STUDIES ON MICRO AND NANOTRIBOLOGICAL BEHAVIOUR OF Ti ALLOYS AND POLYMER

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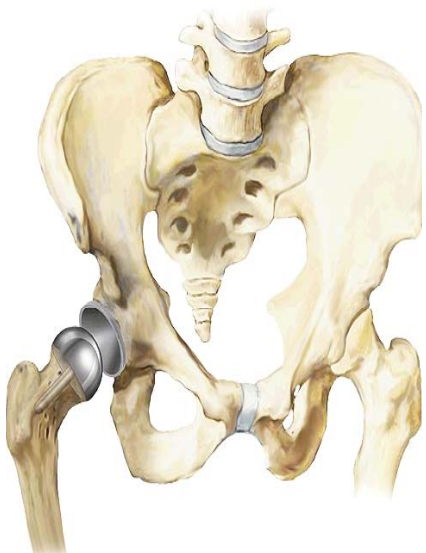
14th September
Trends in Nanotribology
ICTP,Trieste,Italy


VIT University, Vellore India

My presentation will encompass

- Introduction to tribology of prosthesis
- Macro and nanotribology - Current scenario
- Experimental
    - Scratch resistance of Ti and its alloys – from microscopic perspective
    - Nanotribological behavior of Ti and its alloys
- Conclusions

Need to understand the tribology of prosthesis



Life span of the prosthesis 12-15 years

Expected life span – more than 30 years

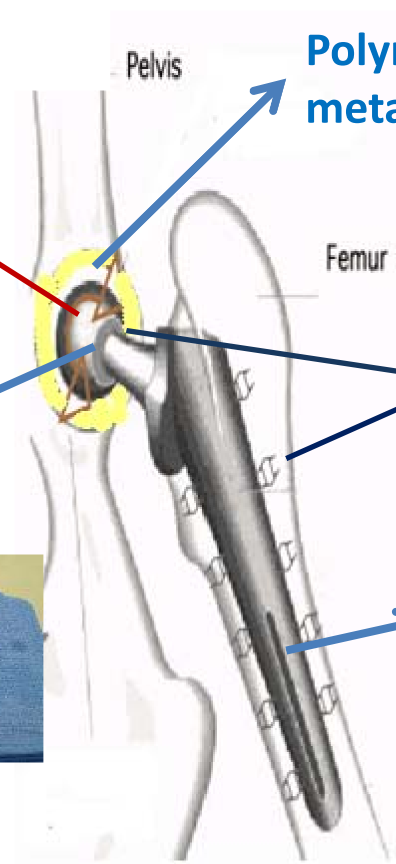
Reasons for short life span

- infections
- corrosion
- **wear**
- lack of complete biocompatibility
- mismatch in modulus of elasticity
- manufacturing defects and surgical procedures

800,000 implants every year in Europe

By 2050, there will be approximately 2 billion elderly people living worldwide.

Tribological contacts in hip joints



Polymer/ceramic/ metallic cup

Sliding wear

Fretting wear

Metallic/ ceramic ball

Metallic stem

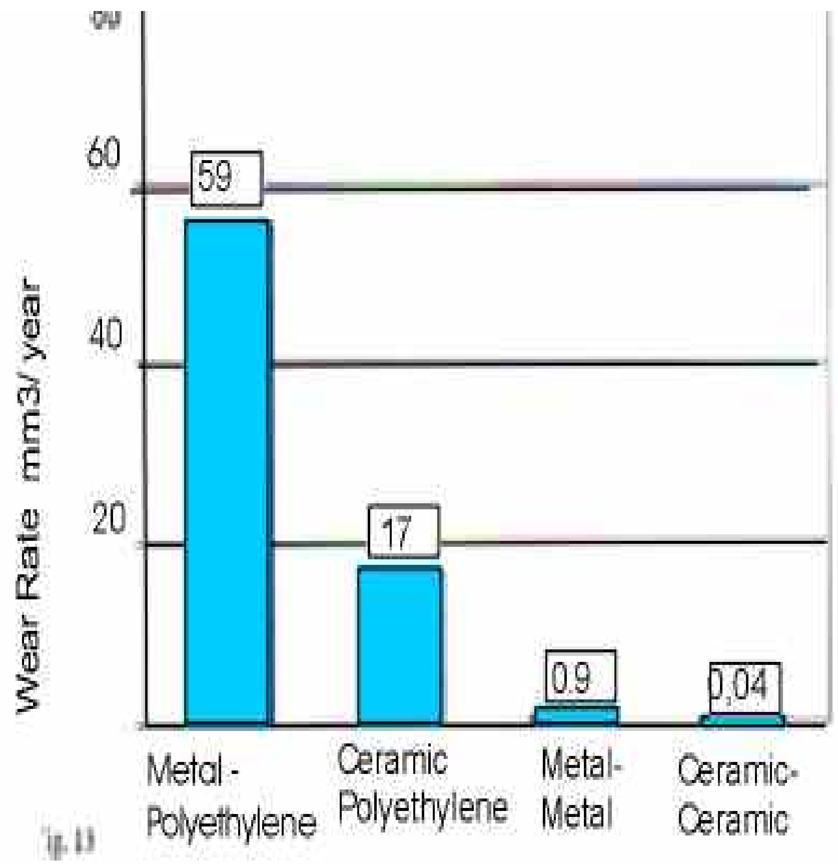
Wear between
Metal-bone
Ball and stem , cup and ball

Materials

Polymer –
- UHMWPE

Ceramics –
- Alumina, Zirconia, TiN coatings on metallic materials

Metals and alloys –
- 316 Stainless steel
- Cobalt chrome
- Ti alloys
  - Ti6Al4V
  - Ti-6Al7Nb
  - **Ti13Nb-13Zr**
  - Ti-XTa-XNb-XZr-O



Ceramic over ceramic –

- installation problem
- Chip formation
- Revision if slight deviation in installation
- Fracture on high impact

excess of 1 million nanopartictles of
with metallic implants
are generated per step

Progress in Materials Science 54 (2009) 397–425

Contents lists available at ScienceDirect

Progress in Materials Science

journal homepage: www.elsevier.com/locate/pmatsci

Ti based biomaterials, the ultimate choice for orthopaedic implants – A review

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A R T I C L E   I N F O

Article history:
Received 28 January 2008
Received in revised form 11 June 2008
Accepted 20 June 2008

Citation index - 120

A B S T R A C T

The field of biomaterials has become a vital area, as these materials can enhance the quality and longevity of human life and the science and technology associated with this field has now led to multi-million dollar business. The paper focuses its attention mainly on titanium-based alloys, even though there exists biomaterials made up of ceramics, polymers and composite materials. The paper discusses the biomechanical compatibility of many metallic materials and it brings out the overall superiority of Ti based alloys, even though it is costlier. As it is well known that a good biomaterial should possess the fundamental properties such as better mechanical and biological compatibility and enhanced wear and corrosion resistance in biological environment, the paper discusses the influence of alloy chemistry, thermomechanical processing and surface condition on these properties. In addition, this paper also discusses in detail the various surface modification techniques to achieve superior biocompatibility, higher wear and corrosion resistance. Overall, an attempt has been made to bring out the current scenario of Ti based materials for biomedical applications.

Understanding of Tribology of Ti alloys – Macroscopic point of view

Ti and its alloys have…

- Low shear strength and low tensile strength- high friction coefficient –
- Low resistance to plastic shearing and low work hardening, and
- Low protection exerted by the surface oxide which may form as a consequence of the high flash temperature induced by frictional heating
- High material transfer due to adhesive wear
- Strong tendency to seize

Nanotribology from a biomaterials perspective

- Understanding the polymer wear using AFM tip –Modeling AFM tip as an single asperity of counter metallic part
- Effect of surface roughness of polymer on wear at different hirecherial levels using microtribometer /AFM
- Nanotribological behavior of Ti and its alloys using -nanotribometer

Experimental Work

Phase 1		
Scratch testing -	Const ./Ramp loading	Cp Ti, Ti13Nb13Zr,Ti-6Al-4V, UHMWPE

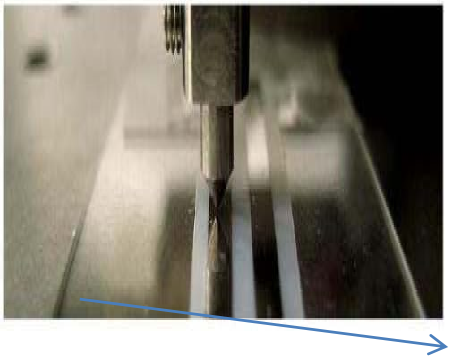
Phase II	
Nanotribological studies	Cp Ti, Ti13Nb13Zr,Ti-6Al-4V

Experimental conditions	
3 Sliding speeds	Dry dry/Simulated body conditions

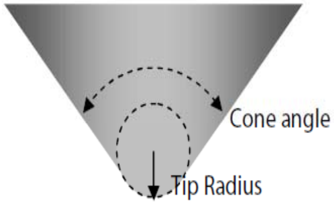
Scratch testing

✓ Rockwell indenter -–C scale

✓ angle (120.Cone)



✓ Tip radius 200µm



✓ **Outcome:** Traction force,coefficient of friction ,critical loads for failure (if coatings are characterized)

Scratch Hardness

- The scratch hardness is calculated by using the following formula,

$$H_s = \frac{8 \times F_n}{\Pi \times b^2} \qquad \text{(in GPa)}$$

where,

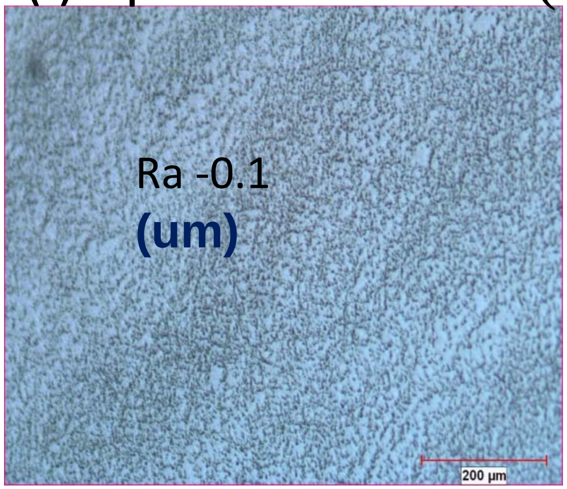
H_s = scratch hardness,

F_n = normal load applied,

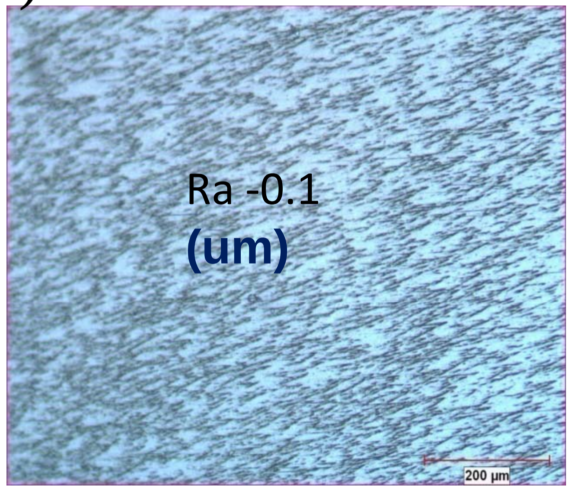
b = scratch width.

Microstructure, Roughness and Hardness

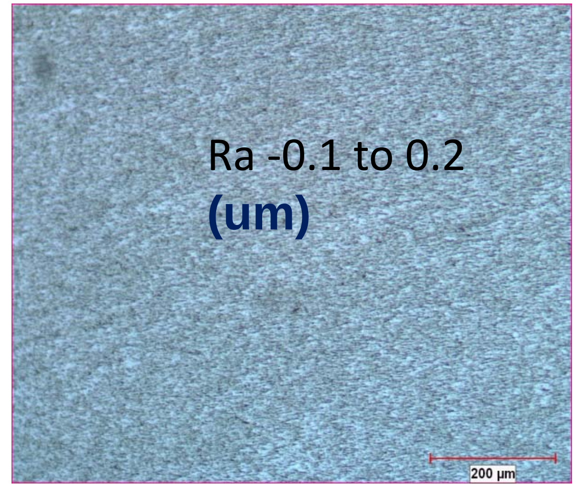
(i) Cp Ti-100x



(ii) Ti-6Al-4V 100x



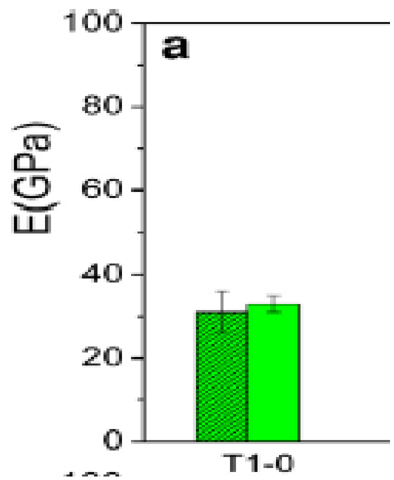
(iii)Ti-13Nb-13Zr -100x

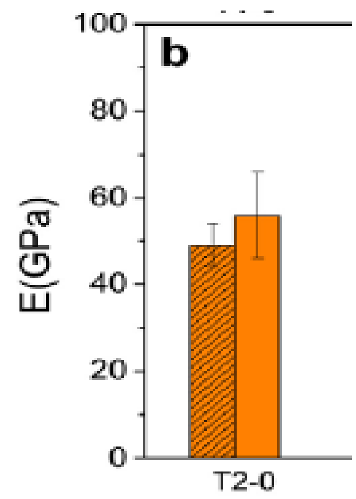


cpTi
Young's Modulus (100 GPa)
Poisson’s Ratio (0.36)

Ti6Al4V
Elastic modulus (112 GPa)
Poisson’s Ratio (0.342)

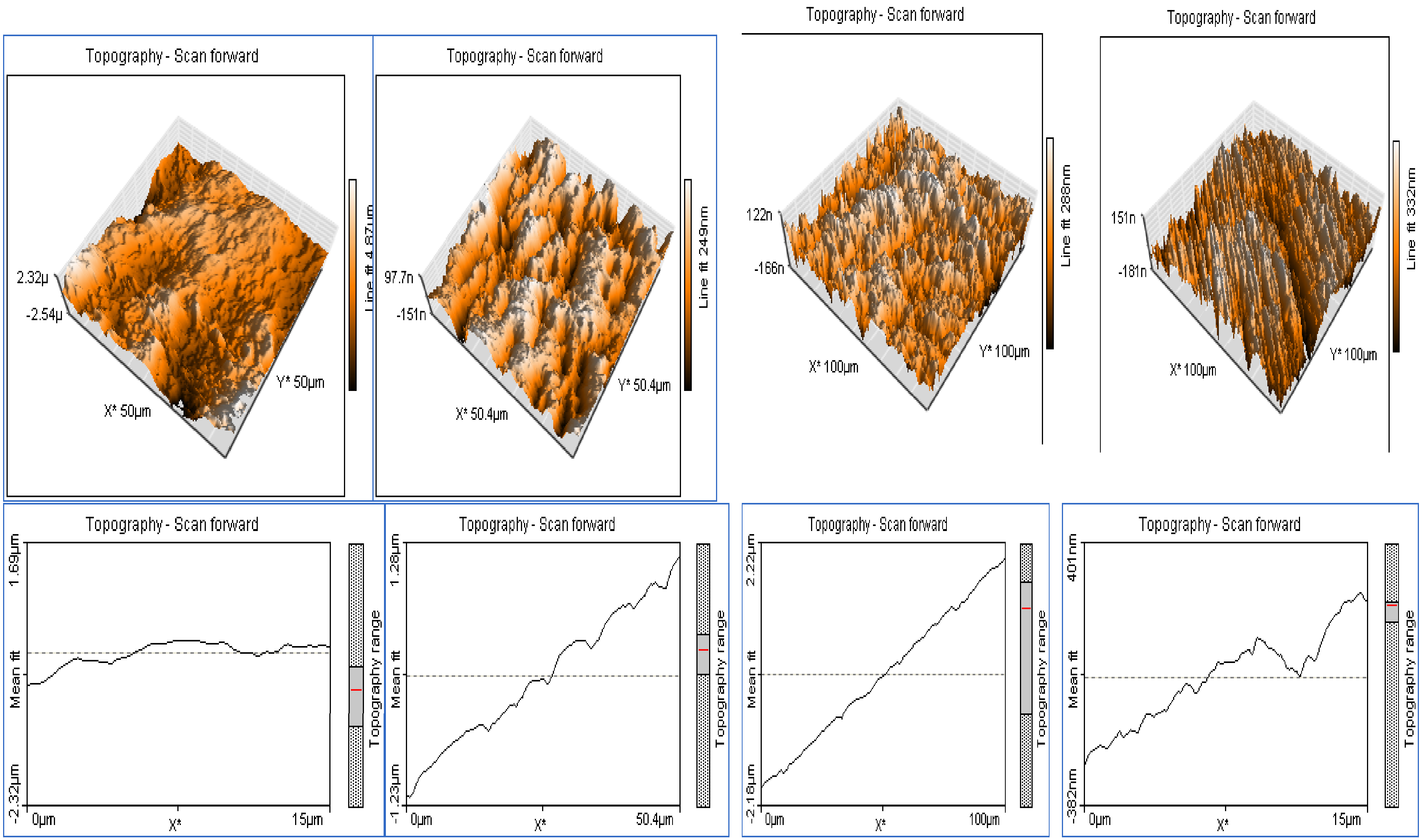
Ti13Nb13Zr
Young's Modulus (77 GPa)
Poisson’s Ratio (0.36)





E of the surface measured using SFM
Acta Biomaterialia 3 (2007) 113–119

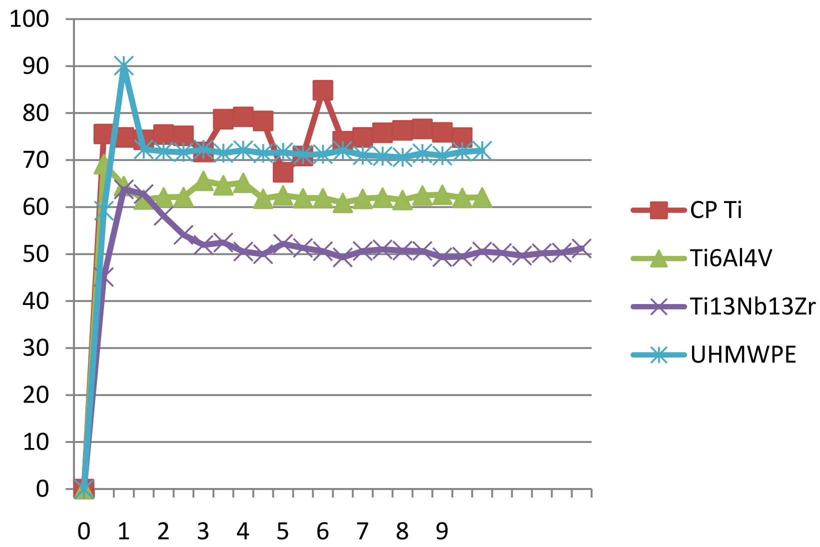
Roughness of all the samples



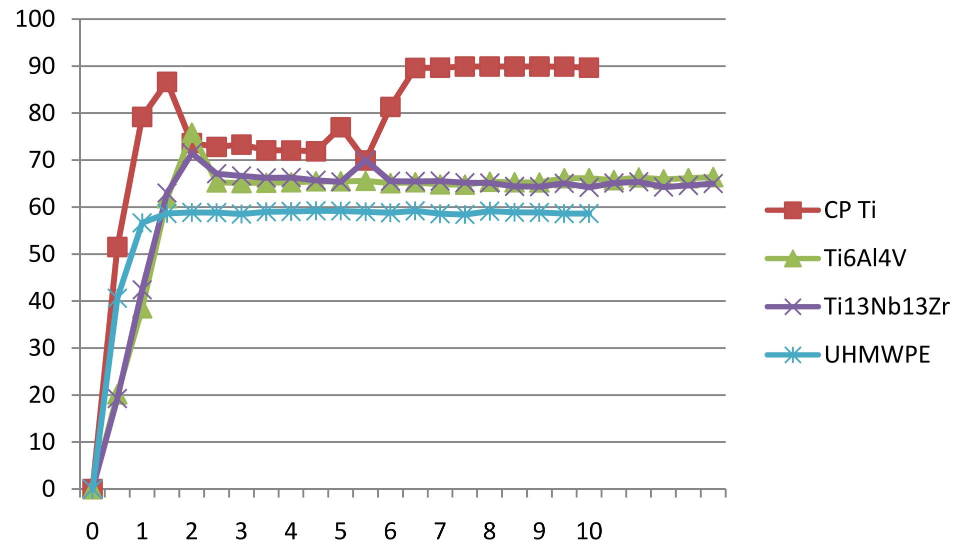
Constant Load Test

	Start Load (N)	Stroke Length (mm)	Speed (mm/sec)	Offset (mm)	Traction Force	Scratch Width (in µm)	Scratch Hardness (in GPa)
Cp-Ti	20N	10	0.50	0.25	**15.81**	**116.41**	**3.76**
Cp-Ti	50N	10	0.50	0.25	**45.86**	**159.13**	**5.63**
Cp-Ti	100N	10	0.50	0.25	**85.47**	**216.7**	**5.43**
Ti-13Nb-13Zr	20N	5	0.50	0.50	**12.94**	**86.88**	**6.75**
Ti-13Nb-13Zr	50N	5	0.50	0.50	**35.72**	**109.90**	**10.55**
Ti-13Nb-13Zr	100N	5	1	0.50	**54.33**	**168.78**	**8.94**
Ti-6Al-4V	20N	10	0.50	0.50	**13.37**	**97.08**	**5.41**
Ti-6Al-4V	50N	5	0.50	0.50	**32.98**	**133.08**	**7.19**
Ti-6Al-4V	100N	5	0.50	0.50	**99.89**	**186.65**	**7.31**

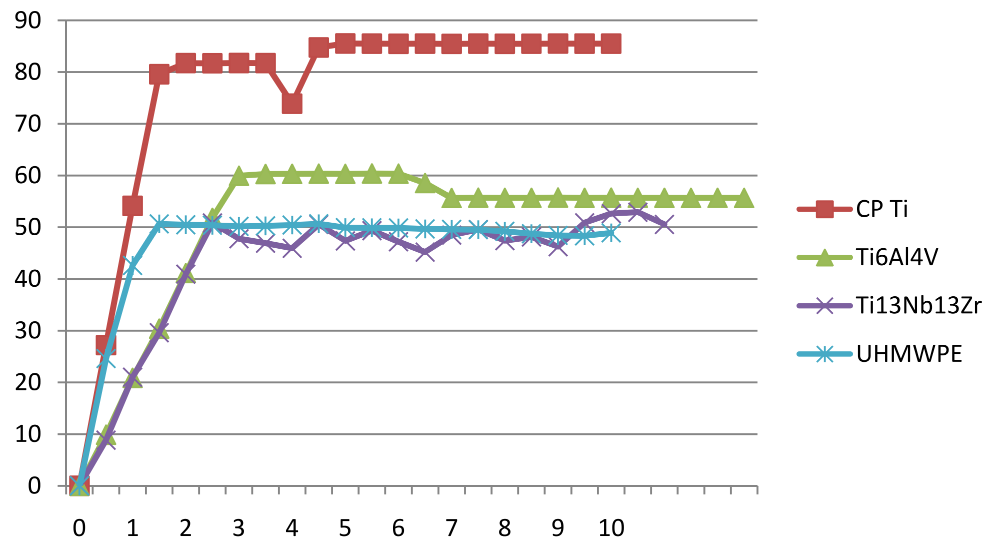
COF Comparison for CL



CL 20N

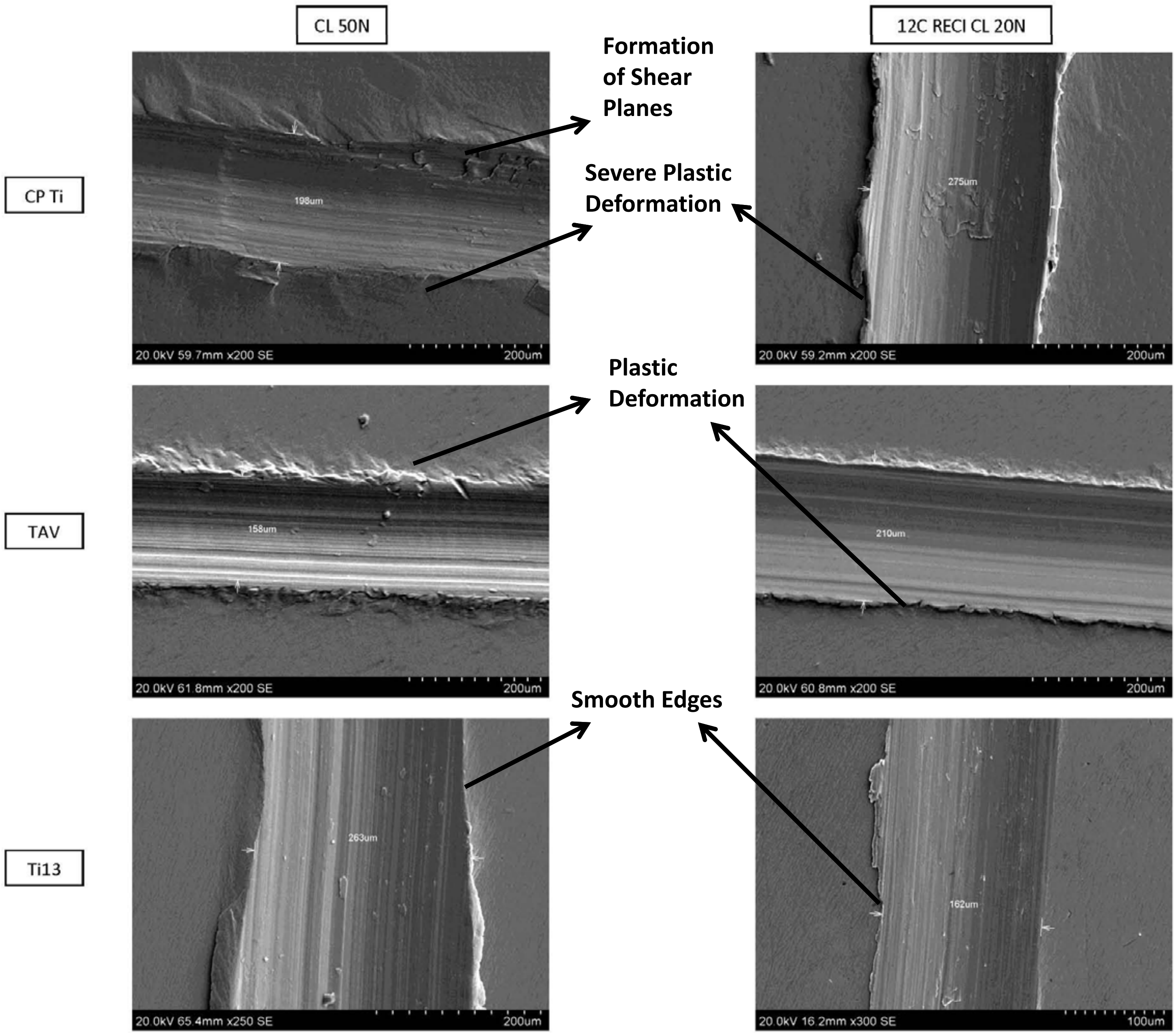


CL 50N

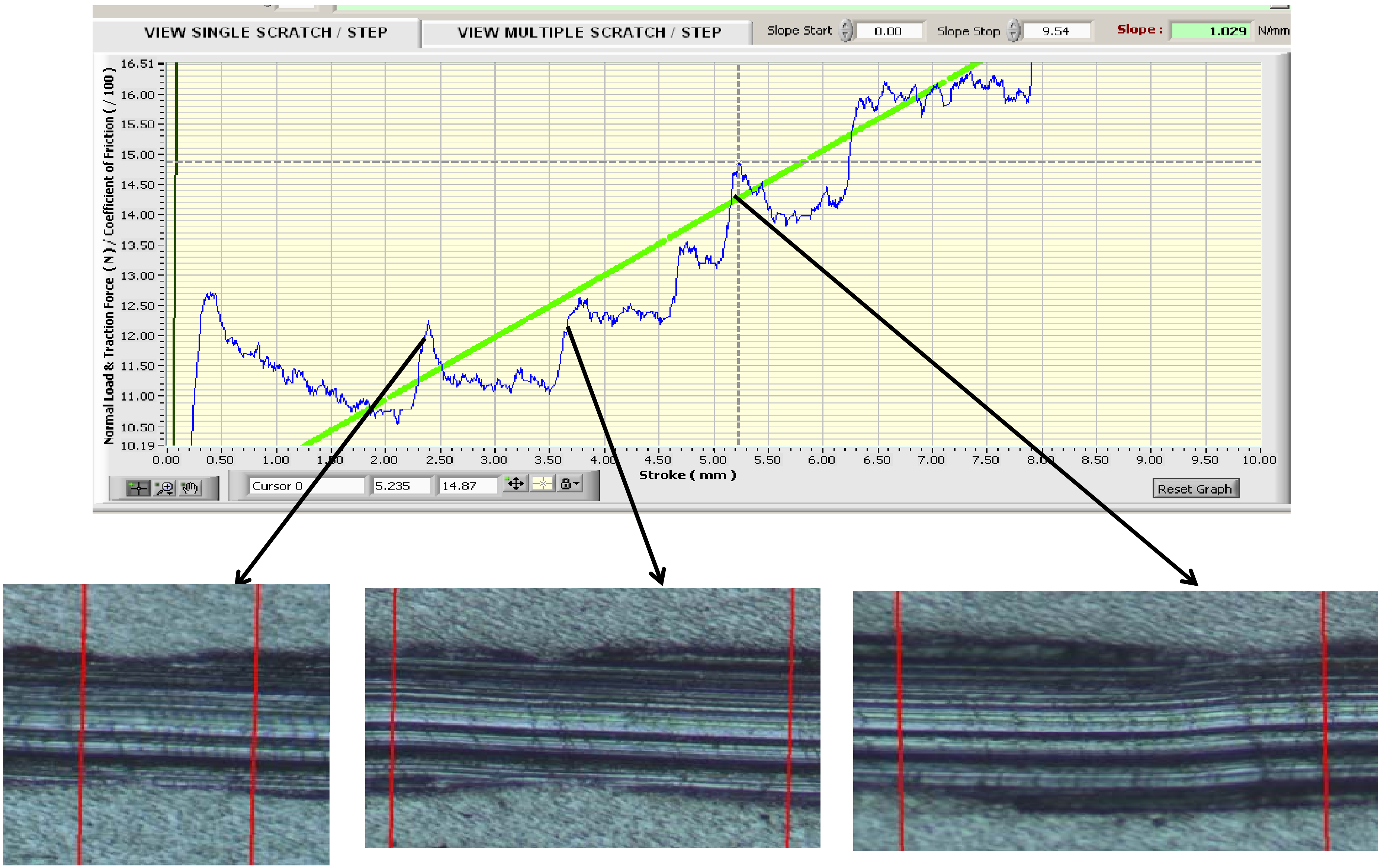


CL 100N

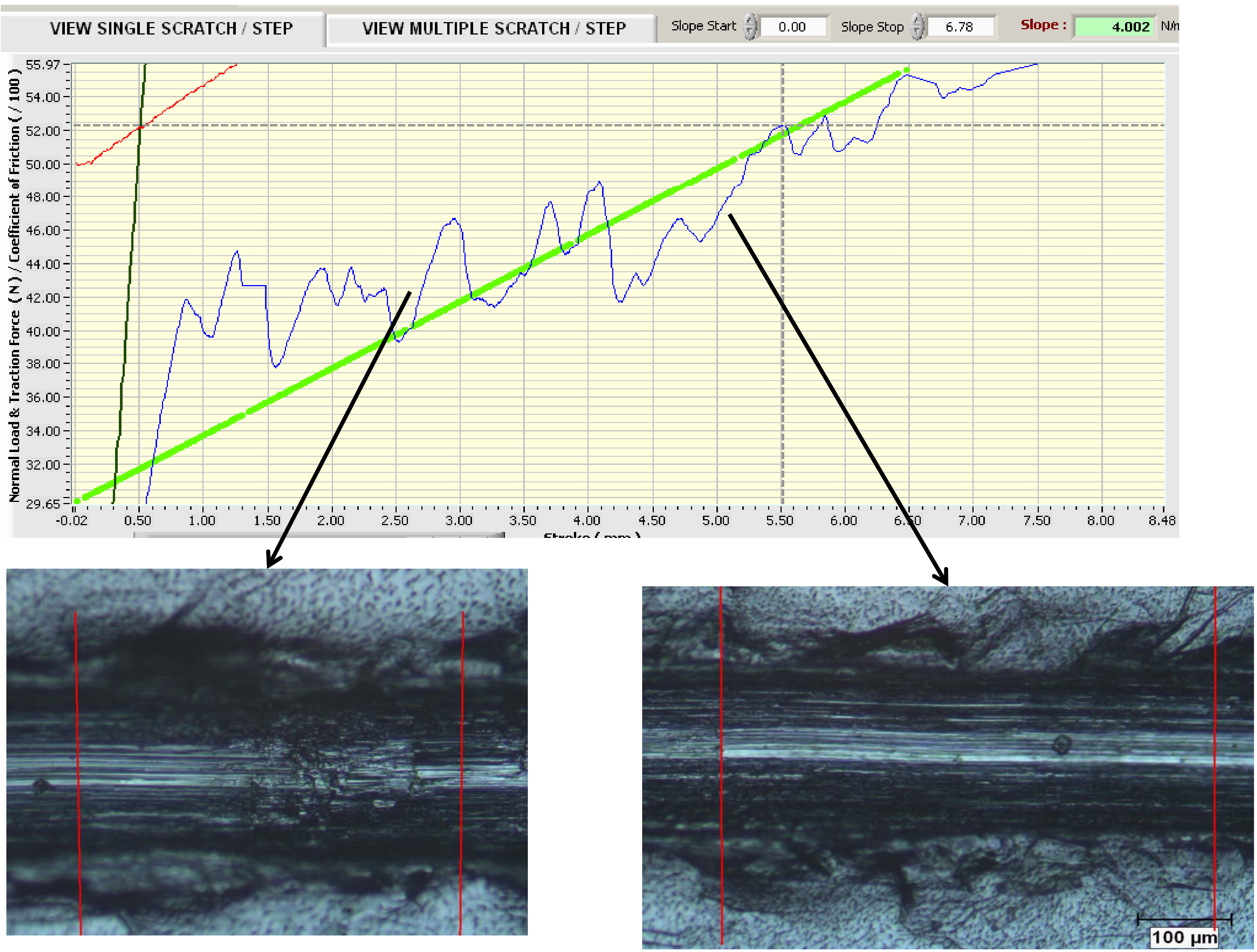
Constant Load Micro Scratch



Traction Force Graph of Ti13Nb13Zr RL 20N



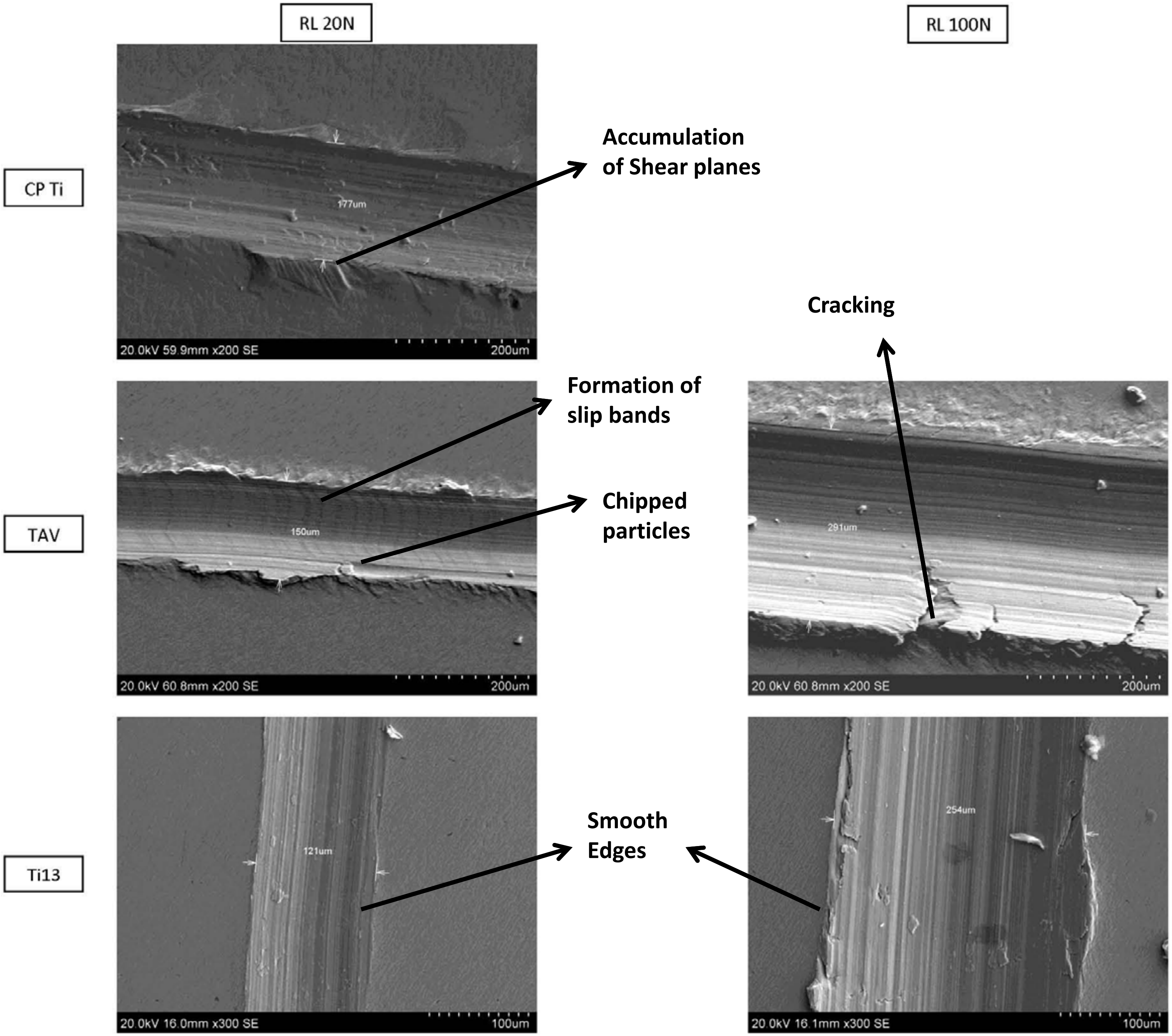
Traction Force Graph of C.P-Ti RL 50N



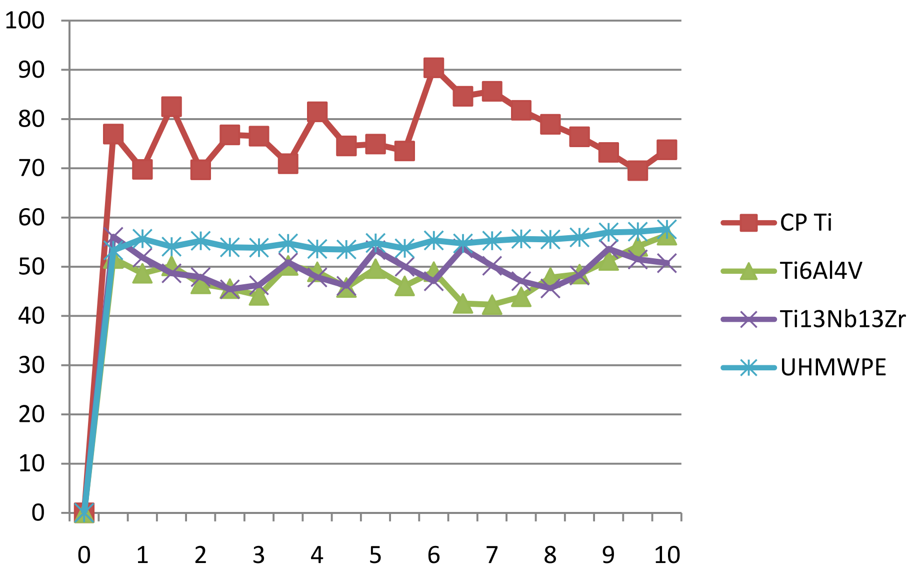
Progressive Load Test

	Start Load (N)	Stroke Length (mm)	Load Rate (mm/sec)	Speed (mm/sec)	Offset (mm)	Traction Force	Scratch Width (in µm)	Scratch Hardness (in GPa)
Cp-Ti	20N	10	2N/mm	0.50	0.25	**28.82**	**132.42**	**2.91**
Cp-Ti	50N	10	2N/mm	0.50	0.25	**48.58**	**171.61**	**4.33**
Cp-Ti	100N	10	2N/mm	0.50	0.25	**81.55**	**211.50**	**5.70**
Ti-13Nb-13Zr	20N	10	2N/mm	0.50	0.50	**14.78**	**115.04**	**3.85**
Ti-13Nb-13Zr	50N	10	2N/mm	0.50	0.50	**29.5**	**132.67**	**7.23**
Ti-13Nb-13Zr	100N	10	2N/mm	0.50	0.50	**57.37**	**178.16**	**8.03**
Ti-13Nb-13Zr	100N	10	5N/mm	0.50	0.50	**65.04**	**157.92**	**10.22**
Ti-6Al-4V	20N	10	2N/mm	0.50	0.50	**13.94**	**104.81**	**4.64**
Ti-6Al-4V	50N	10	2N/mm	0.50	0.50	**35.19**	**142.11**	**6.31**
Ti-6Al-4V	100N	10	5N/mm	0.50	0.50	**79.69**	**198.28**	**6.48**
Ti-6Al-4V	150N	10	5N/mm	0.50	0.50	**80.69**	**217.70**	**8.06**

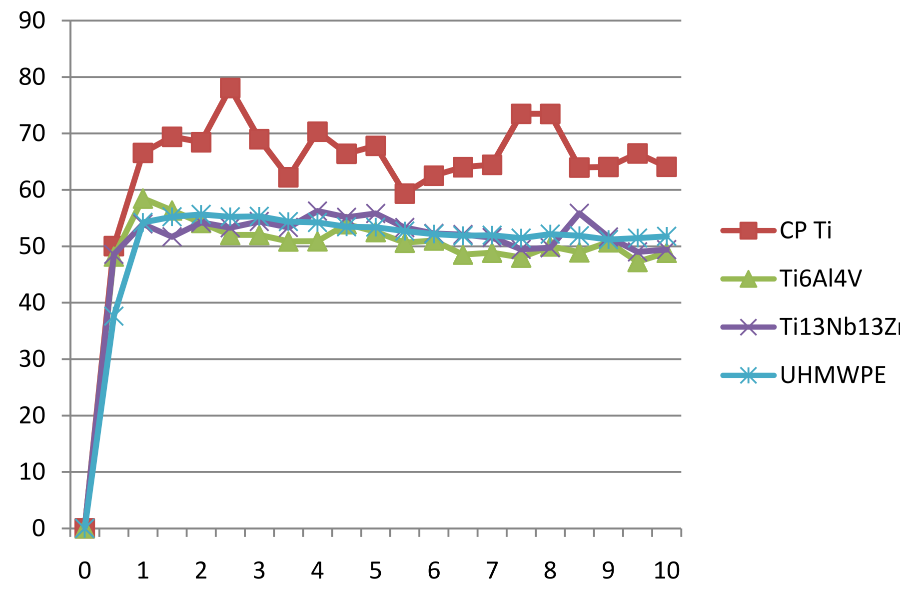
Progressive Load Micro Scratch



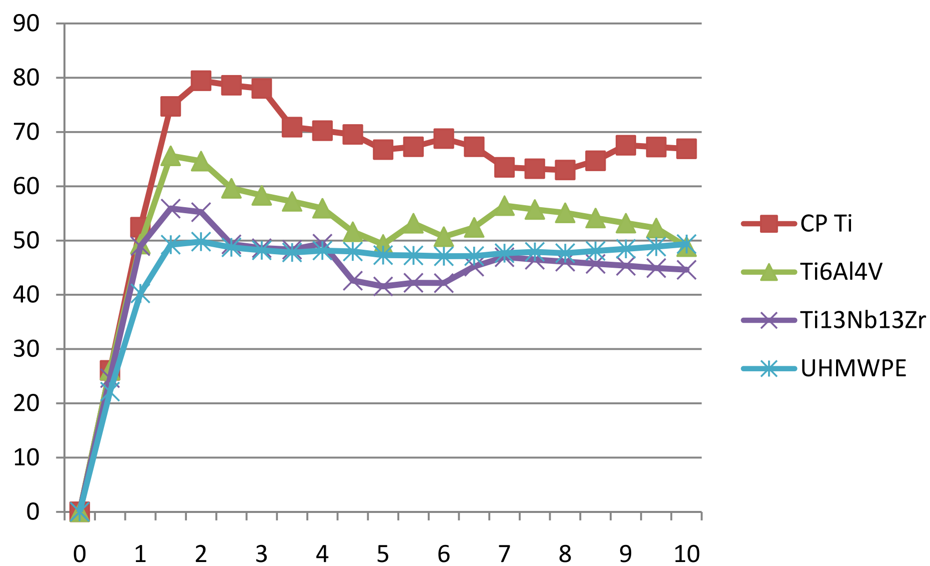
COF Comparison for RL



RL 20N



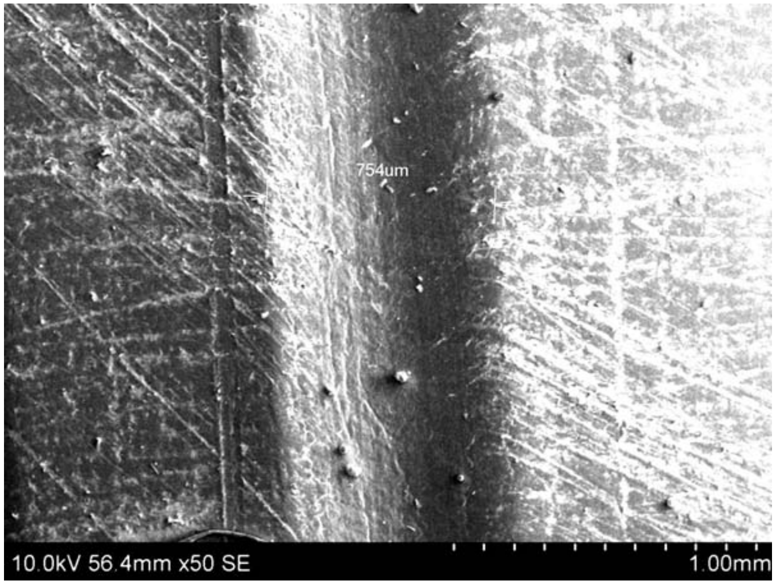
RL 50N



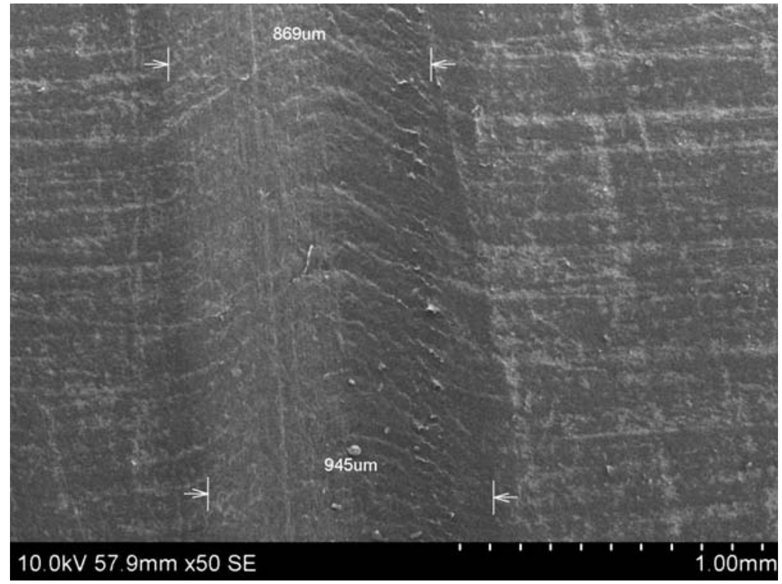
RL 100N

Scratch Testing of UHMWPE

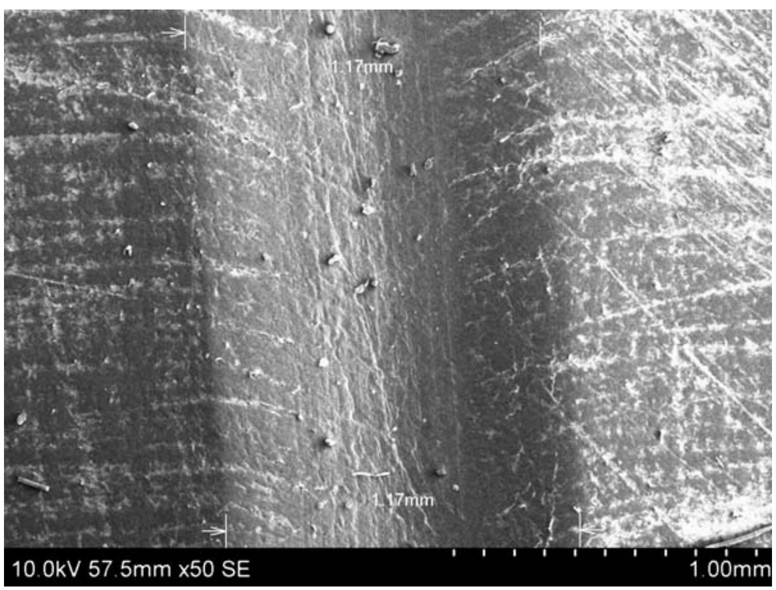
CL 20N



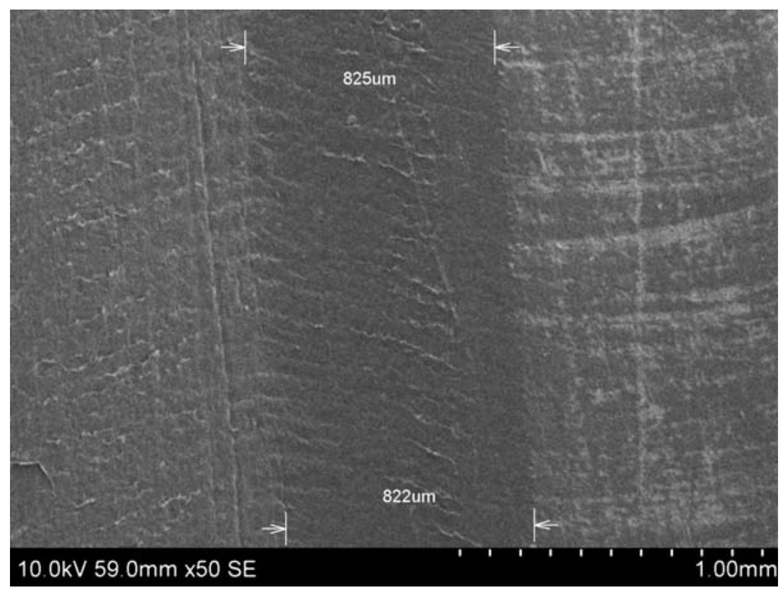
RL 20N



CL 50N



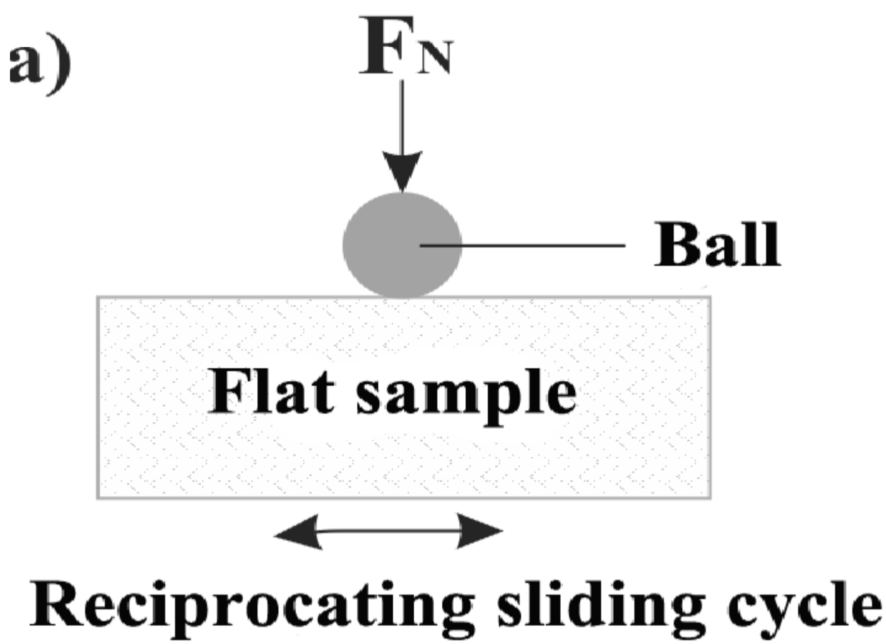
RL 100N

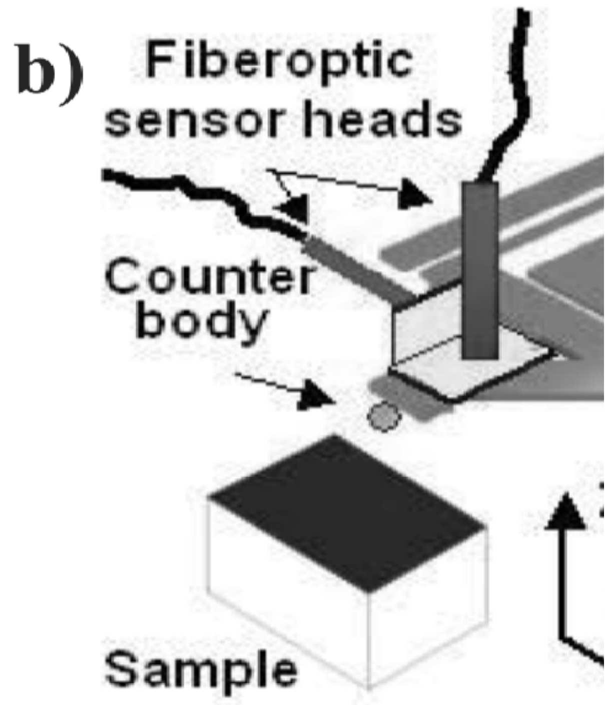


Scratch Hardness of UHMWPE

	Type of Load	Start Load (N)	Stroke Length (mm)	Load Rate (mm/sec)	Speed (mm/sec)	Offset (mm)	Traction Force	Scratch Width (in µm)	Scratch Hardness (in GPa)
UHMWPE	CL	20N	10	2N/mm	0.50	0.25	**14.3N**	**754**	**0.089**
UHMWPE	CL	50N	10	2N/mm	0.50	0.25	**29.5N**	**1170**	**0.093**
UHMWPE	RL	20N	10	5N/mm	0.50	0.25	**15.7N**	**907**	**0.062**
UHMWPE	RL	100N	10	5N/mm	0.50	0.25	**55N**	**823.5**	**0.376**

Nanotribology using CSM Nanotribometer





Ti alloy contact with alumina ball

b Linear reciprocating module

Test conditions

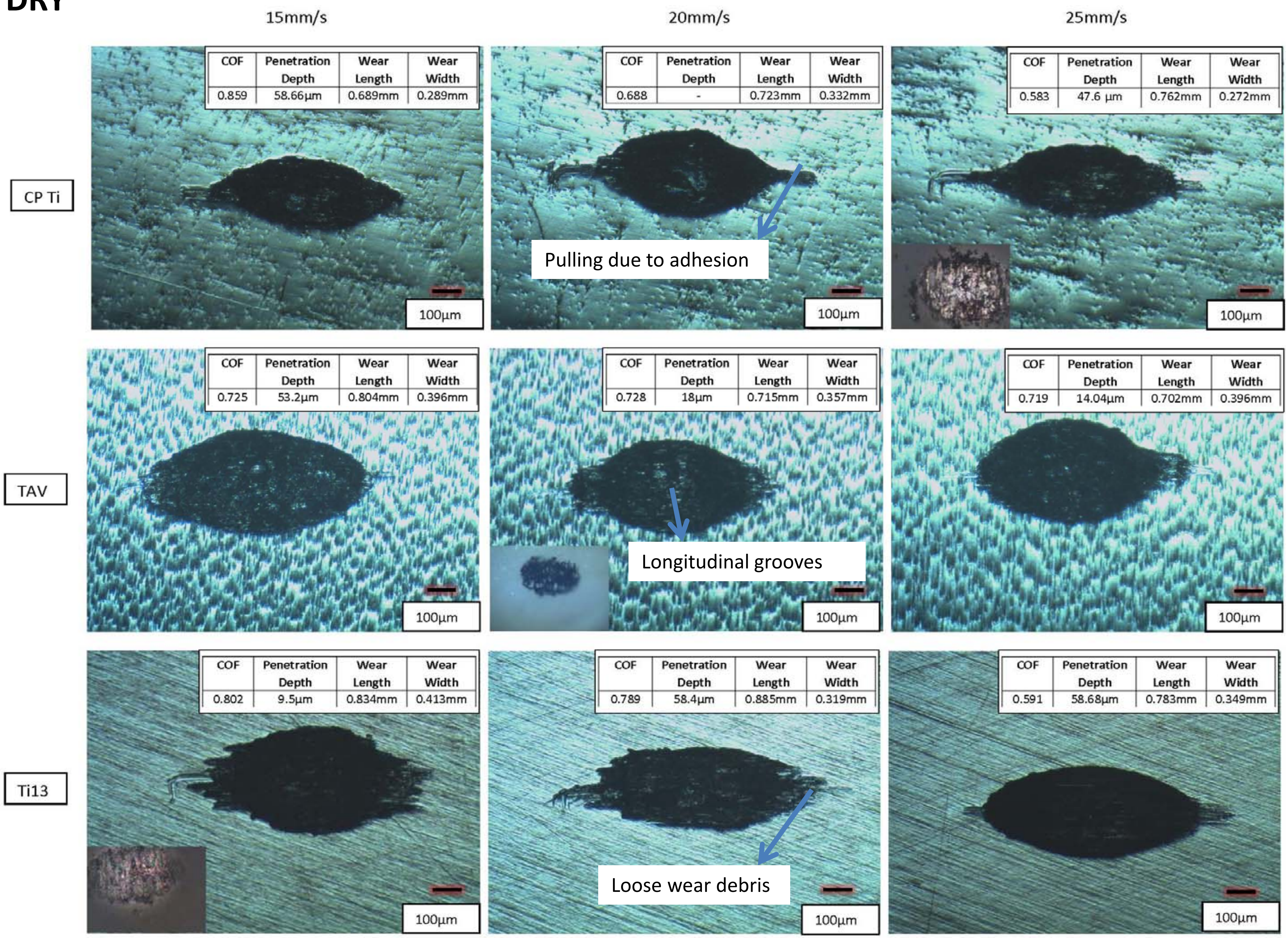
- **Substrate:** - CP Ti, Ti6Al4V, Ti13Nb13Zr
- **Ball:** - Al_2O_3(Alumina)
- **Ball diameter**: - 1.5mm
- **No. of cycles/distance**: - 30000cycles/1mm stroke length
- **Acquisition Rate:** - 20Hz
- **Speed:** - 15, 20 and 25mm/s

  (characteristic for hip joints 0–50 mm/s- Gispert M.P. (2006))
- **Load:** - 1.00N, contact stress - 154.9 MPa
- **Environment:** - Dry and Ringer's Solution
- Ringer‟s solution (g per 1 liter of water): NaCl 8.6; KCl 0.30; CaCl2 - 0.33; Na+ 147.00 mmol; K+ 4.00 mmol; Ca+ 2.25 mmol; Cl+ 155.60 mmol.
- **Air Temperature** :25° C

Output

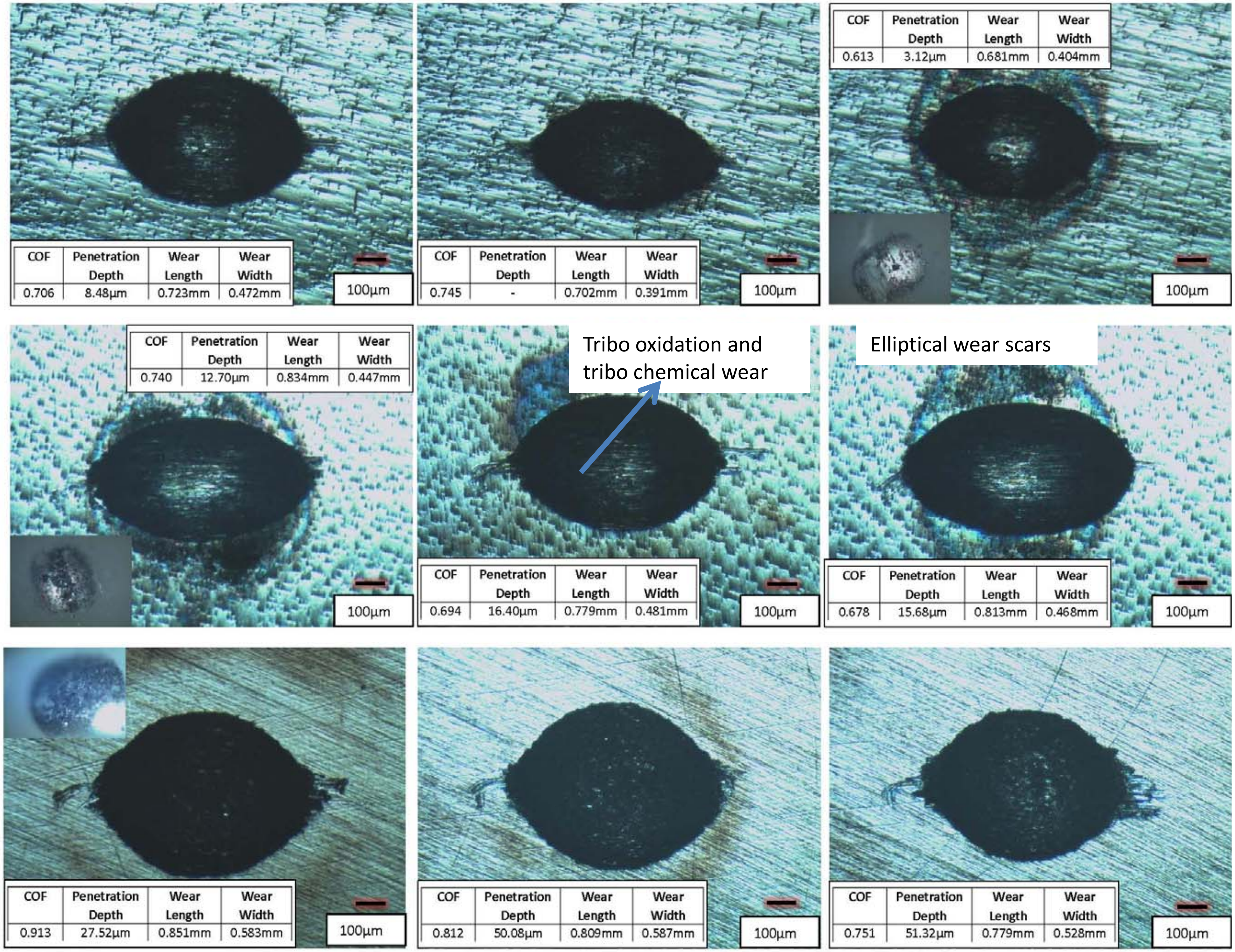
- Penetration depth
- Wear volume (ASTM G133 – 02)
- Coefficient of friction

DRY



15mm/s
20mm/s
25mm/s
CP Ti
TAV
Ti13
COF | Penetration Depth | Wear Length | Wear Width
0.859 | 58.66μm | 0.689mm | 0.289mm
COF | Penetration Depth | Wear Length | Wear Width
0.688 | - | 0.723mm | 0.332mm
Pulling due to adhesion
COF | Penetration Depth | Wear Length | Wear Width
0.583 | 47.6 μm | 0.762mm | 0.272mm
COF | Penetration Depth | Wear Length | Wear Width
0.725 | 53.2μm | 0.804mm | 0.396mm
COF | Penetration Depth | Wear Length | Wear Width
0.728 | 18μm | 0.715mm | 0.357mm
Longitudinal grooves
COF | Penetration Depth | Wear Length | Wear Width
0.719 | 14.04μm | 0.702mm | 0.396mm
COF | Penetration Depth | Wear Length | Wear Width
0.802 | 9.5μm | 0.834mm | 0.413mm
COF | Penetration Depth | Wear Length | Wear Width
0.789 | 58.4μm | 0.885mm | 0.319mm
Loose wear debris
COF | Penetration Depth | Wear Length | Wear Width
0.591 | 58.68μm | 0.783mm | 0.349mm
100μm

RINGER'S SOL'N



	COF	Penetration Depth	Wear Length	Wear Width
CP Ti, 15mm/s	0.706	8.48µm	0.723mm	0.472mm
CP Ti, 20mm/s	0.745	-	0.702mm	0.391mm
CP Ti, 25mm/s	0.613	3.12µm	0.681mm	0.404mm
TAV, 15mm/s	0.740	12.70µm	0.834mm	0.447mm
TAV, 20mm/s	0.694	16.40µm	0.779mm	0.481mm
TAV, 25mm/s	0.678	15.68µm	0.813mm	0.468mm
Ti13, 15mm/s	0.913	27.52µm	0.851mm	0.583mm
Ti13, 20mm/s	0.812	50.08µm	0.809mm	0.587mm
Ti13, 25mm/s	0.751	51.32µm	0.779mm	0.528mm

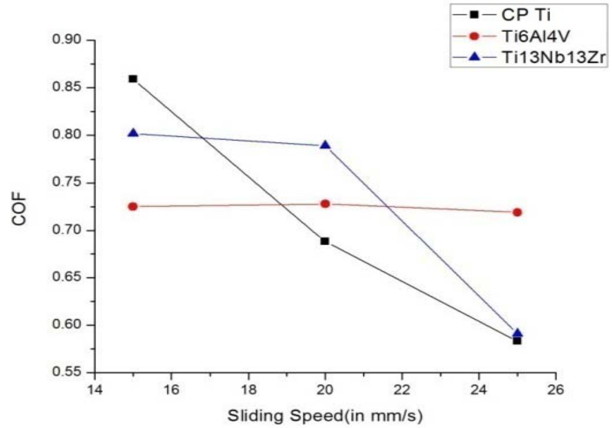
COF Comparison

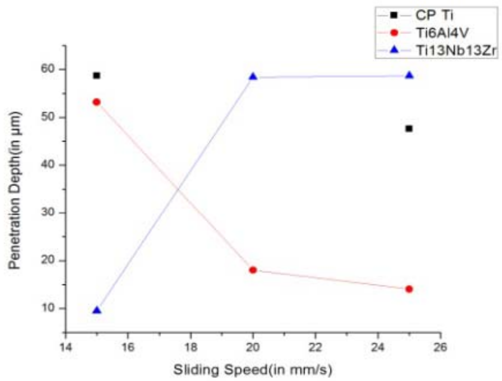
		Sliding Speed						
		Dry				Ringer's Solution		
		15mm/s	20mm/s	25mm/s		15mm/s	20mm/s	25mm/s
Material								
CP Ti		0.859	0.688	0.583		0.706	0.745	0.613
Ti6Al4V		0.725	0.728	0.719		0.74	0.694	0.678
TI13Nb13Zr		0.802	0.789	0.591		0.913	0.812	0.751

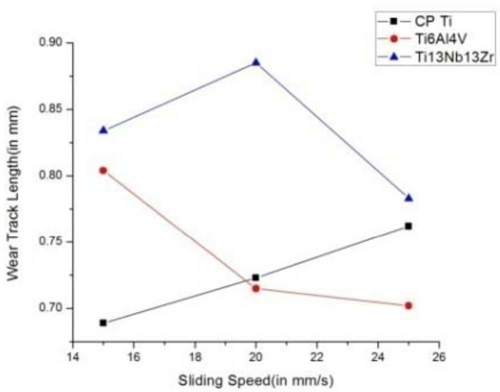
Wear Volume Comparison

	Sliding Speed					
	Dry			Ringer's Solution		
Material	15mm/s	20mm/s	25mm/s	15mm/s	20mm/s	25mm/s
	in mm^3(x 10^-3)					
CP Ti	11.681		9.866	2.894		0.858
Ti6Al4V	16.938	4.595	3.903	4.735	6.145	5.966
TI13Nb13Zr	3.272	16.487	16.035	13.654	23.782	21.109

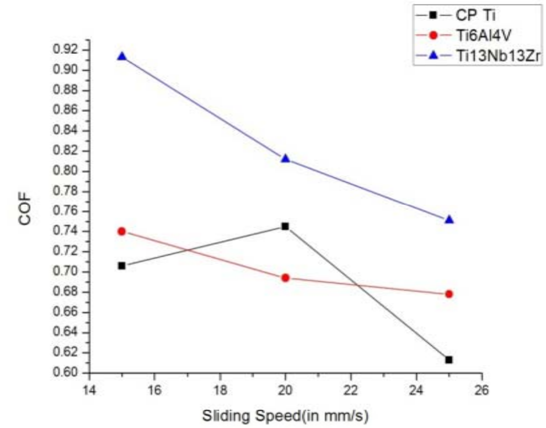
Dry



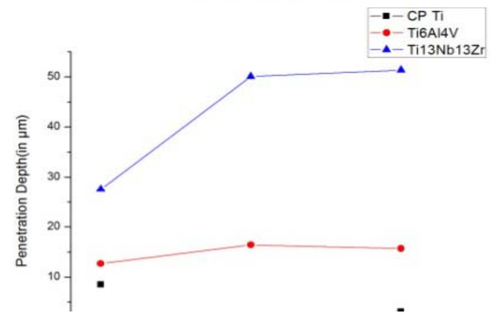


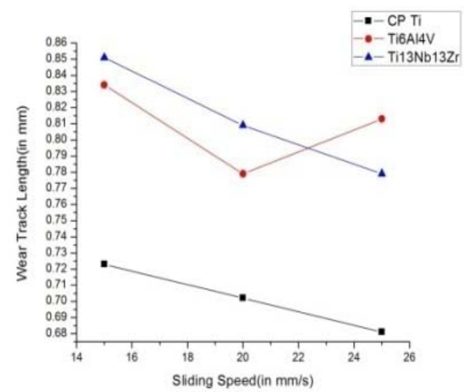


Ringer



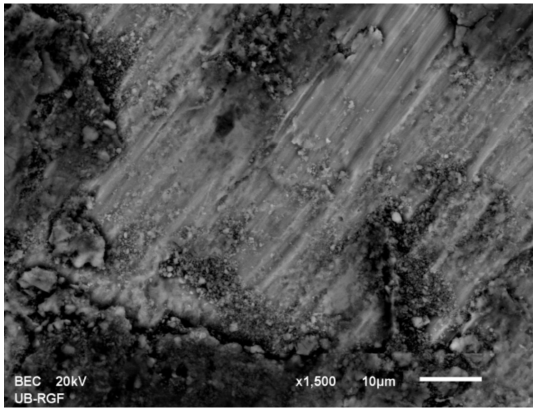
Third body act as protecting layer and reduces friction



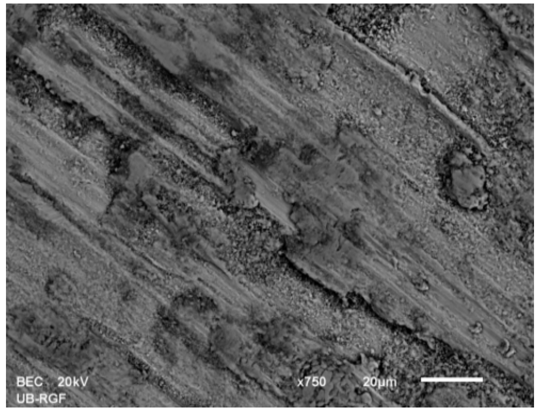


SEM Images of worn track 15mm/s

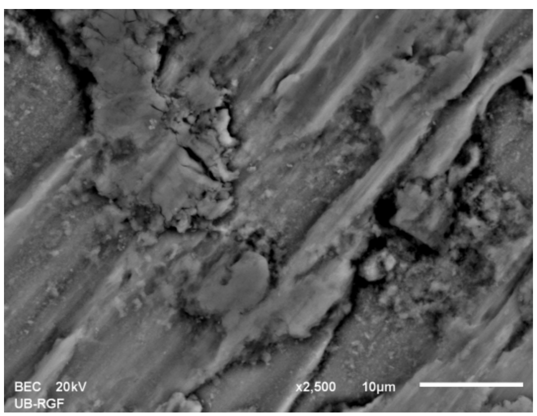
Ti6Al4V-Dry



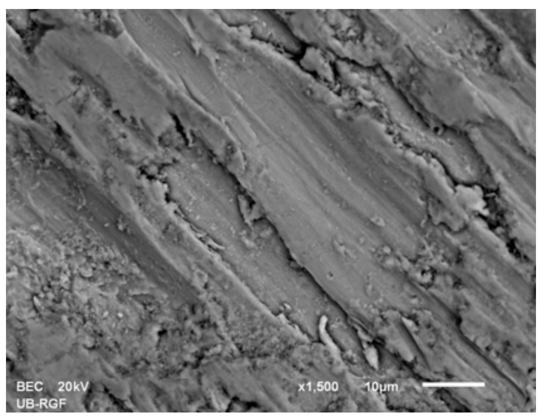
Ti13Nb13Zr-Dry



Ti6Al4V-Ringer’s

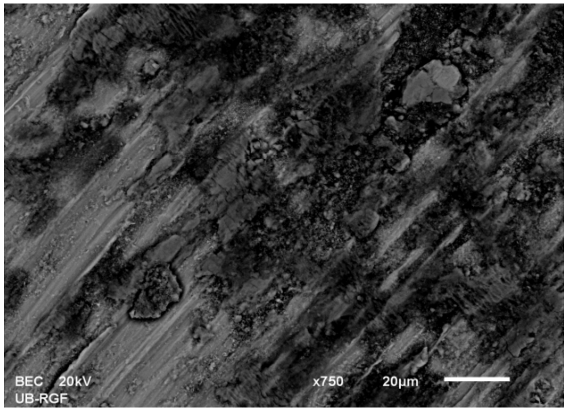


Ti13Nb13Zr-Ringer’s

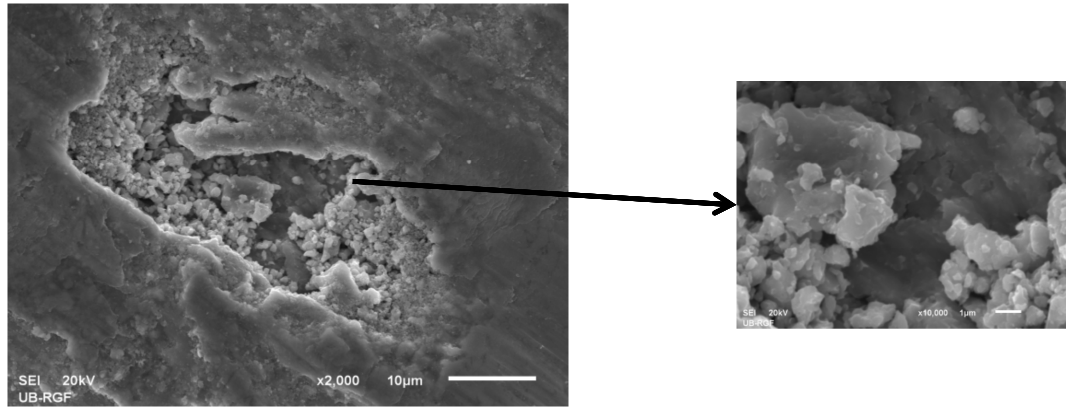


SEM Images of worn track 25mm/s

Ti6Al4V-Dry



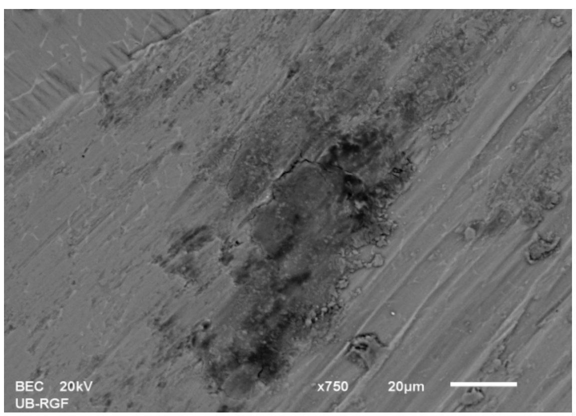
Ti13Nb13Zr-Dry



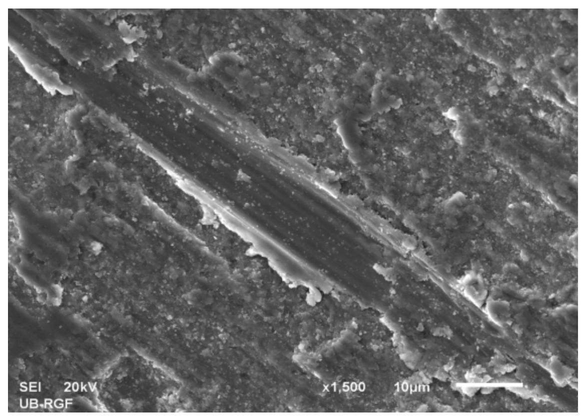
Ti1313 exhibit rather smaller grains as a wear debris

TAV wear debris was larger

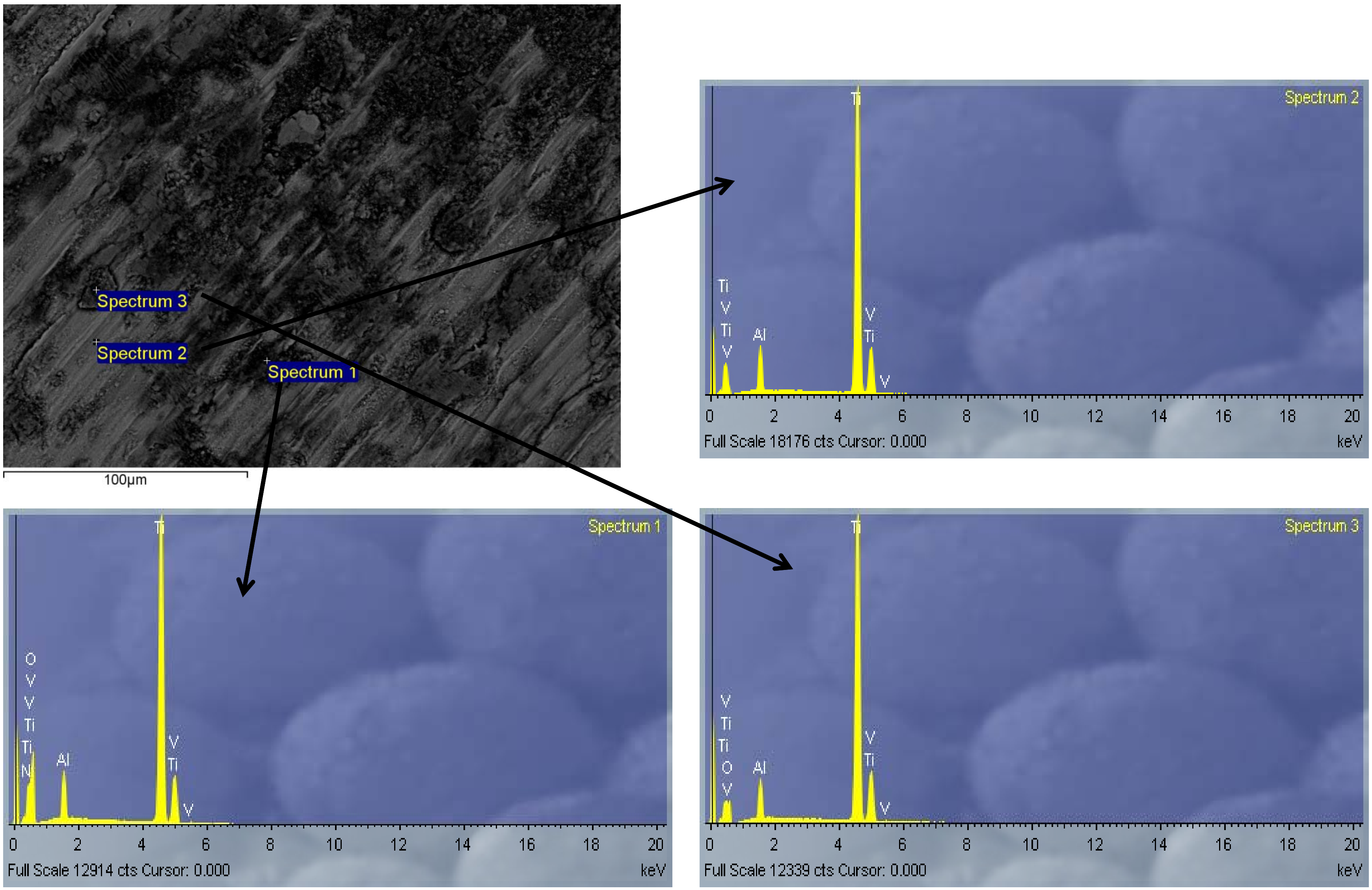
Ti6Al4V-Ringer’s



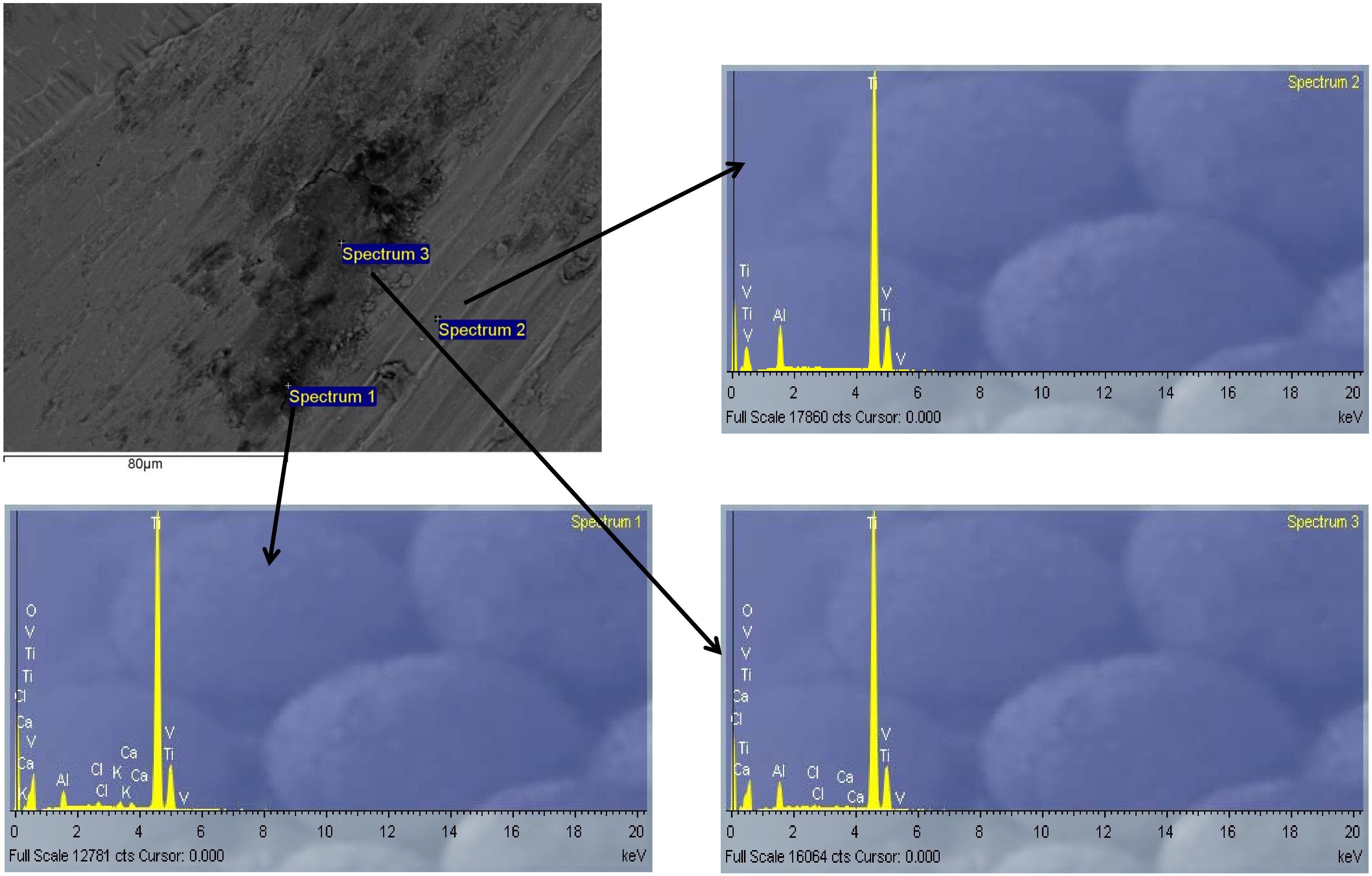
Ti13Nb13Zr-Ringer’s



Ti6Al4V Dry EDS



TI6Al4V Ringer's EDS



Scratch test

- The SEM images show that below the critical loads all samples were damaged by plowing, associated with the plastic flow of material
- Ploughing scratch , Pileup and debris were found on the sides
- In Ti-6Al-4V there were fine debris and large flakes, debris were strain hardened and also flakes abrade the surface and high COF results
- Level of deformation of Ti1313 compared to TAV should be up to 60% higher and exhibits high scratch hardness

Observations

- In TAV- With high sliding speeds, strain hardening is more and hardness increases in ductile material and low wear will occur
- At high speed , large dislocations and twinning occurs, dissipates large amt of energy producing sliding friction and resist the formation of cracks

Observations

- For TAV abrasive and adhesive operate at lower speed, due to accumulation of plastic strain which grows in size , cracks nucleate parallel to the surface and flakes are produced and results in cracking by adhesion and results in materials transfer
- TAV plastic deformation along with strain hardening occurs and increases the surface hardness at high speed
- Increase in wear resistance with increase in sliding speed

Observations

- Ti1313 smearing and delamination
- Wear loss high at high speeds
- The penetration depth depend upon the hardness of the abrasives
- Adhesion is high in 1313 due to ductility and adhesion overlaps abrasion
- In case of 1313 thermal softening occurred and hardness decreased and wear progressed
- COF - smaller grains promote conformal area of contact thus reducing COF values
- The decrease of the grain size happened with speed increase resulting in decrease of COF and increase of wear volume

Observations

- In 1313 the hardness on the surface decreases due to thermal softening and this reduces YS and facilitating delamination
- The alloys' ability to recover their passive state during sliding depends mainly on the mechanical properties of the passive filims and the contact pressure. On one hand the mechanical properties of the passive filim depend mainly on the composition of the alloy

Conclusions

- TAV exhibited more tribooxidation wear than Ti1313 = more black areas throughout the worn track.
- Ti1313 deforms the most under the same normal load, followed by CpTi and then TAV
- smaller particulate size is easier to remove from the wear surface with both abrasive and adhesive wear
- TAV and Ti1313 seems to have similar (abrasive, adhesive, tribochemical wear) but with difference of particulate grain size, especially in Ringer because TAV surface in Ringer is rather smooth, while Ti1313 has this grain surface structure even in Ringer (with smaller grain size in Ringer than at dry sliding)
- Ti1313 is more flexible (less stiff/rigid) than other 2 alloys