CHAPTER 6

CONCLUSIONS

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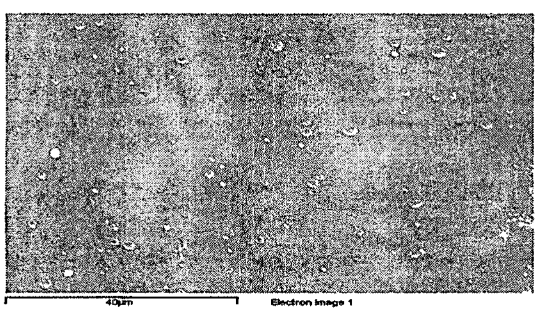
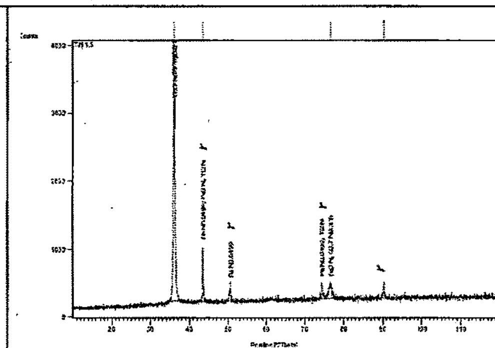
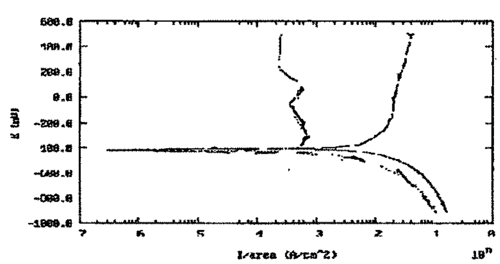
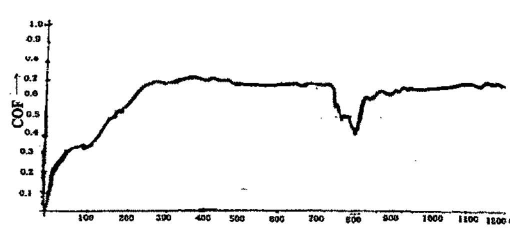
The detailed and complete characterisation of thin films requires many sophisticated instruments and a lot of expertise. The quality of thin films of particular thickness and particular composition depends on the process parameters employed. It is very difficult to optimize different parameters and achieve the required quality of film. As discussed in the previous chapter (Results and discussion) even after taking sufficient care, a lot of unexpected defects are observed due to variation in different parameters.

Here an attempt has been made to characterize each and every parameter and defects observed during whole span of research work using the available characterization technique. The explanation is given in a simple graphical representation. Using these monograms one can easily identify/characterize the given quality of thin films obtained with different parametric conditions.

6.1Characterization of thin films containing Macroparticle:

	
A 1.SEM Image of film containing macro particle	B. XRD Image of coating consisting of macro particle
	
C Potentiodynamic plot for thin film consisting of macroparticle	D Coefficient of friction of thin film consisting of macroparticle.

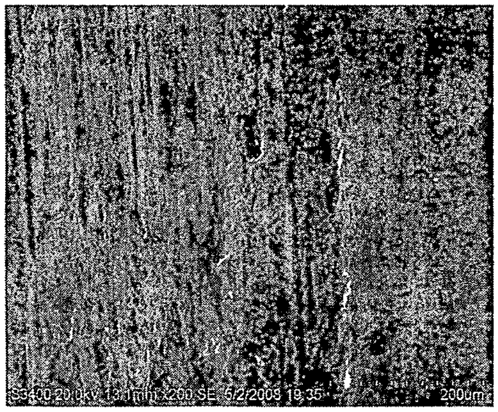
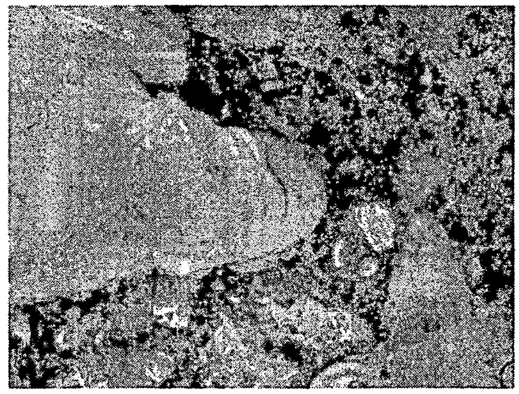
	
E SEM of wear track at low magnification	F.SEM of wear debris at high magnification

Fig 6.1 (A-F) characterizations for macroparticle within the thin film

SEM image 6.1.A indicates presence of large globules(macroparticle) embedded within the film.The XRD 6.1.B consist of sharp high intensity peak corresponding to composition of macroparticle.

Corrosion Behaviour indicates presence of horizontal peak in the anodic region of the curve. Fig 6.1.C this peak corresponds to the presence of additional phase which has different corrosion behavior than coating.

The COF vs. time curve 6.1.D shows that the friction may remain at its initial value for some time and slowly increase to steady-state value .The curve behaviour indicates that increase is associated with ploughing because of roughening and trapped wear particles. The SEM 6.1.E of wear surface indicates that deep grooves and scaly detachments of coating material. The damage pattern indicates that plastic deformation occurred to`some extent via ploughing and wedge formation.

6.2 Presence of Single phase of one orientation within thin film coating

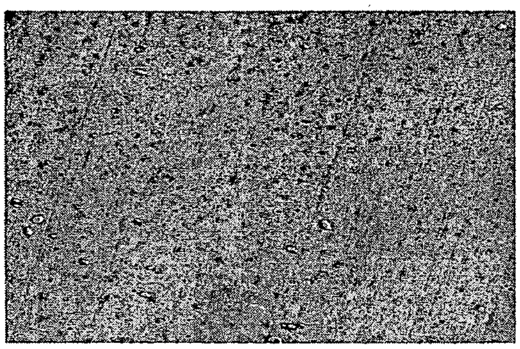
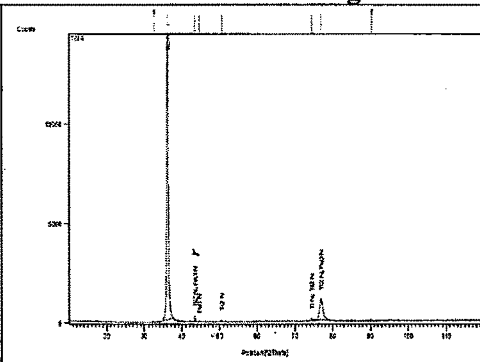
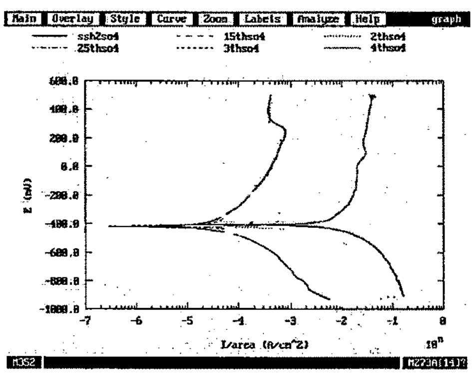
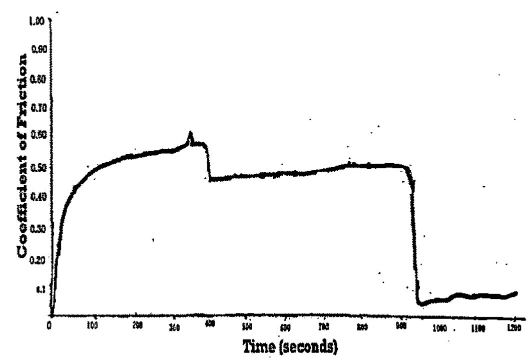
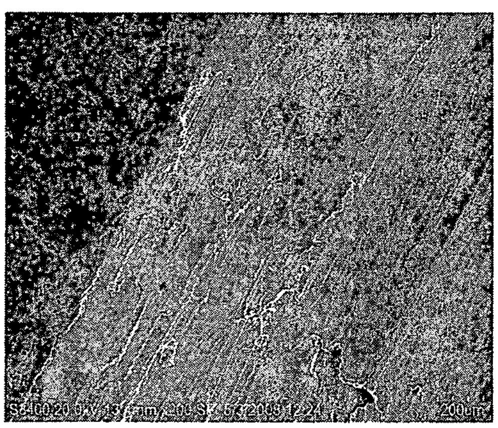
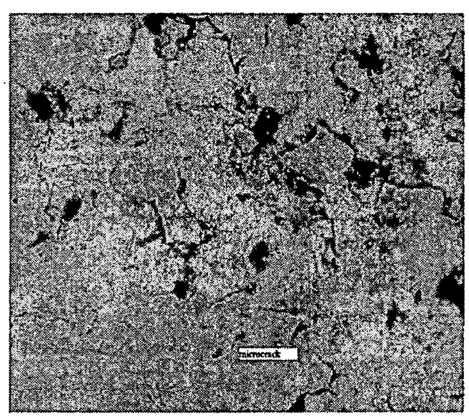
	
A SEM Image of film having single phase	B XRD Image of coating consisting of single phase
	
C. Potentiodynamic plot for thin film consisting of macro particle	D.Coefficient of friction of thin film consisting of macroparticle.
	
E SEM of wear track at low magnification	F SEM of wear debris at high magnification

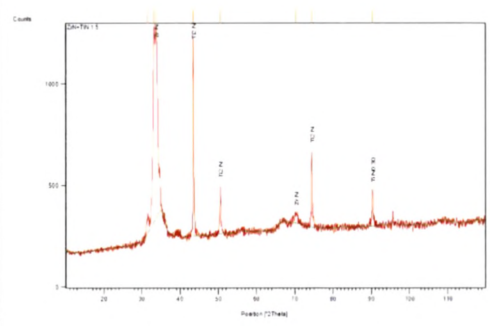
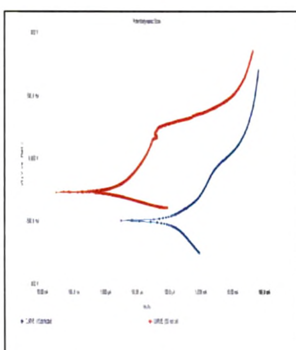
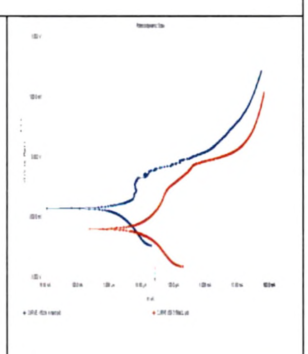
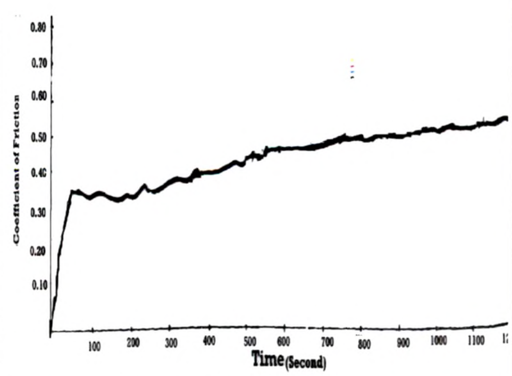
Fig 6.2 (A-F) Characterization for single phase of one orientation in thin film

SEM image 6.2 A. indicates very less amount of globules (macroparticle) embedded within the film. The XRD 6.2 B. consists of sharp high intensity peak corresponding to composition of coating. Peaks corresponding to presence of any other phases are absent. Corrosion behaviour 6.2 C indicates large difference in icorr especially in the anodic region implying good corrosion resistance.

The COF vs. time curve 6.2 D shows that the drop in the coefficient of friction which is associated with smoothing of the two hard surfaces experiencing plastic deformation, resulting in a drop in the ploughing component of the friction.

The SEM of wear surface 6.2 E &F indicates presence of cracks and smooth surface and absence of wear debris.

6.3Presence of pores/insufficient coverage in thin film

	
A SEM Image of film with insufficient coverage	B XRD Image of coating with insufficient coverage.
 (i)0.1N HCl  (ii)3.5%NaCl	
C Potentiodynamic plot for thin film incomplete coverage in (a) 0.1NHCl & (b) 3.5%NaCl	D Coefficient of friction of thin film of incomplete coverage

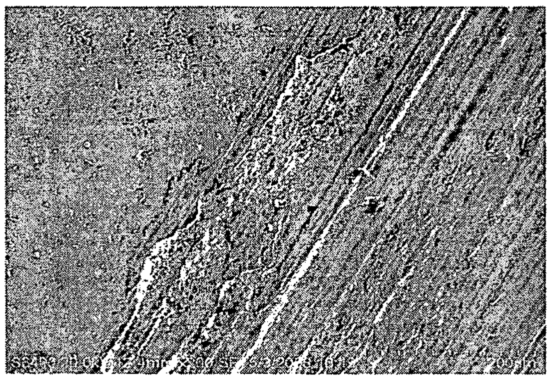
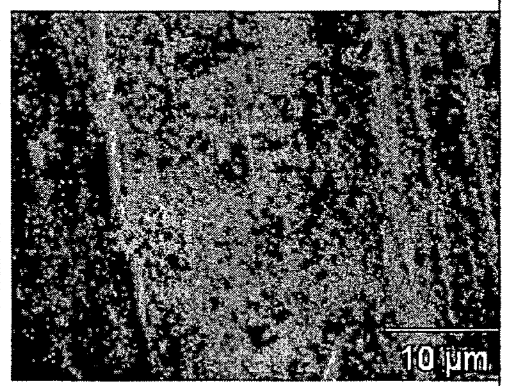
	
E SEM of wear track at low magnification for 1.5µZrTiN	F.SEM of wear debris at high magnification for 1.5µZrTiN

Fig 6.3 (A-F)Characterization for pores/insufficient coverage of thin film

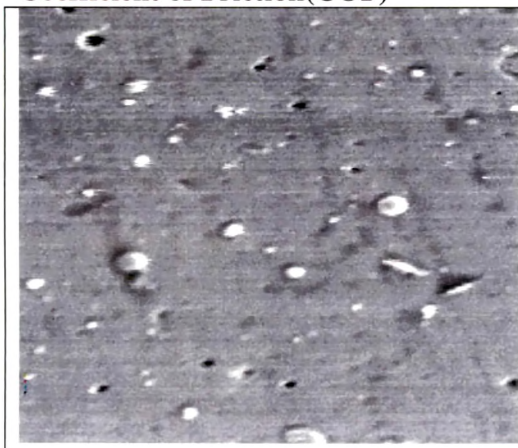
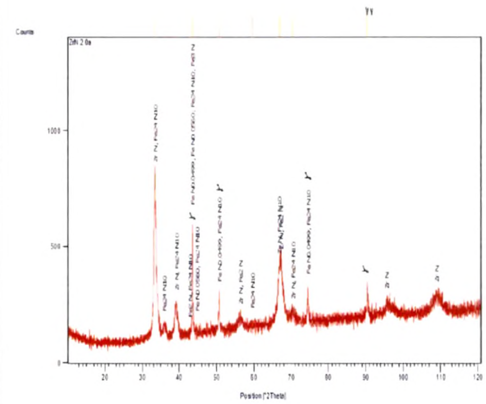
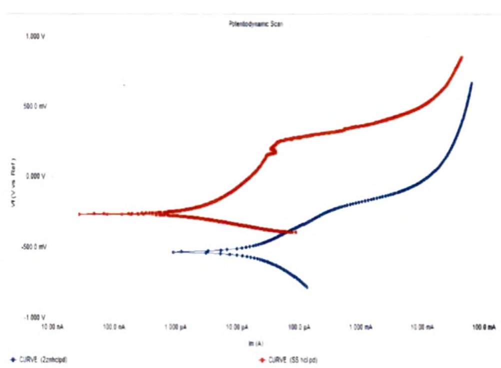
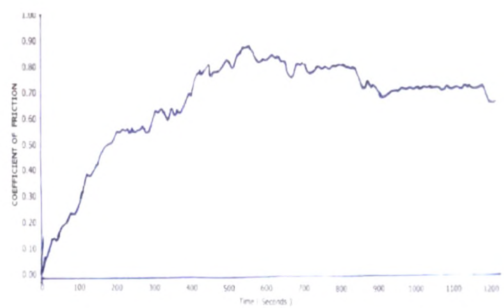
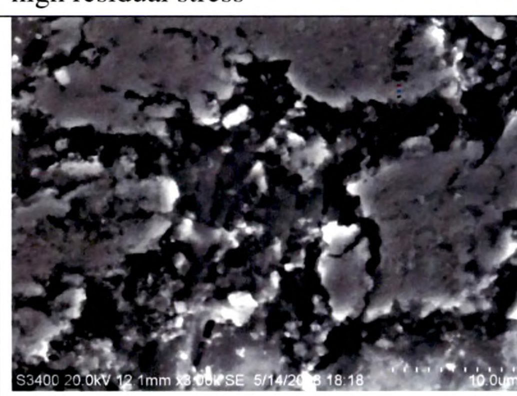
SEM image 6.3 A indicates very less amount of globules (macroparticle) embedded within the film. The XRD 6.3.B consist of sharp high intensity peak (large grain size) corresponding to substrate in addition to peak of coating composition.

In 6.3 C a shift in curve to lower Ecorr and Icorr is observed, when environment is changed. The protection obtained by coating is less than uncoated sample in case of 0.1N HCl however in case of 3.5% NaCl, protection is more than uncoated, indicating effect of environment on corrosion behaviour.

The COF vs. time 6.3.D curve shows that the drop in the coefficient of friction is associated with smoothing of the two hard surfaces experiencing plastic deformation, resulting in a drop in the ploughing component of the friction similar to that of coatings consisting of macroparticles. The SEM of wear surface 6.3.E & F indicates grooved surface and at high magnification, wear debris remained within the track.

Comparing the behaviour of coating consisting of macroparticle and coatings with incomplete coverage, the friction behaviour of two coatings remained almost same, however the SEM micrographs of wear track indicates presence of debris within the wear track which were absent in 6.1.

6.4Characterization of thin films under High Residual stress and having very High Coefficient of Friction(COF)

	
A SEM Image of film under high residual stress	B XRD Image of coating under high residual stress
	
C Potentiodynamic plot for thin film under high residual stress	D Coefficient of friction of thin film under high residual stress
	
E SEM of wear track at low magnification of 2.0μZrN	F SEM of wear debris at high magnification of 2.0μZrN

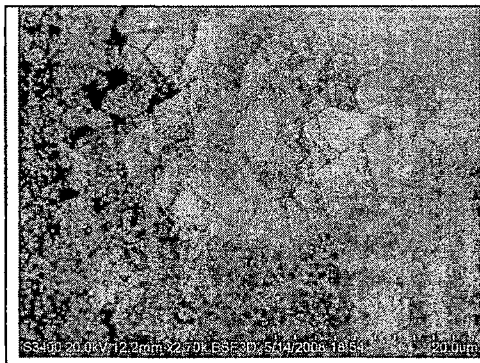
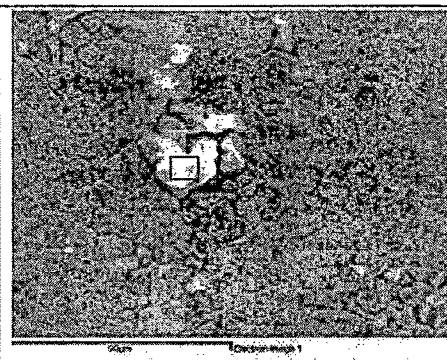
	
G SEM BSE at 3000X for 2.0µZrN	H SEM image after potentiodynamic test of 2.0µZrN

Fig 6.4 (A-H)Characterization for high residual stress and high coefficient of friction in thin film

SEM image 6.4 A indicates presence of small globules (macroparticle typical of cathode arc evaporation process) embedded within the film. The XRD 6.3 B consist of broad peaks indicating increase in stresses within the film.

Fig 6.3 C Anodic current density changes a little with potential increasing potential in certain range. In this interval, the passive layer protects specimen surface from dissolving. While, after reaching certain potential anodic current density increases dramatically with potential, probably due to a pitting corrosion mechanism initiated at the local defects of the film. The corrosion resistance of coated steel is inferior to uncoated indicating the effect of residual stress on corrosion resistance in particular environment.

The COF vs. time curve 6.4 D shows that the friction may remain at its initial value for some time and slowly increase to another steady-state value and with further increase in time the fluctuations in curve is observed. An increase in the friction coefficient values at the beginning of the test suggest cohesive failure of coating, due to the contact pressure applied by the substrate resulting in increased amount of debris.

The SEM of wear surface 6.4.E indicates that deep grooves and scaly detachments of coating material were observed in SEM. The damage pattern 6.4.F indicates complete delamination of coating.

Back scattered SEM 6.4.G after wear test shows that the coating contains a series of fine micro cracks with a honeycomb structure, which could be the result of thermal stress differences between the substrate and the coating upon cooling down from its high deposition temperature. SEM image 6.4.F after potentiodynamic test in 1N H_2SO_4 indicates presence of intergranular cracks indicating high residual stress within coating.

6.5 Characterization of thin films which exhibits Pitting corrosion.

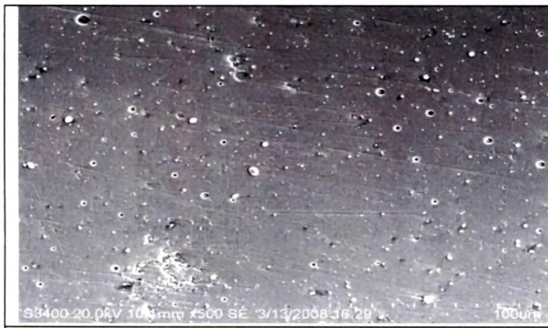
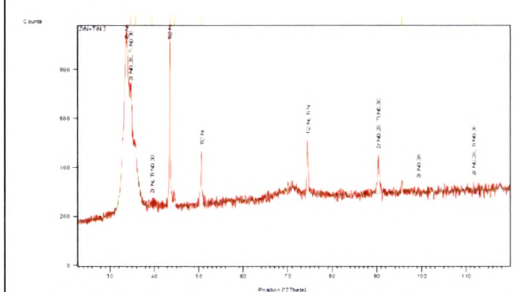
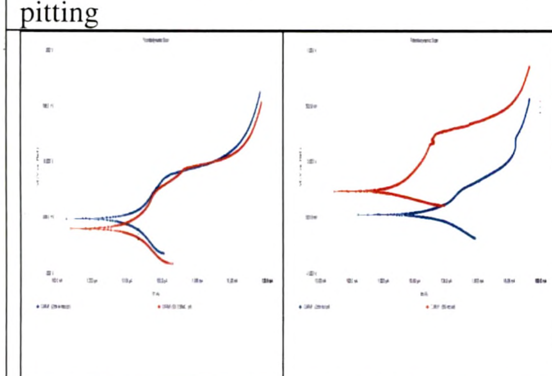
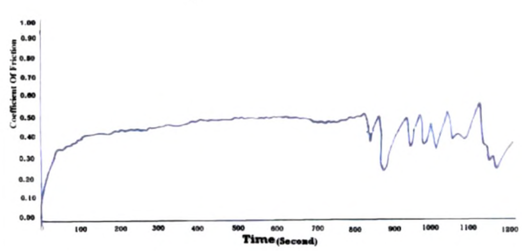
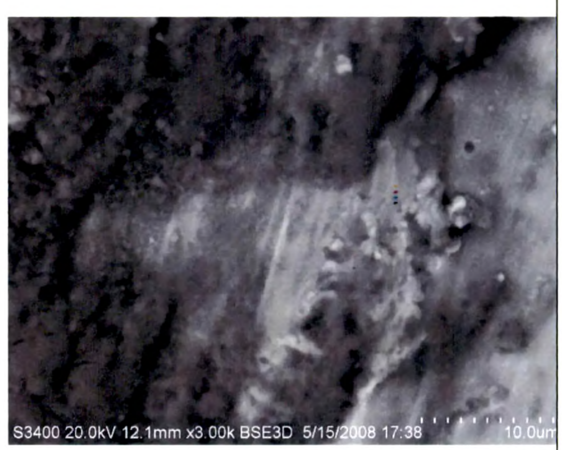
	
A SEM Image of film which exhibits pitting	B XRD Image of coating which exhibits pitting
 3.5%NaCl / 0.1N HCl	
C Potentiodynamic plot for thin film exhibiting pitting	D Variation in Coefficient of friction with time of thin film which exhibits Pitting corrosion
	
E SEM of wear track at low magnification of 2μ ZrTiN	F SEM of wear debris at high magnification of 2μ ZrTiN

Fig 6.5(A-F) Characterization for thin films exhibiting Pitting corrosion

SEM image 6.5.A indicates presence of small globules (macroparticle typical of cathode arc evaporation process) embedded within the film .Some irregularity is observed on the surface of coating. The XRD 6.5.B consists of broad peaks indicating increase in stresses within the film. The 100% intense peak of the substrate is observed.

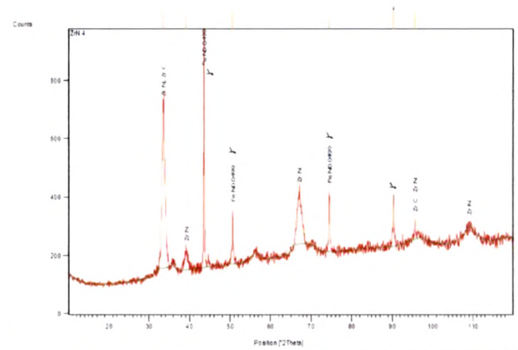
Fig 6.5 C Anodic current density changes a little with potential increasing potential in certain range. In this interval, the passive layer protects specimen surface from dissolving. While, after reaching certain potential anodic current density increases dramatically with potential, probably due to a pitting corrosion mechanism initiated at the local defects of the film.

The COF vs. time curve 6.5 D shows that the increase in COF is associated with ploughing because of roughening and trapped wear particles. The COF remained steady up to 800 sec. The drop in the friction in the later stages of test is associated with ejection of wear particles, and a subsequent increase is associated with generation and entrapment of wear particles

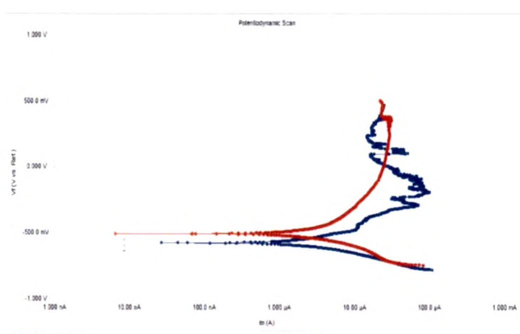
The SEM of wear surface 6.5 E indicates that deep grooves on surface of coating material .At high magnification 6.5 F pores, irregular shaped particles and crack parallel to direction of wear track were observed.

6.6Characterization of thin films which exhibits Incomplete Passivity

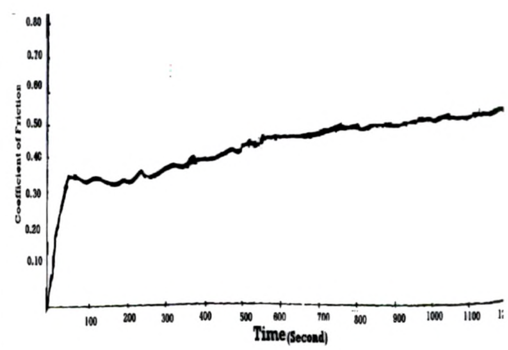
A. SEM Image of film which exhibits exhibits Incomplete Passivity



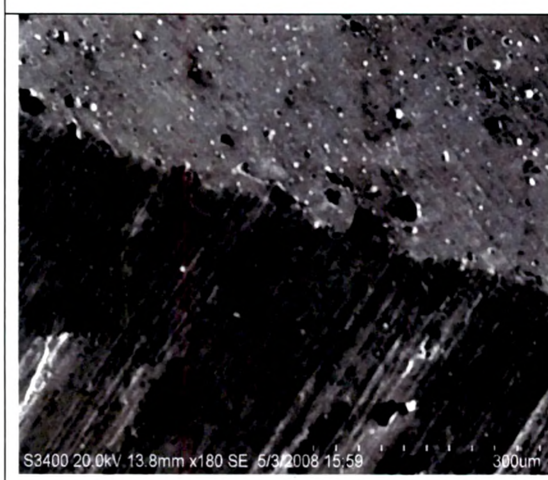
B XRD Image of coating which exhibits exhibits Incomplete Passivity



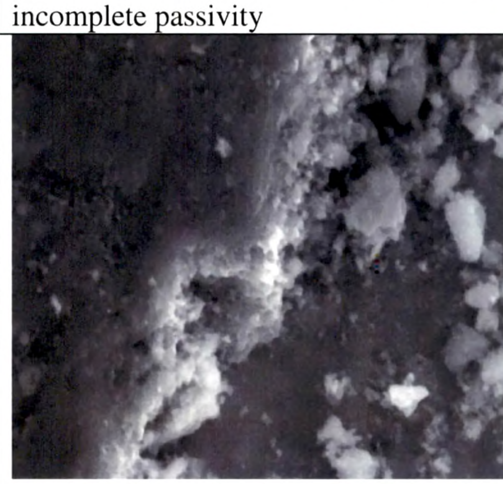
C Potentiodynamic plot for thin film which exhibits incomplete passivity



D Variation in Coefficient of friction with time of thin film which exhibits incomplete passivity



E. SEM of wear track at low magnification of 4μ ZrN



F.SEM of wear debris at high magnification of 4μ ZrN

Fig 6.6(A-F) Characterization of thin films exhibiting incomplete passivity.

SEM image 6.6A indicates presence of small globules (macroparticle typical of cathode arc evaporation process) embedded within the film. Removal of macroparticles from the surface is observed on the coating.

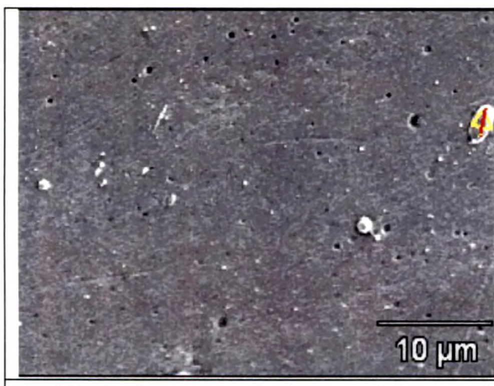
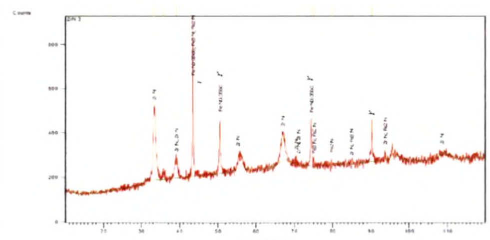
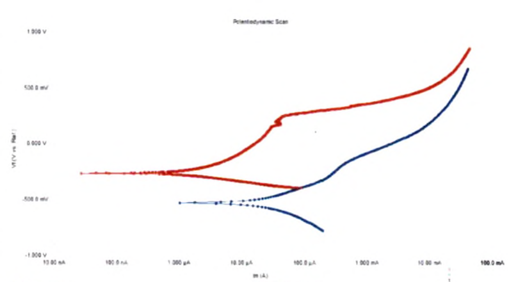
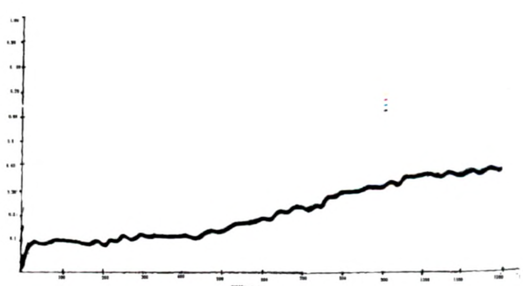
The XRD 6.6. B consists of peaks of phases with varying orientation and size of each phase is less than 10 A^0. The 100% intense peak of the substrate is observed.

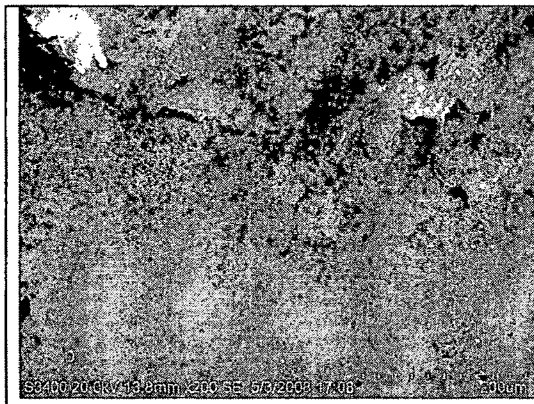
Corrosion Behaviour 6.6 C indicates presence oscillations in the anodic region indicating region of incomplete passivity

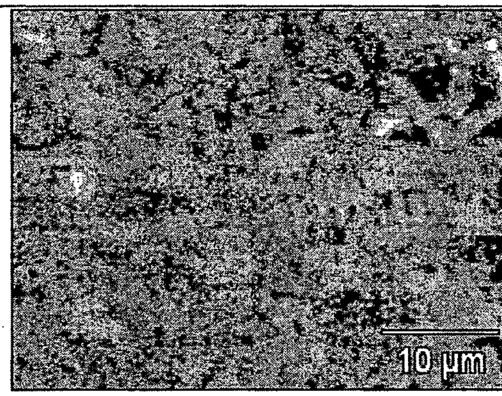
The COF vs. time curve 6.6 D shows that the increase is associated with ploughing because of roughening and trapped wear particles.

The SEM of wear surface 6.6. E indicates that deep grooves on surface of coating material. At high magnification 6.6. F wear debris of irregular size are observed at the interface of coating and wear track.

6.7 Characterization of thin films which exhibits continous increase in COF. (3ZN))

	
A.SEM Image of film which exhibits continous increase in COF	B. XRD Image of coating which exhibits which exhibits continous increase in COF.
	
C Potentiodynamic plot for thin film which exhibits continous increase in COF.	D Variation in Coefficient of friction with time of thin film which exhibits which exhibits continuous increase in COF.





E SEM of wear track at low magnification of 3µ ZrTiN	F.SEM of wear debris at high magnification of 3µ ZrTiN

Fig 6.7 (A-F) Characterisation of thin films exhibiting continuous increase in COF

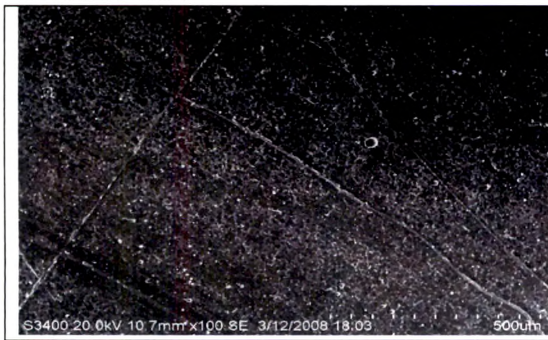
SEM image 6.7.A indicates presence of small globules (macroparticle typical of cathode arc evaporation process) embedded within the film .However quantity of macroparticles is very less. XRD 6.7.B consist of peaks of many phases indicating anisotropic characteristic of coating. The 100% intense peak of the substrate is observed.

For all the values of Potential and current 6.7.C the corrosion behaviour of coating is always inferior to the uncoated stainless steel.

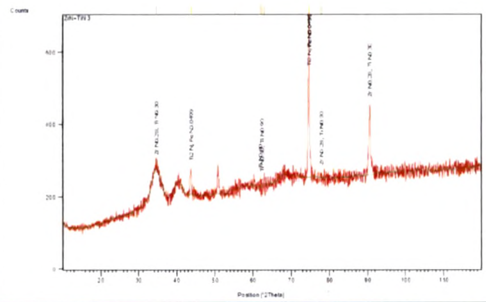
The SEM micrograph indicates less amount of macroparticle hence initially COF as indicated in COF vs time curve 6.7.D is less.

SEM image indicates 6.7.E deep mechanical plowing grooves and large scratched appearance on the substrate after wear test which results in increase in COF. The wear track covered with compacted debris is observed at high magnification. 6.7.F

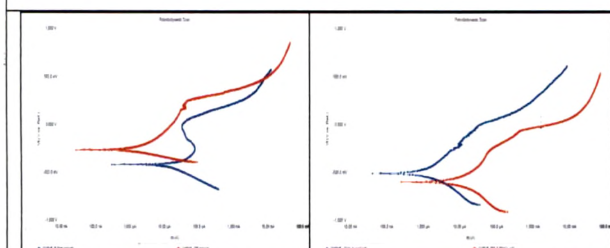
6.9 Characterization of thin films which exhibits phase consisting of solid solution of ZrN and TiN



A.SEM Image of film which exhibits Solid Solution (more than 2 Phases)

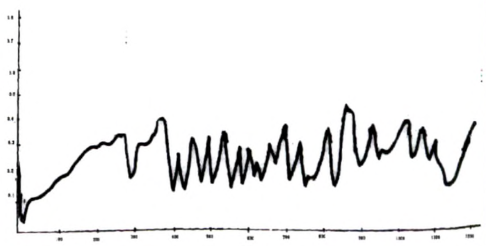


B.XRD Image of coating which exhibits Solid Solution (more than 2 Phases)

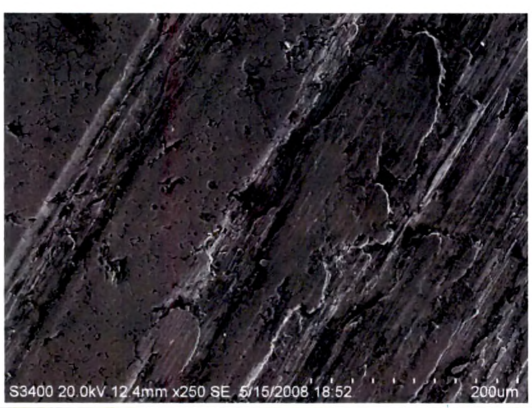


(a)0.1N HCl	(b)3.5%NaCl

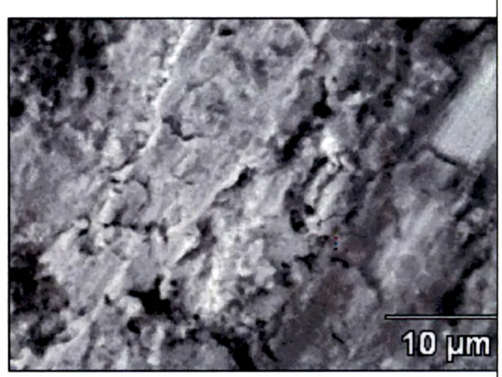
C Potentiodynamic plot for thin film having Solid Solution (more than 2 Phases)



D .Variation in Coefficient of friction with time of thin film having solid Solution (more than 2 Phases)



E. SEM of wear track at low magnification



F.SEM of wear debris at high magnification

Fig 6.8 (A-F) Characteristic of ZrTiN thin film which exhibits phase consisting of solid solution of ZrN and TiN

SEM image 6.8.A indicates presence of small globules(macroparticle typical of cathode arc evaporation process) embedded within the film .The quantity of macroparticles is very less. The XRD 6.8.B consists of broad peaks indicating increase in stresses within the film. The intense peak corresponding to formation of solid solution is observed.

The characteristic of pitting 6.8 C i.e less increase in voltage with increase in current is observed in two environment used. However extent of protection provided is different.

The COF vs time curve 6.8 D shows that the continuous increase in COF with time. This can be interpreted as increase is associated with combined effect of ploughing, adhesive wear and a generation and entrapment of wear particles.

The SEM of wear surface 6.8.E indicates that deep grooves on surface of coating material. At high magnification 6.8.F pores and crack parallel to direction of wear track were observed. The wear debris are entrapped within the wear track.