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Microstructure of nanocomposite TiSiN and nanolayered TiSiN/TiAlN coatings

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During the last few decades, hard coatings have been widely used for protection of mechanical components, particularly to increase the life of cutting tools. Among them, single-layer TiAlN stands up as the most commonly used coating for the protection of cutting and forming tools. TiAlN is based on fcc TiN structure and is characterized by high hardness (around 32 GPa), high stiffness (modulus of elasticity) and high oxidation resistance (up to 800 °C) [1]. Recently, nanocomposite and nanolayered coatings have attracted increasing interest. The most studied TiSiN nanocomposite coating consists of TiN nanocrystals embedded in an amorphous matrix of silicon nitride [2]. This coating exhibits superhardness (higher than 40 GPa) which is attributed to absence of dislocation activity in small crystals and suppression of grain boundary sliding by high cohesion strength between TiN and SiN_x phases. In addition, TiSiN nanocomposite coatings exhibit high oxidation resistance (around 850 °C) and high thermal stability (up to 1100 °C) [2]. Nanolayered coatings are composed of few nanometers thick layers (typically below 10 nm) of two or more different materials (eg. TiN/SiN_x). Their properties depend mostly on thickness of individual layers, number and width of interfaces [3,4].

In this research nanocomposite TiSiN and metastable TiAlN were combined in advance nanolayered TiSiN/TiAlN coating. Single-layer TiSiN and TiAlN were prepared for comparison. All coatings were deposited by magnetron sputtering in an industrial unit equipped with four sources. For the preparation of the TiSiN and TiAlN coatings two TiAl, and two TiSi targets were used, respectively. The nanolayered TiSiN/TiAlN coating was deposited from one pair of TiAl and one pair of TiSi targets positioned on both sides of the chamber. Coating microstructure was analyzed by scanning electron microscopy, conventional and high resolution transmission electron microscopy and X-ray diffraction. Chemical and phase composition were assessed by X-ray photoelectron spectroscopy. Mechanical properties were determined by nanoindentation technique.

According to the results of XRD and XPS measurements it appears that TiSiN and nanolayered TiSiN/TiAlN coatings are nanocomposites composed of crystalline and amorphous phases. The single-layer TiSiN coating is built of TiN crystals embedded in amorphous Si_3N_4 matrix, while the nanolayered TiSiN/TiAlN coating consists of crystalline TiN and Ti1-xAlxN phases along with amorphous Si_3N_4.

Both coatings are characterized by fine-grained morphology as can be seen in Figure 1 and in Figure 2. The TiSiN coating is composed of nanocolumns approximately 4 nm wide and few tens of nanometers long (Figure 1a). The fast Fourier transformation analysis of zones selected in HRTEM image revealed that each nanocolumn is composed of several nanocrystals. The results of FFT analysis of several zones of different nanocolumns are presented in Figure 1b. Nanolayered structure of TiSiN/TiAlN coating can be clearly distinguished from Figure 2. The TiSiN layer blocks the growth of TiAlN crystallites which are equiaxed and size around 5 nm. The SAED patterns shown in the inserts of Figure 1a and Figure 2a confirm nanocrystalline nature of both TiSiN and TiSiN/TiAlN coatings. The diffraction rings correspond to fcc TiN-like phases.

The hardness of 39 GPa was measured on nanolayered TiSiN/TiAlN coating. This is considerably higher from the value of 29.7 GPa which was estimated by applying the rule of mixture for the measured hardness of 46.3 GPa and 25.2 GPa of constituting TiSiN and TiAlN coatings, respectively. This high hardness value is attributed to hindering of dislocation motion due to grain size refinement along with blocking of grain boundary sliding by relatively sharp interfaces.

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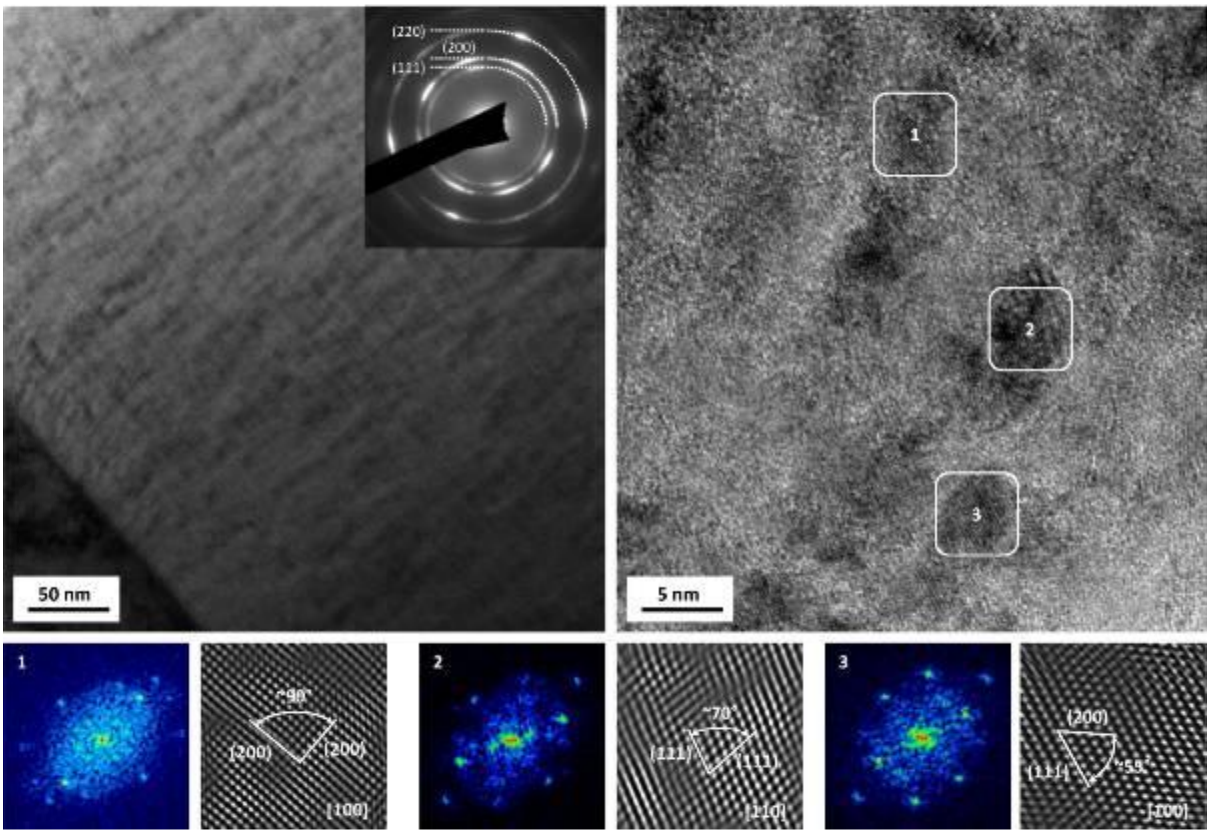

Figure 1. Bright field image a) and HRTEM image b) of the cross-section through TiSiN coating, with FFT patterns along with inverse FFT images. The SAED pattern is included in the figure a).

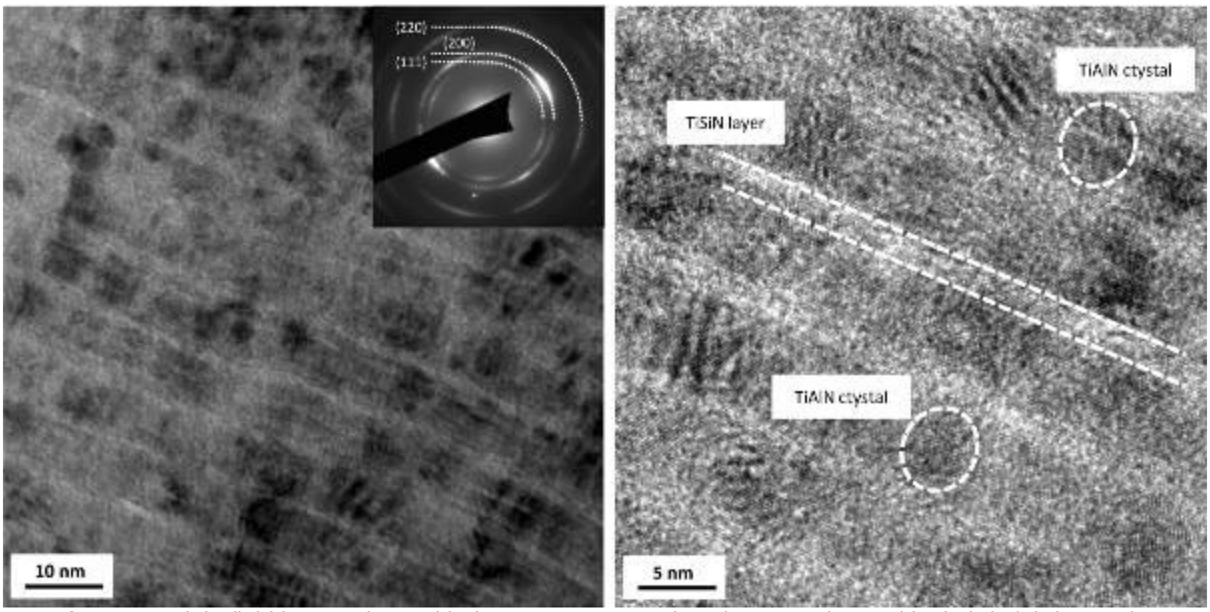

Figure 2. Bright field image along with the SAED pattern a) and HRTEM image b) of TiSiN/TiAlN coating