Properties of niobium-based wear and corrosion resistant hard PVD coatings deposited on various steels

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*B*ulk niobium is well known for its excellent corrosion resistance. To utilise the advantages of this exceptional material we have combined the materials niobium and chromium to form superhard PVD coatings with a plastic hardness typically of 45 to 60 GPa.
To minimise the well-known pin-hole problem inherent to PVD coatings a multi-stage coating process has been developed featuring the following deposition sequence: Low energy Nb^{3+} ion-implantatrion followed by the deposition of Nb barrier layer, CrN base layer and CrN/NbN superlattice coating. The ion implantation is carried out using a steered cathodic arc plasma as metal ion source, whereas the PVD coatings were grown by unbalanced magnetron sputtering. The superlattice coatings are prepared in a multi-target PVD coater by simultaneous operation of two targets of Nb and Cr each. Potentiodynamic polarisation tests in chlorine containing aqueous media show corrosion current densities for coated stainless steel even for coated mild steel samples as low as 10^{-6} $A.cm^{-2}$ and pitting potentials beyond +600mV. In salt spray tests coated mild steel samples show first sign of corrosion damage after 300 h compared to 200 h of 25 µm thick electroplated hard chromium.
The sliding wear resistance is ten-fold as high as that of electroplated hard chrome.

INTRODUCTION

The environmental pressures on manufacturers for changing from hexavalent hard chrome to more environmentally friendly coatings have now reached the point where tackling this problem is seen as a necessity. Although the PVD technique provides the best conditions from the environmental point of view, the columnar structure [1] of PVD coatings is an inherent drawback, which prohibited their broad industrial use as of protective corrosion resistant coatings. Interrupting the columnar grain growth when exploiting a multi-layer concept [2, 3] can amend this disadvantage. The introduction of nanoscale multilayer/superlattice structures, where the thickness of the individual layers is brought into the nm range, opened further prospect not only for significant hardness enhancement [4,5] but also for more effective protection against corrosion [6]. It has been demonstrated that superlattice coatings dedicated to serve applications as tribological, high temperature and corrosion resistant coatings can be economically manufactured in industrially sized machines using the combined cathodic arc/unbalanced magnetron deposition method, (ABS: Arc Bond Sputtering), [7, 8, 9, 10]. Belonging to the category of superhard materials (HP = 56 GPa), the CrN/NbN system has shown excellent corrosion behaviour both in potentiodynamic and salt spray test conditions [6, 11]. The ABS technology exploits low energy metal ion implantation during the metal ion etching step, to engineer the coating substrate interface. It has been demonstrated that 1.2 KeV Nb^+ etching ions and sufficiently high bias voltages during coating (Ub = - 120V) considerably reduce the corrosion current density in potentiodynamic polarisation tests and increase the life time in salt spray tests

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[10]. However the CrN/NbN superlattice coating alone can not provide reliable protection when deposited directly onto mild steel material. This paper reports on 5-7 µm thick nanoscale PVD multilayer/ superlattice coatings protecting stainless steel and mild steel against wet corrosion. In the case of mild steel the coating consists a monolithically grown pure Nb layer, which acts as corrosion barrier between substrate and the base layer of the CrN/NbN superlattice. Such layer system provides similar corrosion protection and improved wear resistance than 25 µm thick electroplated Cr when mild steel is used as substrate material.

NANOSCALE MULTILAYER/SUPERLATTICE COATING DEPOSITION BY COMBINED STEERED ARC/UNBALANCED MAGNETRON TECHNOLOGY

Three types of substrate materials were used for this study: mild steel BS 6323 (0.2% C), mirror- polished discs substrates 25 mm dia., or cylinders 50 mm dia. and 60mm – 120 mm high. The other two materials were M2 high speed steel (HRc= 65) and 304L stainless steel discs. Prior to coating the samples were cleaned and rinsed in an automated cleaning line comprising a series of ultrasonically agitated aqueous alkaline solutions and deionised water baths followed by a vacuum drier.
A commercially available HTC 1000-4 ABS system [12, 13] comprising four rectangular cathodes that can be operated either in steered arc or in unbalanced magnetron mode was used for the coating deposition. Fig. 1a schematically outlines the cross-section of the combined arc-magnetron PVD system and the target arrangement. In the case of CrN/NbN coatings two opposing pairs of cathodes were furnished with Cr and Nb (99.8% pure) targets. Sputtering took place in a reactive atmosphere of Ar + N_2.
Fig. 1b schematically shows a cross-section of the multilayer / superlattice coating developed to protect mild steel
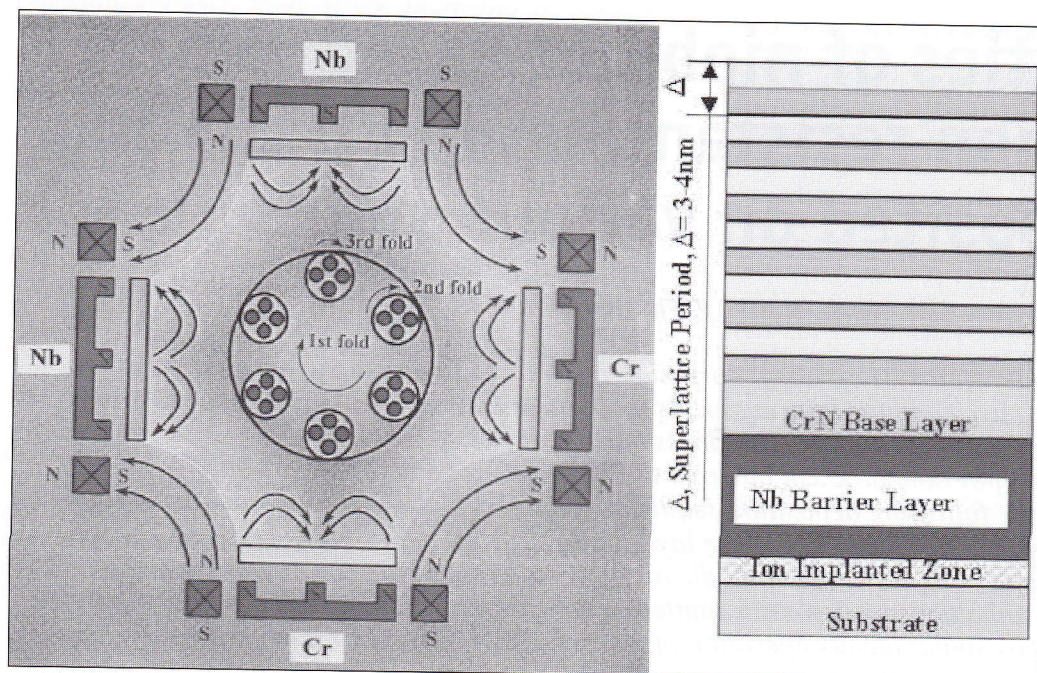
Fig. 1: Schematic cross-section of: a) HTC 1000-4 ABS system and b) multilayer / superlattice Nb-CrN- CrN/NbN coating.
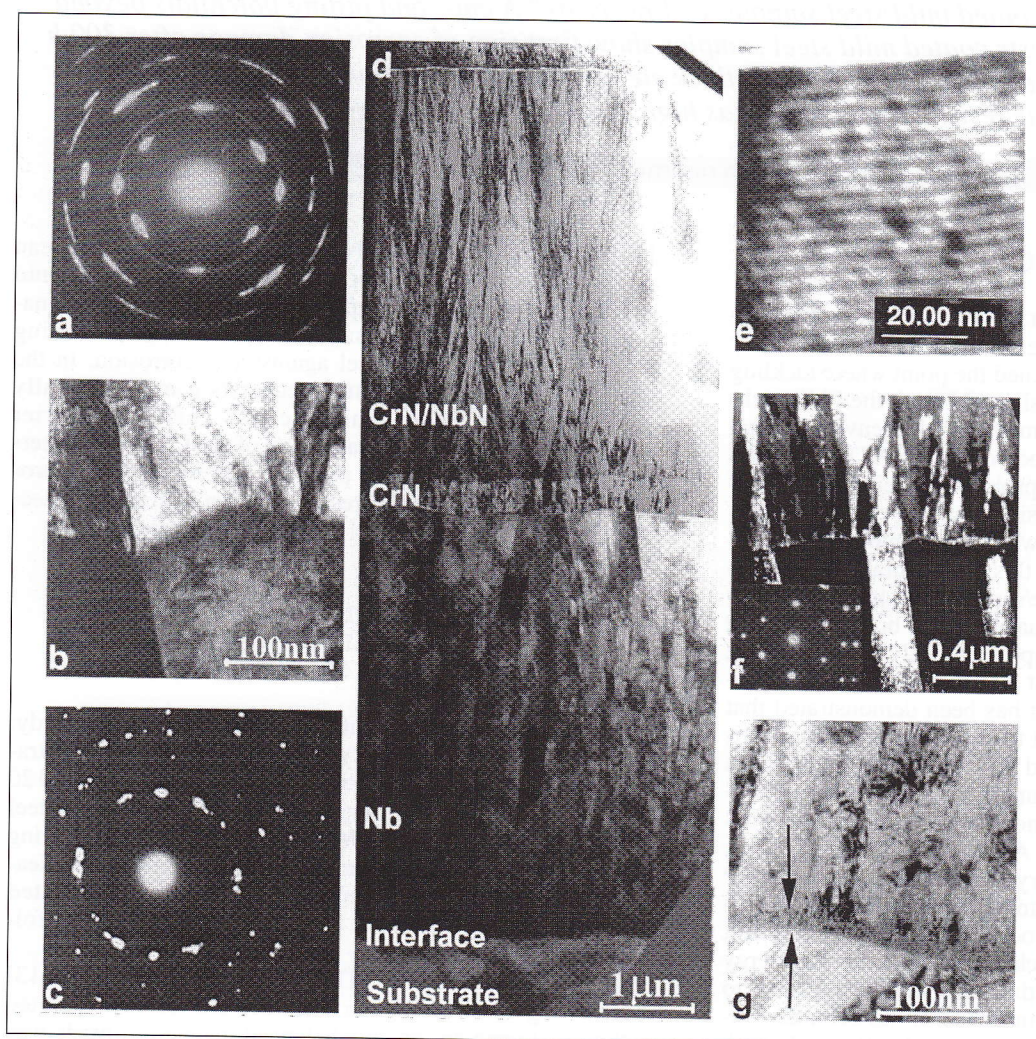

Fig 2: XTEM cross section from the Nb-CrN-CrN/NbN coating ; a) selected area diffraction pattern (SADP) taken from the superlattice coating, b) a higher magnification image of the Nb-CrN interface, c) SADP taken from the Nb layer, d) an overview of the multilayer/superlattice Nb-CrN-CrN/NbN coating archtecture, e) Higher magnification of the CrN/NbN superlattice coating, e) dark field image from the Nb-CrN-CrN/NbN coating including interface regions from Nb-CrN and CrN-CrN/NbN, g) higher magnification image of the Nb coating-substrate interface showing the thickness of the amorphised layer.

against corrosion. The coating includes the following regions: Nb ion implanted zone, Nb barrier layer, CrN base layer and CrN/NbN superlattice structured main layer. In the first stage of the process a steered arc Nb+ metal-ion etching was employed to guarantee both a superior adhesion and corrosion resistance. The bombardment by highly ionised Nb^{3+}, generated by the cathodic arc discharge, provides intensive sputter cleaning of the substrate surface and low energetic ($E_i \approx 3.6$ KeV) ion implantation resulting in compositionally and structurally tailored interface formation [13, 14]. Additional benefit of Nb as an etching metal is given by providing a smooth with minimised droplet generation substrate surface [15]. In the case of mild steel substrates, the metal ion etching step was followed by non reactive unbalanced magnetron sputtering of 1-3 µm of Nb, using two Nb targets. The Nb barrier was deposited in three thick-

nesses, namely 1, 2, and 3 μm. A 0.3μm thick CrN base layer was then deposited operating two Cr targets in reactive Ar and N_2 atmosphere. The 4.5μm thick CrN/NbN superlattice main coating was deposited by reactive sputtering using all four targets simultaneously.

In the case of stainless steel substrate the deposition of Nb barrier layer was omitted, thus the CrN base layer was deposited directly on the Nb^{3+} metal ion etched surface. The substrate temperature during deposition was 450°C. Detailed deposition parameters are given in [11, 16].

MICROSTRUCTURE OF THE COATINGS

The microstructure of the Nb-CrN-CrN/NbN multilayer/ superlattice system used to protect mild steel against corrosion was determined by XTEM analyses (Fig. 2a to 2g).
An overview of the structure of the multilayered system comprising a 3μm thick Nb barrier layer is given in Fig. 2d. The characteristic features are the interface zone, followed by the dense, columnar 3μm Nb coating, the dense columnar 0.3μm thick CrN base layer and finally the 4.5 μm thick CrN/NbN superlattice main coating. The interface zone is shown at a higher magnification in Fig.2g. The dark ribbon marked by arrows corresponds to the 8nm thick implantation zone resulting from the low energetic Nb^+ ion implantation step carried out prior to the sputter deposition [10, 13]. Furthermore XTEM analysis revealed that sputter deposited Nb coating exhibits a dense structure free of voids and pores. The selected area diffraction pattern (SADP) taken from the Nb layer (Fig.2c), showed a {110} texture. In Fig. 3b a higher magnification image of the interface between the Nb coating and the CrN is shown, revealing a highly facetted surface of the Nb coating with a column size larger than 100nm. It also reveals that the column size of the CrN coating is much smaller, which indicates little or no orientation dependence from underlying Nb coating.
The microstructure of the CrN base layer is shown in the DF

X-TEM image, Fig. 2f. SADP reveal a {110} texture. This texture is typical for sputter deposited CrN [17]. The introduction of the CrN base layer has effectively interrupted the growth of the large Nb grains In Fig. 2e a higher magnification image of the CrN/NbN superlattice coating illustrates the individual layers of the sequentially deposited CrN and NbN. Fig.2e allowed direct determination of the superlattice period. The selected area diffraction pattern (SADP) taken from the top region of the coating (Fig. 2a), indicated {200} texture.No pores or voids were observed in the superlattice stack. A similar dense structure with well pronounced nanocrystalline Nb layer at the coating substrate interface, a {200} texture and precise layered structure was observed in the case of CrN/NbN superlattice coating deposited on stainless steel.

MECHANICAL AND TRIBOLOGICAL PROPERTIES

The mechanical and tribological properties of the Nb-CrN-CrN/NbN multilayers deposited on soft mild steel (HK$_{25g.}$ = 260) and hardened HSS (HK$_{25g.}$ = 950) substrates are summarised in Table 1. These parameters are compared to hard electroplated Cr, as well as CrN/NbN superlattice coating deposited directly onto both types of substrate materials without a metallic Nb barrier layer.
The hardness measurements revealed that the underlaying Nb film does not influence significantly the hardness of the 4.5μm thick CrN/NbN superlattice main coating. The introduction of the Nb layer has led to a hardness decrease of less than 10% more or less independent of the substrate material. The adhesion of the multilayer coating measured by scratch test (critical load, Lc) however, is strongly influenced by the thickness of the Nb underlayer. Critical load values Lc as low as 20N were obtained with 3μm thick Nb layer compared to 70 N of a Nb barrier free coating. Closer observations of the scratch track morphology revealed that intensified crack formation within the CrN/NbN coating was taking

Coating / Parameter	Nb+, CrN/NbN Ub= -120V on HSS 0 μm Nb	Nb+, Nb-CrN-CrN/NbN On MS 0 μm Nb	Nb+, Nb-CrN-CrN/NbN On HSS 1μm Nb	Nb+, Nb-CrN-CrN/NbN On MS 1μm Nb	Nb+, Nb-CrN-CrN/NbN On HSS 2μm Nb	Nb+, Nb-CrN-CrN/NbN On MS 2μm Nb	Nb+, Nb-CrN-CrN/NbN On HSS 3μm Nb	Nb+, Nb-CrN-CrN/NbN On MS 3μm Nb	Hard Cr on HSS	Hard Cr on MS
Thickness, μm	4.2	4.0	5.4	5.4	6.6	6.6	7.8	7.8	25	25
Hardness, HK25 gr.	3750	3620	3750	3580	3500	3500	3520	3350	1450	1300
Hardness, HP$_{50mN}$, GPa	52	51	52	50	51	50	48	48	18	17
Adhesion, Lc, N	70	65	40	34	31	30	21	20	15.2	10
Coeff. of Frict. μ	0.87	0.80	0.9	0.81	0.75	0.73	0.70	0.70	0.72	0.78
Abr. Wear Coeff., Kc, [m²/N]	5.8×10^{-13}	5.8×10^{-13}	7.2×10^{-13}	7.0×10^{-13}	7.6×10^{-13}	6.8×10^{-13}	6.0×10^{-13}	7.3×10^{-13}	8.8×10^{-13}	9.8×10^{-13}
Sl. Wear Rate, Kc, [m²/N]	3.2×10^{-15}	4.4×10^{-15}	7.2×10^{-15}	6.7×10^{-15}	5.8×10^{-15}	5.7×10^{-15}	5.2×10^{-14}	6.2×10^{-14}	5.8×10^{-14}	2.3×10^{-13}

Table 1. Mechanical and tribological characteristics of various coatings.
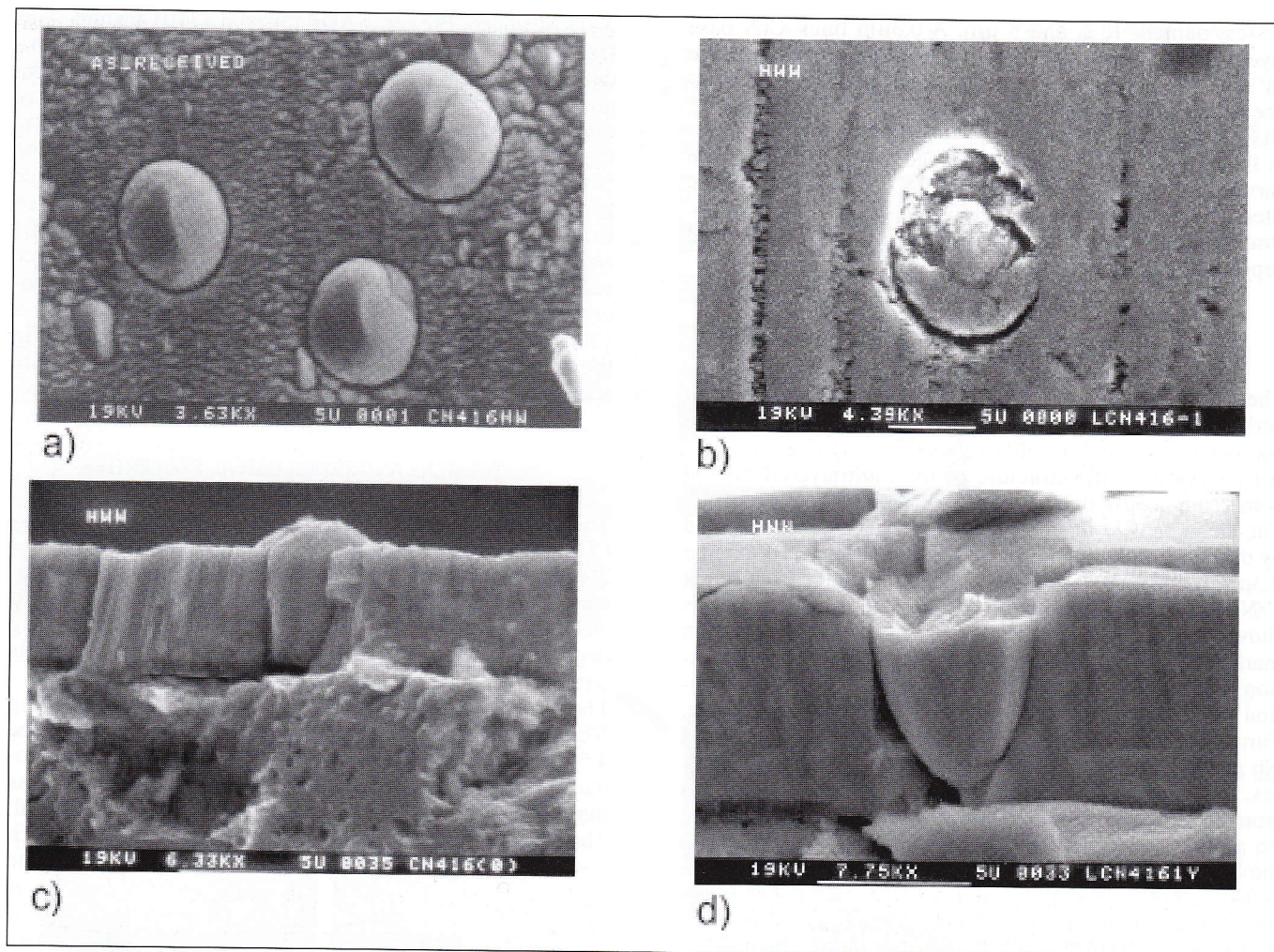
Fig.3. Plain view and fracture cross- section SEM micrograpfs showing the coatings morphology:
a, b- before erosion, c, d- after erosive wear.

place when the coating was deposited on a thicker Nb layer. The critical load values Lc, of the electroplated hard chrome were even lower than those of the PVD coatings, namely 10N to 15N depending on the hardness of the substrate, (see Table 1). The coefficient of friction, determined by Pin-on-disk test, of the Nb-CrN- CrN/NbN multilayers was found to vary in the range between 0.9 to 0.7 decreasing with increasing Nb barrier layer thickness. Using the thicker Nb barrier layer, these values are comparable to the values of the coefficient of friction obtained for the electroplated hard chrome. Tests have also been carried out to evaluate the wear coefficients both in dry sliding and microabrsive wear conditions using CSEM Pin-on-disc and Calowear test equipment. The microabrasive wear coefficient Kc of the CrN/NbN based PVD coatings was found to be marginally lower than that of the electroplated hard chrome coating However, in dry sliding wear tests the Nb-CrN-CrN/NbN multilayer showed more than a factor of 10 higher resistance against wear compared to 25 µm electroplated hard chrome. The results show that 3 to 5 times thinner PVD coating clearly outperform 25 µm electroplated hard chrome. Even in the worse case, when the superlattice coating was deposited on 3 µm Nb barrier layer, the sliding wear test showed approximately 8 times higher wear resistance compared to electroplated on mild steel hard Cr.
The excellent wear behaviour of CrN/NbN superlattice coating was confirmed in corrosion-erosion tests as well [18]. The coatings were subjected to solid particle erosion under simultaneous corrosion, by potentiodynamic polarisation, in a turbulent alkaline slurry [19]. The slurry was an 0.5 M mixed solution 1:1 by volume of $NaHCO_3$ and Na_2CO_3 prepared out of de-ionised water, which contained 150 µm alumina particles. In erosive wear, ring samples were rotated at 2000 rpm for 24 hours in the slurry rig at an equivalent linear speed of 240 m.min^{-1}, with potentials maintained at – 1.0 or + 0.1V. The specimen surface after test was characterised mainly by means of scanning electron microscopy or gravimetric measurements. The wear mechanism in the conditions of this test can be described mainly as glancing angle micro-cutting wear. As reported previously [10], the coating encompasses a significant hard CrN/NbN superlattice matrix and growth defects generated from macroparticles deposited on the substrate during the metal ion etching step of the process. Although they represent only a small volume of the coating it has to be taken into account that due to the imperfect structure, growth defects can be significantly softer compared to the superlattice matrix. Fig. 3 a,b show SEM planar and cross sectional fractoview of the as deposited CrN/NbN coating. The imbedded defects were found to be through-thickness forming a dome like structure on the coatings surface. Shrinkage clearance separated the defects from the adjacent coating matrix opening direct channels for the environmental attack of the substrate material. Under erodent impact, the growth defects have been preferentially "topped-off" by abrasive cutting wear (lapping wear), with the remaining coating matrix largely intact,

Fig. 3 c,d. This behaviour could be explained by the lower hardness of the defected coating material and the geometrical form of these defects. The protruding dome like structured surface defects induce turbulent flow in the slurry leading to higher abrasive concentration on the defects. However due to the negligibly small amount of the defected material, the weigh loss after 24 hours was almost unmeasurable,` while the not protected sample material (for both stainless steel and mild steel) was heavily eroded after only 15 min. in the test conditions used in this study.

POTENTIODYNAMIC POLARISATION AND SALT SPRAY TEST RESULTS

Fig. 4 shows potentiodynamic polarisation data for stainless steel, hard Cr and CrN/NbN superlattice coating deposited at two different bias voltages tested in aerated 0.1M acetic acid, sodium acetate buffer solution with 0.1M added NaCl solution specially developed to promote pitting.
In these conditions electroplated Cr did not show any passivation behaviour with corrosion current densities as high as $1.0 \, 10^{-4} \, mA \, cm^{-2}$. In contrast CrN/NbN superlattice coatings demonstrated enhanced corrosion performance with passive curr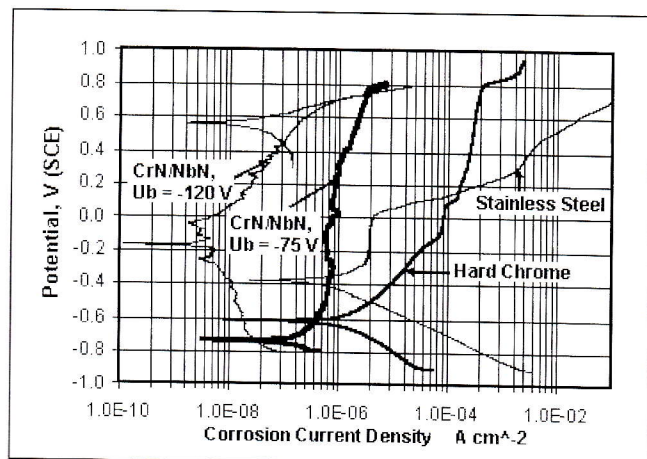ent densities as low as $10^{-6} – 10^{-8} \, mA \, cm^{-2}$ when deposited at Ub= - 75V and Ub= - 120V respectively. Prior to the deposition, the stainless steel substrate was subjected to Nb$^+$ ion etching. The XTEM analyses showed that the Nb$^+$ ion etching in this case led to formation of 5-10nm Nb implanted zone and deposition of 5-8nm thin nanocrystalline Nb layer at the coating substrate interface. The extremely dense, almost amorphised structure of this layer together with the electrochemical stability of the Nb itself enhances dramatically the corrosion resistance of the successively deposited coating.
Comparative studies, involving coating porosity evaluation by impedance measurements, reviled that the CrN/NbN superlattice coating provides greatly improved corrosion protection compared to monolithically grown CrN coatings due to the presence both the highly passive element Nb and particularly to an altered microstructure resulting in diffused porosity and fewer through thickness defects [6]. The role of the through thickness, growth defects was studied by cross sectional SEM and models were suggested to explain corrosion failure mechanisms of PVD superlattice coatings [20]. It has been shown that the corrosion attack of the substrate takes place mainly through growth defects, while the coating itself remains intact. Applying sufficient ion bombardment during film growth significantly eliminated the dentritic, open structured film growth as well as the growth defects density. Increasing the bias potential from Ub = - 75 to Ub = - 120 V led to decrease in surface roughness from 0.72μm to 0.3μm respectively Fig. 5.
For the smoother coating deposited at Ub = - 120V, the passive current density of CrN/NbN superlattice coating was reduced two orders of magnitude to $1.0 \, 10^{-8} \, mA \, cm^{-2}$ and no pitting was observed evident from the reverse part of the potentiodynamic polarisation curve in Fig. 4.
Salt spray tests confirmed the excellent performance of CrN/NbN to protect stainless steel against corrosion. After 1500 hours at temperature of 35°C and 98% humidity no pitting of the coating was observed. The accepted in the electroplating industry standard life- time for hard chrome in these conditions is 1000 hours.
The multilayer/ superlattice Nb-CrN-CrN/NbN system was designed to protect mild steel against corrosion The coating architecture comprises of 8nm nanocrystalline Nb layer followed by 3μm thick Nb layer, 300nm CrN and finally 4.5 μm thick CrN/NbN superlattice coating.
The potentiodynamic polarisation in 3% NaCl solution revi-

Fig. 4. Representative polarisation curves for Nb3$^+$ etched CrN/NbN coatings, hard chrome and 304L stainless steel material.
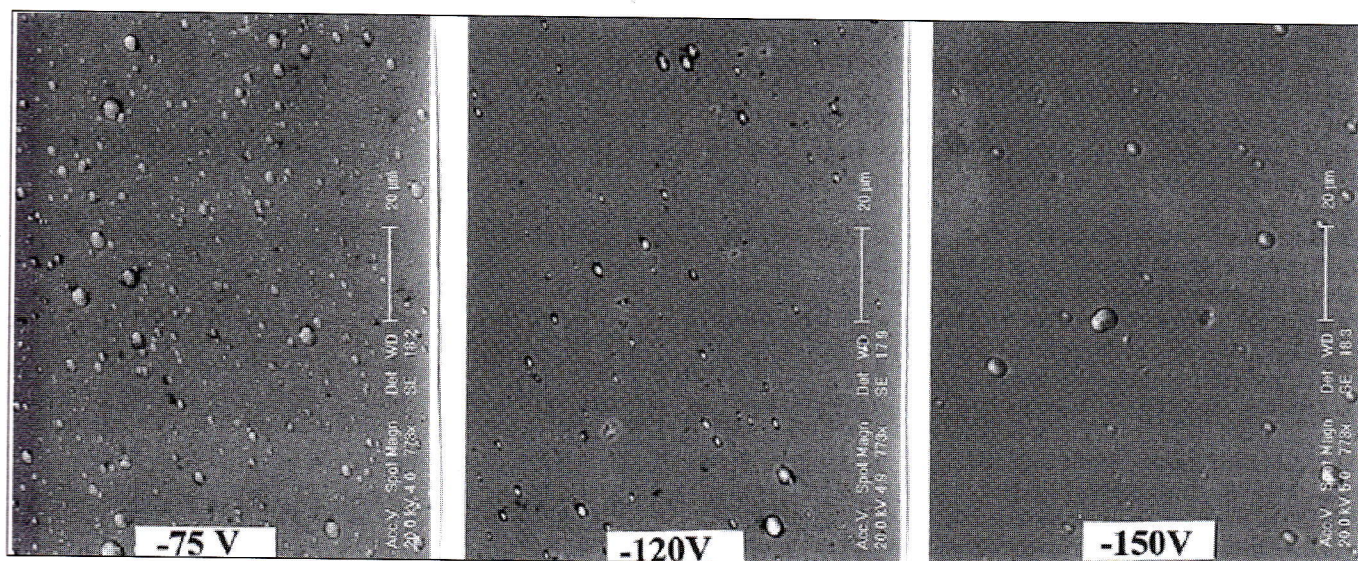

Fig. 5: SEM images of the surface of CrN/NbN superlattice coating deposited at various bias voltages.
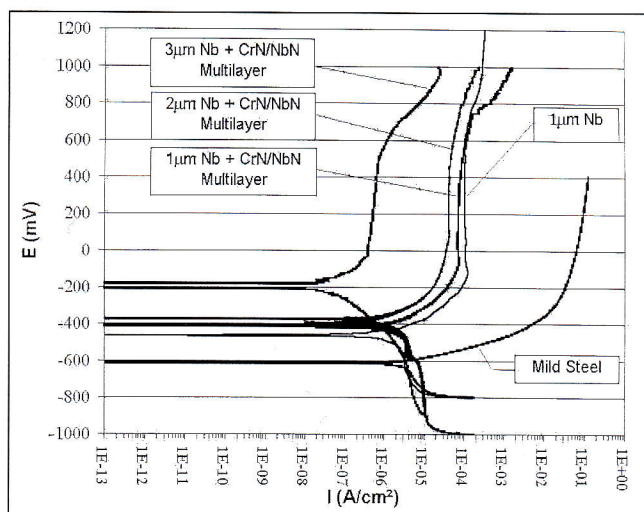
Fig. 6 summarises representative polarisation data for un-coated mild steel, Nb coated 304L stainless steel and mild steel protected with CrN/NbN superlattice coating deposited on Nb base layer.

Fig. 7 Salt spray test: T = 35°C, Humidity: 98%, Solution concentration: NaCl 50 g/l, Ph= 6.5-7.2:
A-4.5 μm CrN/NbN on mild steel after 160 hours, B- 25μm electroplated hard chrome on mild steel, after 200 hours, C- 3 μm Nb- 0.3 μm CrN -4.5 μm CrN/NbN on mild steel after 300 hours.

led that the coating was passive over a wide range of potentials with no evidence of pitting up to +800 mV. At higher potentials a smooth increase of the corrosion current is observed resulting from the Cr dissolution, Fig.6. The corrosion current density is in the range of <5. 10^{-7} A cm^{-2}, which is two orders of magnitude lower than that of Nb coating tested in the same conditions and more than 5 orders of magnitude lower than that of mild steel. For comparison the polarisation curve in the same electrolyte of the electroplated hard Cr has a similar to that of the Nb based multilayer / superlattice coating shape, however shifted to the higher corrosion current density range of 10^{-5} A cm^{-2} (see Fig. 4).
Salt spray tests were carried out to investigate further the corrosion protection properties of the multilayer/ superlattice Nb-CrN-CrN/NbN system. The life- time of 25 μm thick electroplated hard chrome when directly deposited on mild steel in salt spray test was estimated to be approximately 200 hours. As shown in Fig. 7, only localised damage of the Cr coated surface can be observed after this exposure time. In contrast for the CrN/NbN superlattice coating deposited directly on mild steel almost 100% of the surface was corroded after 160 hours with first sign of corrosion after only 24 hours. The Nb based, Nb-CrN-CrN/NbN coating, 7.8 μm thick, deposited on mild steel provided reliable protection against salt spray corrosion for 300 hours with much less corrosion damage of the surface compared to hard chrome. Taking into account the higher wear resistance of the Nb base multilayer/superlattice coating it becomes clear that it is feasible electroplated hard chrome to be partially replaced by more environmentally friendly and significantly thinner PVD coating in some special applications.

CONCLUSIONS

Stainless steel and mild steel can reasonably be protected against wear and corrosion by the deposition of a multilayer/ superlattice Nb based PVD coating using the combined steered cathodic arc/ unbalanced magnetron deposition method. In case of the mild steel, the architecture of such coating
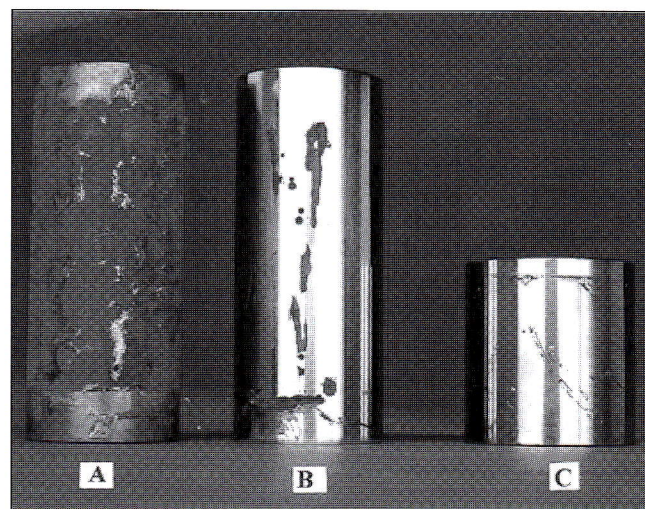
comprises of a Nb implanted zone produced by Nb3+ ion etching, followed by a 1-3μm UBM deposited Nb barrier layer, a 300nm CrN base layer and finally a 4.5 μm thick CrN/NbN superlattice main coating.
The Nb-CrN-CrN/NbN multilayer, outperforms 25 μm thick electroplated hard chrome by factor of 8-10 in dry sliding wear tests. In salt spray tests, depending on the thickness of the Nb barrier layer, the PVD coating shows a life-time of 230 –300 hours, compared to 200 hours for hard chrome.
The barrier Nb layer can be omitted, when more corrosion resistant substrate material such as stainless steel needs to be protected. In salt spray tests the life-time of stainless steel coated with CrN/NbN superlattice coating exceeds 1500 hours, compared to 1000 hours for hard chrome.

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A B S T R A C T

PROPRIETÀ DI RIVESTIMENTI PVD A BASE DI NIOBIO RESISTENTI A CORROSIONE E USURA DEPOSITATI SU DIVERSI ACCIAI

Il niobio è ben noto per le sue eccellenti proprietà di resistenza della corrosione. Per sfruttare i vantaggi di questo materiale sono stati abbinati niobio e cromio per creare rivestimenti PVD con elevata durezza plastica da 45 a 60 GPa.
Per minimizzare il ben noto il problema tipico dei rivestimenti PVD è stato sviluppato un processo di rivestimento a più stadi caratterizzato dalle seguenti fasi di deposizione: impianto a bassa energia di uno ione Nb3+ seguito da deposizione di uno strato barriera di Nb; strato a base di CrN e rivestimento in superlattice di CrN/NbN.

L'impianto di ione è stato effettuato usando un plasma catodico ad arco diretto come fonte di ione metallico, mentre i rivestimenti PVD sono stati creati mediante unbalanced magnetron sputtering. I rivestimenti di superlattice sono stati preparati in un dispositivo di rivestimento a induzione PVD. Le prove di polarizzazione potenziodinamica in cloro contenente media acquosi hanno dimostrato densità di correnti di corrosione per l'acciaio inossidabile rivestito anche per i campioni rivestiti di acciaio molto dolce del tenore di 10-6 A.cm-2 e potenziali di vaiolatura superiori a +600mV. Nelle prove con spray di sale i campioni rivestiti di acciaio dolce hanno mostrano il primo segno di danneggiamento da corrosione dopo 300 h rispetto alle 200 h di un a cromatura di 25 µm elettrodeposti. La resistenza all'usura si è rivelata dieci volte superiore a quella della cromatura.