



Bridge You and Nano

## Exponential Business and Technologies Company

### XRD Residual Stress Measurement of a TiN Coating

Residual stress can have a significant impact on a material's properties. These permanent, internal forces can have a positive or negative effect on a surface's durability, corrosion resistance, tribological and mechanical behaviors. For instance, the residual stress induced from improper welding can make pressure vessels fail prematurely. On the other hand, materials susceptible to stress corrosion cracking, such as nuclear pressure vessels, concrete rebars, and medical implants can show improved corrosion resistance when made with compressive residual stress.

Residual stress is often found in surfaces and coatings. Its presence may be a result of accelerated cooling during part fabrication such as iron and steel casting or plastic injection molding. Intentionally modifying the surface chemistry with processes such as nitriding can also induce residual stress in the nitrided steel surface. Coating processes such as physical vapor deposition can result in residual stress due to microstructural mismatch between coating and substrate material.

X-ray diffraction (XRD) provides a convenient, quantitative, and non-destructive method to measure residual stress. Unlike bending methods, it can measure stress in-situ. It works in principle by comparing the distance between atomic planes in the out of plane direction to the planes undergoing stress along in-plane directions. It is relatively surface sensitive and measures stress in 10s of microns for metals.

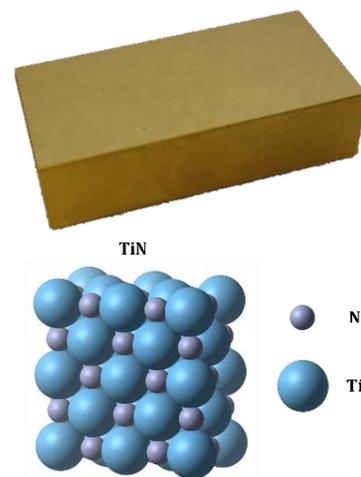


Figure 1. TiN coated steel block (above); crystalline structure of TiN (below).

In this application note, the residual stress of a titanium nitride (TiN) coating commonly added to improve tool bits' performance is determined using XRD. It is known that the hardness and scratch resistance are related to the residual stress in the coating – the higher the residual stress is, the harder and more scratch resistant the coating is. Shown in Figure 1 are a picture of the TiN coating on a stainless steel block and a schematic crystalline structure of the TiN compound.

Residual stress causes changes in the elastic strain of a crystalline material which corresponds to changes in the lattice spacing. The elastic strain can be measured by XRD using Bragg's Law at several values of  $\psi$ , the angle of tilt from the surface normal. The in-plane residual stress component is proportional to  $\sin^2(\psi)$ . By measuring the change in lattice spacing as a function of  $\psi$ , the in-plane strain can be calculated. The in-plane strain is used in conjunction with the Young's modulus and the Poisson's ratio of the material to determine the residual stress through the equation shown below:

$$\frac{d_{\psi} - d_0}{d_0} = \frac{1 + \nu}{E} \sigma \cdot \sin^2 \psi$$



Before measurements were conducted on the TiN coating test specimen, the instrument alignment and performance were verified using an aluminum powder standard sample known to be in a zero-stress condition. As can be seen from Table 1, the measured residual stress of the Al zero-stress standard fell within the ASTM Standard required accuracy of  $0 \pm 14$  MPa. After the instrument had been verified, XRD  $\omega$ - $2\theta$  scans of the TiN (422) peak at  $2\theta = 124.5^\circ$  were conducted at several different  $\psi$  angles. The shifts of the (422) peaks were measured to calculate the amount of strain along the in-plane direction.

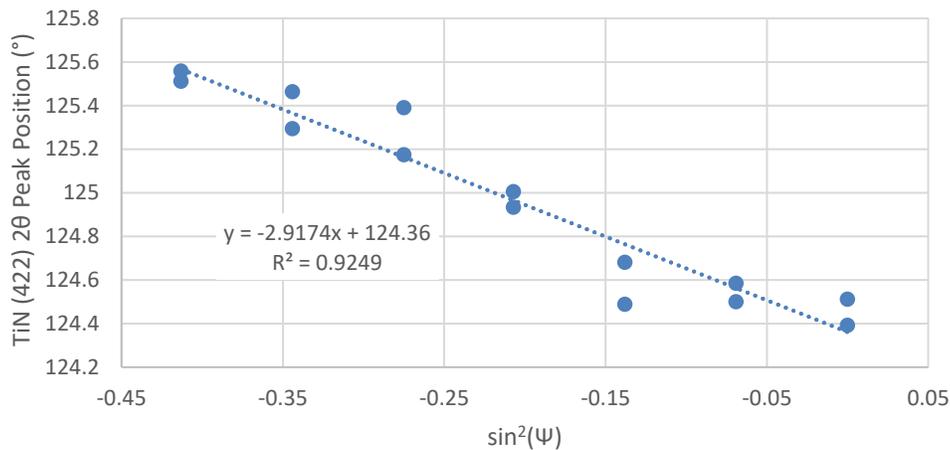


Figure 2. TiN peak position as a function of the tilt angle  $\psi$ .

Figure 2 shows the TiN (422)  $2\theta$  peak position as a function of  $\sin^2(\psi)$ . The slope of this fitted line, along with the material’s mechanical properties, were combined to determine the residual stress using the equation shown above. The Young’s modulus of this particular TiN coating was determined to be 448 GPa using nanoindentation. A Poisson’s ratio of 0.25 was used to obtain a stress constant K1 of 2149 MPa/°. The in-plane strain was calculated to be -2.4%, which led to a residual stress of  $-6.27 \pm 0.50$  GPa. Although this seems to be a large value for residual stress, it is comparable with published literature values of TiN films. Large compressive residual stress like this could provide increased resistance to scratch and wear along with possible better adhesion to the substrate.

Table 1 Residual Stress Measurement Results

Sample	K1 Stress Constant (MPa / °)	Peak Used for Measurement	Residual Stress (MPa)
Al Zero-Stress Standard	438.9	Al (422)	$-10 \pm 1$
TiN Coating on Steel	2149.5	TiN (422)	$-6271 \pm 496$