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NANOHARDNESS AND MODULE OF ELASTICITY OF CR-BASED NITRIDE COATINGS DEPOSITED AT TEMPERATURES BELOW 200°C

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ABSTRACT

Sets of Cr-based hard coatings were prepared via reactive closed field unbalanced magnetron sputtering (CFUBMS). Substrate temperature was kept in range of 150 to 200℃ during deposition. Nanohardness (H) andeffective modulus of elasticity (E) of the CrN, CrAlN, CrTiN and CrTiAlN coatings were examined using depth-sensing indentationmethod. The results from the combined examination of the research ratios showed that the triple and quaternary hard coatings have superior mechanical properties and they strongly depend on the deposition conditions.*

1. INTRODUCTION

Deposition of hard coatingson machine componentsand toolsis an effective method of improving their performance and they expand theirvariousapplications worldwide [1]. Cr-based coatings are widely applied in industry for different applications [2, 3]. Deposition temperature ranges below 200℃ seriously bother the obtaining of optimal mechanical characteristics for the conventional PVD coatings. Different heat sensitive industrial materials like cold working alloyed steels, copper alloys, die steels, polymers, etc. [4] could improve their exploration time if hard coatings with optimal mechanical properties are deposited at these lower temperatures.

Magnetron sputtering hasbeen applied for a long time as an effective method of physical vapour deposition ofthin films [5]. Closed magnetic field, generated by unbalanced magnetronseffectively improves coating quality. This technology popular as Close Filed Unbalanced Magnetron Sputtering (CFUBMS) [6] could be used for deposition of Cr-based coatings under 200°C.

The nanohardness of the materials characterised their resistance to plastic deformation [7], while elastic module describes the resistance of a certain material to elastic deformation [8]. These parameters are easy to obtain via instrumented nanoindentation on very fine scale.

The focus of this research is the investigation of the nanohardness and module of elasticity of Cr-based hard coatings, deposited via CFUBMS at low temperatures and intended for real industrial applications.

2. EXPERIMENTAL DETAILS

All of the coatings were deposited onto hardened high-speed steel (HSS) substrates type EN 1.3343 via CFUBMS. The process was carried out with four rectangular magnetrons two of them Ti, one Cr and one Al all of them of high purity (99,999%). Current in the range 0.5-8 A was applied to thetargets in DC regime for the Ti and Cr targets and in pulsed regime (150 kHz, 1500 ns)to the Al target. The substrates were degreased and dried prior to the deposition process.

The Ar flow of 25 sccm was controlled by mass flow controller (MFC). The nitrogenflow, also controlled by MFC was variating between 15 and 30 sccm. The working pressure was maintained between 0.19 to 0.36 Pa, correspondingly. The rate of the samples holder was kept at 5 rpm. Prior to deposition the samples were cleaned in Ar plasma for 30 minutes at a bias voltage of -500 V for removing contaminants from the substrate surface. Bias voltage of -70V was applied to the samples andthe substrate temperature was in diapason of 150 to 200ºC during all of the deposition processes.

Nine Cr-based coatings were depositedwith different ratio of contained metals and nitrogen: Cr, CrN, CrAlN, CrTiN and CrTiAlN.

The triple and quaternary coatings were deposited with Cr adhesive layer (~0.1 µm) and CrN transition layer (~ 0.2 µm).

The mechanical properties of the coatings were characterised via depth sensing methodappropriate for characterization of the nanohardness and module of elasticity [9]. A Nanoindentation Hardness Tester (NHT) (Anton Paar), equipped with a diamond Berkovich indenter was used. The applied load was in range of 10 to 20 mN. The maximum penetration depth is chosen to be between 10% and 25% of the total coating thickness to avoid the substrate influence [10, 11]. Indentation depths lower than

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10% of the coating thickness is not recommended because they will lead to an increase in the Indentation Size Effect [12]. The hardness and the elastic modulus were calculated, applying the method, proposed by Oliver and Pharr [10, 11].

3. RESULTS AND DISCUSSION

The calculated values of nanohardness (H) and effective elastic modulus (E*) are shown in Table 1. The coating mechanical properties were examined by the application of different load, chosen in the range of 10 to 20 mN for each sample depending on its thickness.

Table 1 *Mechanical properties of the substrate and the coatings*

#	Coating	H, [GPa]	E*, [GPa]
1	Cr	8.1	295
2	CrN	17.9	383
3	CrAlN(1)	19.1	368
4	CrAlN(2)	20.1	279
5	CrTiN(1)	27.4	272
6	CrTiN(2)	29.9	392
7	CrTiAlN(1)	39.1	374
8	CrTiAlN(2)	32.4	399
9	CrTiAlN(3)	30.5	358
10	HSS substrate	11.5	274

During the nanoindentation any significant cracks were not detected in the load-displacement curves. Improved mechanical properties of the coatings, compared to HSS substrate were obtained.

Fig. 2 presents nanohardness and effective modulus of elasticity of the examined coatings and of the bare substrate.

The measured value of the Cr layer 8 GPa is near to the values cited in the literature in range of 11-14 GPa. The CrN hard coating show higher nanohardness of 18 GPa because of the formation of nitride phase that could increase the nanohardness even to 28 GPa [13, 14]. For the triple and quaternary hard coatings are measured higher values of nanohardness supported from the including of Ti and Al in their composition [15]. The nanohardness of the CrAlN hard coatings is 19-20 GPa and this value depends strongly on the deposition conditions asthe nanohardness of CrAlN coatings could be also in a wide range: 15-36 GPa [16].

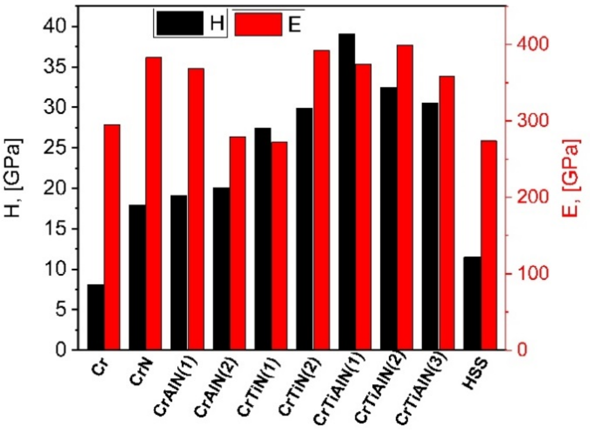

Fig. 1. Nanohardness and modulus of elasticity

The CrTiN coatings are characterized with nanohardness between 27-30 GPa identical with the reported values for CrTiN coatings between 25-29 GPa [17]. The superior nanohardness of this group of Cr-based hard coatings is seen for the CrTiAlN coatings measured between 30-39 GPa compatible with the value of 35 GPa reached from other researchers [17]. The nanohardness values of the different coatings compared to each other shown that their mechanical properties strongly depend on the process conditions like target current, nitrogen flow and working pressure. The measured values of modulus of elasticity for the different layers are as follows: Cr (~300 GPa), CrN (~380 GPa), CrAlN (~280-370 GPa), CrTiN (~270-390 GPa) and CrTiAlN (~ 360-400 GPa). It seen that even hard coatings with similar hardness could also have a different modulus of elasticity and the main contributing factor for this result is the variation in their chemical composition [18]. As a conclusion, the nanohardness and modulus of elasticity are improved for the ternary and quaternary Cr-based coatings compared tothe substrate and the binary CrN coatings.

4. CONCLUSIONS

The nanohardness and module of elasticity are studied for Cr-based hard coatings deposited at temperatures under 200°C. The results show that independently of the lower deposition temperature the mechanical properties of the researched coatings are comparable with the cited in literature for this class of coatings. The highest nanohardness is measured for the CrTiAlN hard coatings. The module of elasticity strongly depends on the composition of the hard coatings.

The results of the measured nanohardness H and module of elasticity E could be used for investigation of the different H to E ratios and the different tribological and fracture resistance properties to be identified, which is our next research go.

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