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The effect of nitrogen pressure on cathodic arc deposited NbN thin films

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ABSTRACT

NbN thin films were deposited on non-standard grade high speed steel (HSS) (79.90 wt.% Fe, 0.71 wt.% C, 6.09 wt.% W, 4.52 wt.% Mo, 3.95 wt.% Cr, 1.82 wt.% Co, 1.75 wt.% V and a hardness of 65 HRC) using cathodic arc deposition at 0.125, 0.5, 1.0 and 1.5 Pa nitrogen pressures (P_{N2}), with a bias voltage of −150 V. X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Nanoindentation and Rockwell C analysis were used to characterize the thin films in order to identify the NbN phases and to investigate the influence of P_{N2} on mechanical properties. Hexagonal β-Nb_2N, ε-NbN and δ′-$NbN_{0.95}$ are identified in XRD analysis. Hardness values derived by nanoindentation technique are 20 GPa for β-Nb_2N, ε-NbN and 40 GPa for δ'-$NbN_{0.95}$. Due to the complexity of phase system special attention was focused on identification of NbN phases by deconvolating the XRD peaks especially at 0.5 Pa in which both ε-NbN and β-Nb_2N were found. Rockwell C analysis revealed that the film adhesion is found to be poor at lower P_{N2}, due to the brittle nature of β-Nb_2N.

1. Introduction

Niobium nitride has attracted serious attention, not only with its superconducting properties but also with its mechanical properties where wear resistance is necessary. In the last decade, studies that are focused on NbN showed that this NaCl type compound's superconducting properties are strongly related to its crystal structure and chemical composition [1–3]. Like many other nitrides, NbN coatings also have high hardness and toughness which make them a good choice for mechanical applications. Moreover as a ceramic coating, thin film NbN has good thermal expansion coefficient that matches with widely used tool steels and this enhances its utility for wear resistant purposes. It was also reported that the mechanical properties of NbN thin films are influenced by their crystallographic structure and chemical composition [4–9].

Although there are many studies on Nb–N system, there are also still arguments on NbN's structural properties due to the complexity of Nb–N system. Different phases in the Nb–N system (Fig. 1) may be listed as [8,10,11]: β-Nb_2N (hexagonal), γ-Nb_4N_3 (tetragonal), δ-NbN (cubic), δ′-NbN (hexagonal), ε-NbN (hexagonal), η-NbN (hexagonal) and α-Nb(N), a cubic solid solution formed by dissolving N_2 in Nb metal. In thin films, occurrences of these phases are related with the PVD deposition system's type, configuration and deposition parameters. In this aspect, formation and structural properties of Nb–N phases are mainly influenced by P_{N2} and bias voltage applied during deposition process [8,9,12–16].

In previous studies, several PVD techniques are reported for NbN thin film deposition which are: reactive magnetron sputtering [12,13,17–24], arc PVD [8,9,14,15,25], ion beam assisted processes [26–28] and pulsed laser deposition [29]. Specifically when arc deposition system is focused, it is clear that the deposition systems' configuration affects the structure by changing film growth mechanism. In this aspect, Zhitomirsky et al. [8,9], Bendavid et al. [15] and Cansever [30] studied NbN thin films deposited with different parameters in arc PVD systems and their results are discussed comparing the coating deposited at lower P_{N2}.

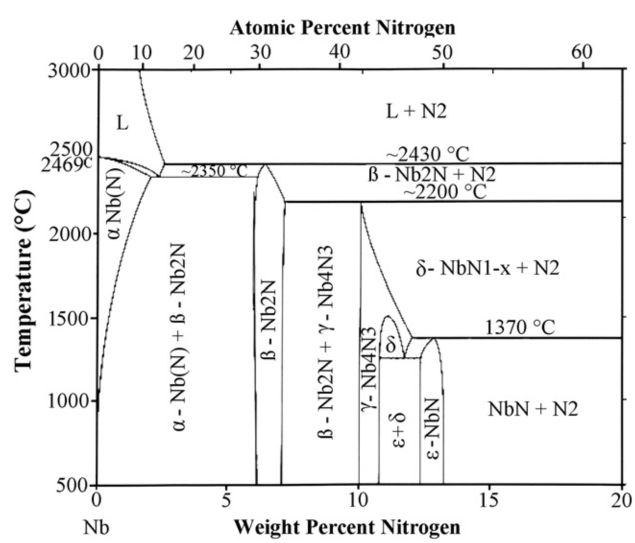

Fig. 1. Nb–N phase diagram [11,8].

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Table 1
Deposition parameters that are used for NbN deposition on HSS substrates

P_{N2} (Pa)	N_2 flow rate (sccm)	Arc current (A)
0.125	17.5	130
0.5	13	140
1.0	35	100
1.5	55	140

Zhitomirsky [9] investigated the structure and properties of NbN coatings deposited by vacuum arc deposition (VAD) systems equipped with and without magnetic plasma guiding. Author reported the HV hardness of NbN coatings deposited by without magnetically guided plasma system as 34–36 GPa at 0.13 Pa and with magnetic plasma guiding as 42 GPa at 0.4 Pa. Moreover for both systems the hardest coatings were consisted of β-Nb_2N with brittle structure.

Bendavid et al. [15] also studied Nb–N by changing the bias voltages and P_{N2} systematically in a filtered vacuum cathodic arc deposition system. At 0.4 Pa, the author reported a mixture of cubic α-phase and hexagonal δ'-NbN phases with 47 GPa hardness by measuring with a Berkovich indenter equipped ultra-microindentation system.

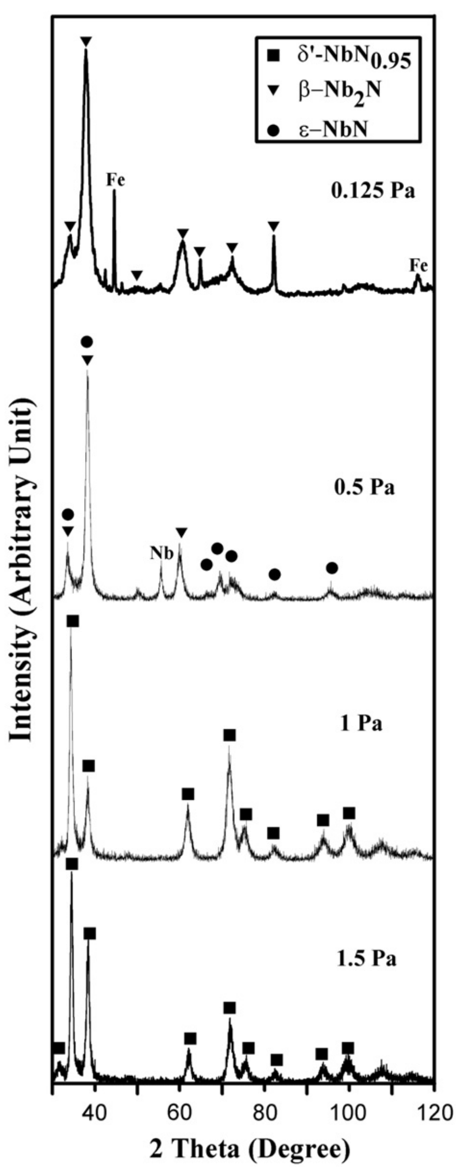

Fig. 2. XRD patterns of NbN thin films deposited at different P_{N2} with −150 V bias voltage.

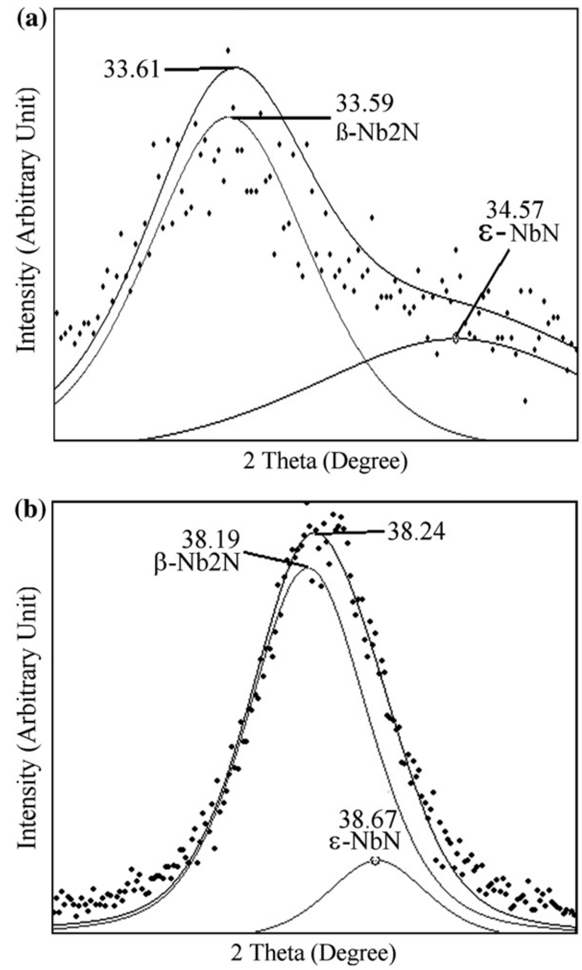

Fig. 3. Deconvolated peaks of the sample deposited at 0.5 Pa P_{N2}. a) $2\theta=33.61°$ b) $2\theta=38.24°$.

In this paper, the influence of the P_{N2} on the phase composition of the cathodic vacuum arc deposited Nb–N coatings, their microstructure, morphology, nanohardness and Young's modulus was investigated by following deposition processes at 0.125, 0.5, 1.0 and 1.5 Pa P_{N2} with −150 V bias voltage.

2. Experimental details

NbN thin films were deposited on 30 mm × 10 mm HSS substrates by cathodic arc deposition system (Novatec-SIE, Model:NVT-12). The surfaces of the HSS substrates were prepared by standard metallographic techniques and the final polishing was done with 1 μm diamond paste. After polishing, the specimens were ultrasonically cleaned in acetone and alcohol bath. In order to deposit NbN thin films substrates were mounted on a stationary holder opposite to the cathode in a deposition system where DC arc discharge was used for thin film

Table 2
Experimental results of NbN coating procedures

P_{N2} (Pa)	Phase	*a* (Å)	*c* (Å)	*c*/*a*	Apparent grain size (nm)	Thickness (μm)
0.125	β-Nb_2N (*Experimental*)	3.066	4.945	1.61	17	3.2
	β-Nb_2N (40-1274)	3.055	4.948	1.62		
0.5	ε-NbN (*Experimental*)	2.980	11.303	3.79	10	3.1
	ε-NbN (20-0801)	2.960	11.270	3.81		
1.0	δ′-NbN (*Experimental*)	2.930	5.619	1.91	17	3.1
	δ′-NbN (25-1361)	2.968	5.535	1.86		
1.5	δ′-NbN (*Experimental*)	2.985	5.465	1.83	12	5.5
	δ′-NbN (25-1361)	2.968	5.535	1.86		