introduced in the last decade. HiPIMS

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<sup>&</sup>lt;sup>1</sup> Denotes equal contribution as first authors.

#### Table 1

Deposition conditions of HiPIMS TiN, TiSiN and TiSiCN coatings.

Deposition conditions		TiN	TiSiN	TiSiCN	Deposition conditions	TiN	TiSiN	TiSiCN
Pressure (mTorr)		5	5	5	Cathode voltage (V)	-630	-630	-630
Gas		Ar:N <sub>2</sub>	Ar:N <sub>2</sub>	Ar:N:CH4	Bias voltage (V) Current (A)	- 50 1.2	- 50 1.3	— 50 0.9
Flow ratio (sccm)		18:1	18:1	18:1:1	Frequency (Hz)	100	100	100
Applied power (W)	Ti Si	400 0	400 80	400 80	Dep. rate (nm/s)	400-500	700-800	700-800

sputtering technology has demonstrated greater flexibility to fabricate application tailored nanostructured thin films/coatings. Typical region of sputtering in HiPIMS falls within a short range of about 10–200 µs of the total pulse width of 20–40 ms [26,27]. These short impulses carrying high peak power density up to few kW/cm<sup>2</sup> are dissipated on the target material to generate large amount of ions in the plasma [28–30]. Report suggests that component surface facing the cumulative phased ion impact during HiPIMS process [31–34], undergoes continuous plasma chemical change at regular intervals of impulse and improves coating properties. Analyses of such observations have also shown accurate control of phase composition in the coating with preferred orientation of crystalline grains [35], tight atomic density micro/nanostructures and very importantly ability to grow coatings under low temperature [36].

The objective of the present work is to investigate the properties of various combinations of HiPIMS deposited TiN, TiSiN and TiSiCN protective coatings on top of the cold spray Ti substrates. This duplex coating structure is considered as a prototype for a possible repair route for Ti alloy compressor blades or landing gear components. The tribological properties of these coatings were studied with regard to both their performance and the effect of the properties of underlying substrates. For this investigation, tribological performance was evaluated with a reciprocating tribometer combined with microstructural characterization of wear track morphology and cross-sectional observation of coating/substrate featured by focused ion beam microscopy.

## 2. Methodology

## 2.1. Cold spray substrate deposition process

Cold spray coatings, to be used as substrates, were produced from spherical, commercially pure, Ti powder (Raymore Industries, Inc.) with 25 µm average particle diameter [37], accelerated to supersonic velocities with Kinetic 4000 cold spray gun (CGT Technologies, Germany) systems. Three different specimens were deposited with the helium gas preheated to 350 °C and using three different gas pressures of 2, 3 and 4 MPa.

The velocity of particles for each deposition condition was measured in free jet with a DPV2000 (Tecnar Automation, St. Bruno, QC, Canada). The gun traverse velocity was maintained at 330 mm/s and the feeding rate of 20 g/min was used to produce substrates 5 mm in thickness on top of commercially manufactured Grade 2 bulk Ti plate (McMasterCarr, Aurora, OH) 3 mm in thickness. The substrates were polished on 320, 400, 600, 800 and 1200 grit grinding papers. The final polishing steps consisted of 0.05  $\mu$ m colloidal silica with 10% hydrogen peroxide until scratch free surfaces were obtained.

#### 2.2. HIPIMS process

TiN, TiSiN and TiSiCN coatings were deposited in dual magnetron sputtering system equipped with Ti and Si targets. The base pressure in the chamber was  $\sim 10^{-5}$  Torr. Prior to coating deposition, bulk Ti and cold sprayed substrates were pretreated by HiPIMS etching process using Ti + and Ar + ions at a constant bias of -900 V. After pretreatment, coatings were fabricated in 2 steps, where the Ti target power was constant throughout. In the first step, ~200-400 nm thick Ti metal base layer was deposited by HiPIMS followed by 2 µm thick TiN, TiSiN and TiSiCN coatings, with the deposition parameters listed in Table 1. Nitrogen to argon gas flow rate ratio during the deposition was about 1:18. A bias voltage of -50 V was used to deposit TiN, TiSiN and TiSiCN. Based on the average current measured during each of the depositions (Table 1) and the substrate dimensions the average current density in units of A/cm<sup>2</sup> was 0.09 for TiN, 0.11 for TiSiN and 0.07 for TiSiCN. Substrates were unheated and their temperature never exceeded 100 °C during deposition. The coating deposition parameters are summarized in Table 1.

#### 2.3. Microstructural characterization

The phase analysis of the HiPIMS coatings was done using Bruker D8 Discover X-ray diffractometer with Cu-K $\alpha$  radiation in a standard  $\Theta$ -2 $\Theta$ configuration. The filament current and accelerating voltage were 40 mA and 40 kV, respectively. The range of diffraction angle used was 10 to 100°. The microstructural characterization was performed with Light Optical Microscope (LOM), Hitachi SU3500 and SU8000 Scanning Electron Microscopes — SEM (Hitachi, Japan) and Philips XL30 FEG-SEM (FEI Company, Netherlands). The porosity of the cold spray substrates was measured from the LOM top view images and with Image analysis software (Clemex Vision Professional 5.0, Clemex Technology Inc., Longueuil, QC, Canada). An average of the six porosity measurements was taken while standard deviation was used as an error bar.



Fig. 1. Top view LOM images of Ti cold spray coatings: a) CS1, b) CS2 and c) CS3.

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