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# Residual stress control in TiN/Si coatings deposited by unbalanced magnetron sputtering

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**Abstract**

TiN coatings deposited by unbalanced magnetron sputtering were used in a case study to determine the relationship between some deposition parameters, such as substrate bias and working gas pressure, and residual stress magnitude in the coatings. The test coatings, ranging in thickness from 1 to 12 μm, were deposited on thin silicon wafers and evaluated for residual stress magnitude and anisotropy, as well as nanohardness. It was found that the properties of TiN coatings could be linked to the stress level, which in turn was strongly affected by the deposition process conditions. The stress–temperature correlations were investigated by subjecting the coatings to a temperature cycle from room temperature to about 100 °C above the maximum deposition temperature. Stress–temperature plots were used to characterize residual stress relaxation in the coatings during post-deposition heat treatment.


*Keywords:* Magnetron; Reactive sputtering; Titanium nitride; Residual stress; Thermal relaxation

## 1. Introduction

Unbalanced magnetron sputtering (UMS) is one of the leading PVD technologies in the development of advanced protective coatings for aerospace applications. The functions of these coatings include corrosion, wear and erosion protection of airplane components working under extreme load, temperature and environmental conditions. A common challenge for all these applications is achieving high coating integrity by controlling residual stresses in the coatings.

The issue of residual stress is particularly important in the case of erosion-resistant coatings used to protect compressor components of gas turbine engines [1–3]. Damage caused by eroding particles, such as sand or airborne ash, gradually lowers engine power, decreases fuel efficiency and, in effect, severely shortens engine service life. Protective coatings used in this application need to conform to a number of conflicting, from the point of view of residual stresses, requirements. Firstly, they need to be relatively thick, up to 10–20 μm, to provide required lifetime under erosion conditions. This would imply a low level of residual stress, as highly stressed coatings could fail, most likely by spallation. On the other hand, erosion-resistant coatings work better when they remain under relatively high compressive stress. In the case of hard and brittle ceramic coatings, when fracture dominates the mode of erosion, a controlled amount of compressive stress, in the order of 1 to 4 GPa, can enhance erosion resistance [4–7]. Another factor that needs to be considered in aerospace applications is a fatigue life of coated components. Under this criterion, it is beneficial when the surface layer of a substrate material remains under compressive stress, effectively inhibiting propagation of fatigue cracks in the substrate. The latter implies presence of tensile stress in the coating.

From the preceding discussion it is evident that residual stress management is a critical step in the design of erosion-resistant coatings for turbine engine compressor components. Whether it is a humanitarian mission in

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Africa, forest firefighting in Canada or the US, or a military mission in a desert area, the aerospace industry needs this type of coating to extend the life of engines in service. Because of strict regulatory requirements, the only coating material already approved for erosion protection of compressor components in airplane engines is TiN. This coating can extend service life of coated components up to 2–3 times. However, in many applications it is not sufficient and work is ongoing for the development of new coatings, as well as improving TiN coatings through modeling of more efficient multilayered architectures and effective use of residual stresses.

The modeling efforts require extensive data on magnitude of residual stresses and mechanical properties of the coating, such as hardness (or Young's modulus), and their possible variations as a function of deposition conditions. Since erosion coatings in the compressor section of the engine can be exposed to temperatures exceeding 400 °C, stress and mechanical properties of TiN coatings need to also be known after high temperature exposure. Even with extensive literature available on TiN coating deposition and evaluation, a consistent set of such data would be hard to find. Therefore, it was produced for the purpose of this project, creating an opportunity to study correlations between residual stresses and coating properties in a wide range of deposition conditions. The experimental work was divided into two steps. In the first step, reported in this paper, TiN coatings deposited on silicon substrates were evaluated. In the second step (in progress at the time of this publication), the test procedure was repeated for TiN coatings on steel substrates.

## 2. Residual stress in UMS coatings

Residual stresses in thin PVD films have been investigated extensively for years and there are many good papers on this subject available in the literature [6–9], to mention a few. Provided below is a brief discussion of residual stress origin and magnitude in the case of magnetron sputtered coatings.

The major source of stress in UMS coatings is the deposition growth process. Due to a relatively low temperature and possibly working gas entrapment, not all atoms settle in their least energetic positions. This type of stress is known as an intrinsic residual stress ($\sigma_I$) and its value depends on film thickness, Young's modulus, and morphology and density of the film. The last two factors are clearly related to deposition conditions such as gas pressure, substrate bias and temperature. Typically, the UMS coatings have a high compressive residual stress level, reaching several GPa.

The other source of residual stress is thermal stress resulting from the difference in coefficient of thermal expansion (CTE) between the substrate and the coating. PVD coatings are normally deposited at elevated temperatures, and upon cooling to room temperature, thermal residual stress ($\sigma_T$) arises:

$$\sigma_T = \frac{E_F}{1 - n_F}(\alpha_F - \alpha_S)(T_D - T_M) \quad (1)$$

where $E_F$ is the Young's modulus of the film; $n_F$ is the Poisson ratio of the film; $\alpha_F$ and $\alpha_S$ are the CTE of the film and the substrate; $T_D$ is the deposition temperature; and $T_M$ is the temperature during stress measurement (typically room temperature). Eq. (1) is valid for a thin film on a thick substrate, which is the case for magnetron sputtered coatings.

Since the deposition temperature of UMS coatings is relatively low, the magnitude of thermal stress at room temperature rarely reaches more than a couple of hundreds of MPa, either tensile or compressive, depending on $\alpha_F$ and $\alpha_S$ difference. For example, in the case of a TiN coating on silicon substrate ($E_F$=300 GPa, $n_F$=0.25, $\alpha_F$=9.4e−6 $K^{-1}$, $\alpha_S$=4.0e−6 $K^{-1}$, $T_D$=190 °C, $T_M$=20 °C), the stress in the coating is tensile with $\sigma_T$=367 MPa. On the other hand, when the coating CTE is lower than the CTE for the substrate, as occurs for TiN coating on steel substrate ($\alpha_S$=11e−6 $K^{-1}$), thermal stress in the coating is compressive (negative) and $\sigma_T$=−109 MPa.

The total residual stress in the coating is a sum of thermal and intrinsic components:

$$\sigma = \sigma_T + \sigma_I. \quad (2)$$

High-modulus materials (such as ceramics) may store significant amounts of energy in the form of residual stress. On the other hand, low-modulus materials (such as metals) quickly relieve the stresses by plastic deformation. Accumulated stress energy in high-modulus coatings can be released either in post-deposition processing (annealing) or spontaneously by microcracking or spallation. This energy may be either detrimental (lower adhesion, tensile cracking) or beneficial (higher hardness and erosion resistance) to coating properties [7,11].

## 3. Experimental methods

### 3.1. Coating deposition

The tested coatings, with thicknesses from 1 to 12 μm, were deposited on 300 μm thick and 50 mm in diameter silicon substrates, using an UMS coater (Teer UDP-650), under various processing parameters such as deposition pressure, substrate bias, and deposition time. All coating runs were performed in an argon/nitrogen atmosphere with gas flow control. The amount of reactive gas was controlled by an optical emission monitor. Silicon substrates were washed in acetone and alcohol in an ultrasound bath, and were subsequently ion-cleaned in vacuum prior to deposition. The deposition temperature was measured using an infrared thermometer, type Modline3 by Ircon Inc.