

# Effect of Surface Treatment on Surface Characteristics of 316L Stainless Steel

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# Presentation Outline

- Introduction
- Motivation
- Experimental Procedure
- Analytical Techniques
- xxx
- yyy
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- Conclusions
- future work



# Introduction

- AISI 316L Stainless Steel
  - Ni (10~14%), Cr (16~18%), Mo (2~3%)
  - Austenitic
- Used widely in medical devices
  - Fracture fixation plates, screws
  - Intramedullary nails
  - Stents
  - Etc
- Thermomechanical/processing history affects
  - Bulk properties
  - Surface properties



# Surfaces & Biocompatibility

- Biocompatibility affected by surface characteristics
  - Surface chemistry
  - Surface roughness
  - Surface porosity
  - Surface thermodynamics
    - Critical surface tension (of solid phase)
  - Surface charge
  - Others ??
- Chemical & mechanical processing affects/changes surface characteristics



# Previous Studies

- Relatively large number of studies relating various aspects of surfaces to biocompatibility
- Primarily *in vivo* and/or *in vitro* tests
- Wide variety of testing methods for biocompatibility
- Detailed correlation of effect of processing on surface characteristics and subsequent effect on biocompatibility still needed



# Objective

- Investigate the effect of surface treatment on the surface characteristics and surface chemistry of NiTi, AISI 316L, Co-Cr alloys
- Relate to biocompatibility and corrosion behavior
- This work:
  - Effect of mechanical polishing, electropolishing and plasma treatment on surface roughness, surface chemistry and critical surface tension



# Experimental Approach

## ● Materials

- AISI 316L
- 19 mm x 19 mm x 0.737 mm coupons
- Cleaned
- Composition verified by GCMS

## ● Surface treatments:

- Mechanical polishing (MP)
  - #1000 SiC paper
- MP + Electropolishing (EP)
  - 63%  $H_3PO_4$ , 15%  $H_2SO_4$ , 22% DI  $H_2O$
- MP + Plasma treatment
  - 98% He, 2%  $O_2$
- EP + Plasma treatment



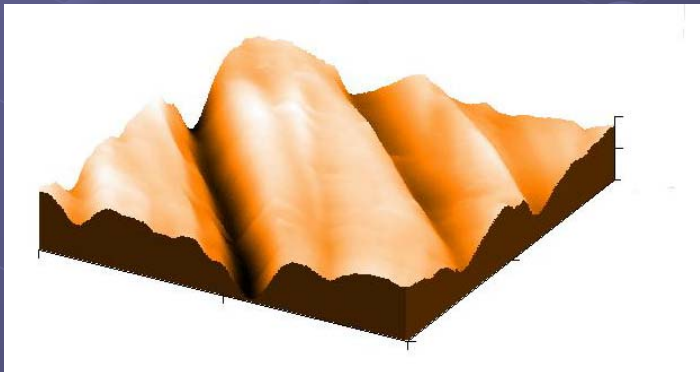
# Analysis

- Surface Roughness: Atomic Force Microscopy
- Surface Chemistry: X-ray Photoelectron Spectroscopy
- Depth Profile: Auger Electron Spectroscopy
- Critical Surface Tension: Zisman Plots



# Surface Roughness - 1

	MP	EP	EP+ Plasma	MP+ Plasma
Roughness Average ( $\text{\AA}$ )	32.15	14.66	14.06	29.83
Standard deviation ( $\text{\AA}$ )	8.03	3.60	5.23	8.82



Mechanically polished surface



Electropolished surface

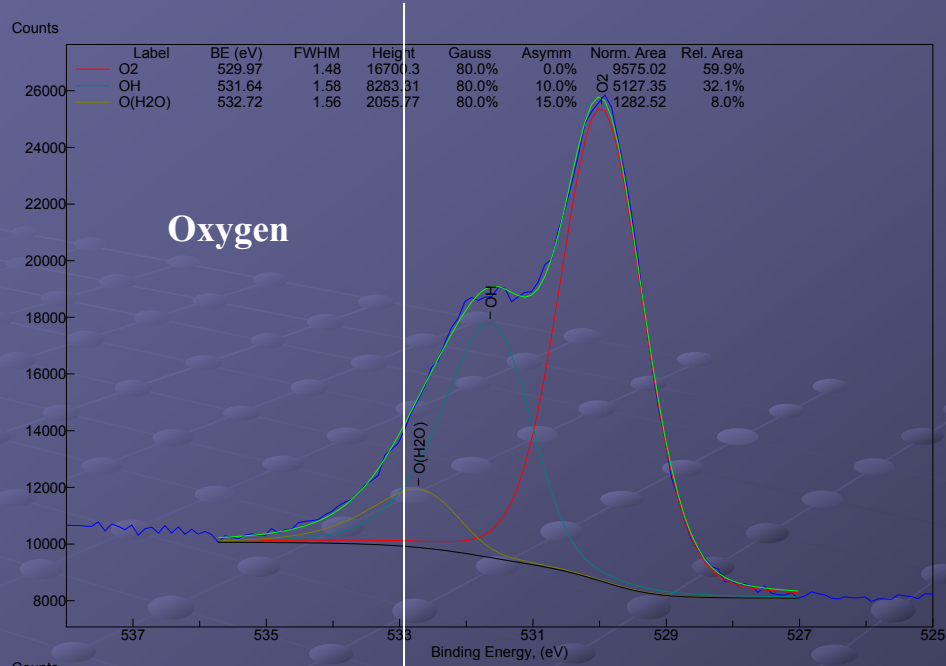


# Surface Roughness - 2

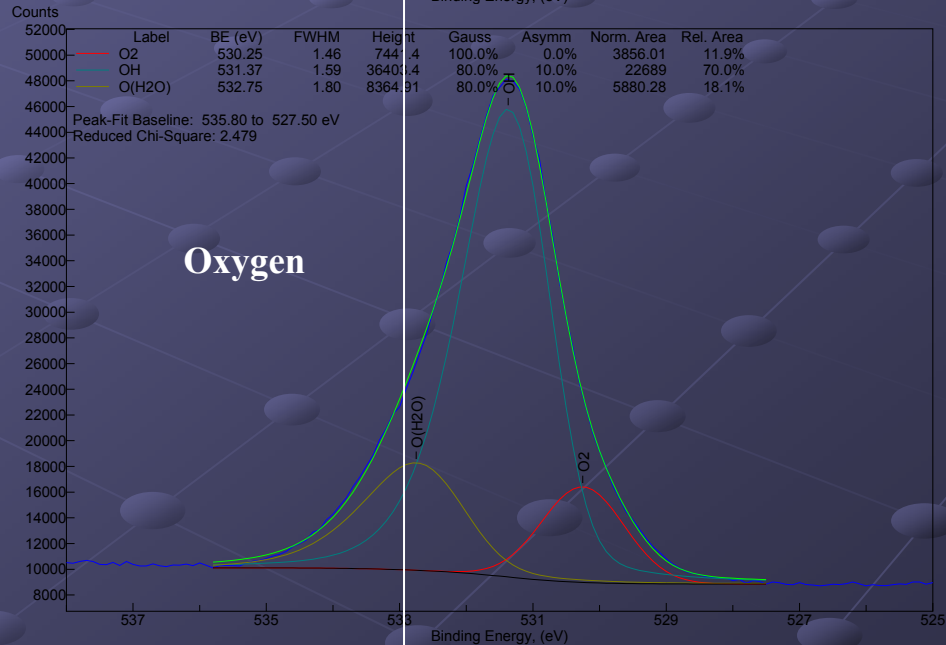
- Electropolishing very effective in reducing surface roughness
- Electropolishing improves surface uniformity
- Plasma treatment had no observable effect on surface roughness



# X-ray Photoelectron Spectroscopy



Mechanically polished



Electropolished



# X-ray Photoelectron Spectroscopy

	Bond Type	MP	EP	EP+ Plasma	MP+ Plasma
C	C-C/C-H	72.2	71.6	74.7	77.1
	C-O	15.5	16.2	14.8	12.4
	C=O	5.9	5.3	3.9	4.5
	COO-	5.9	5.1	3.9	3.7
	O-C=O	0.5	1.7	2.7	2.4
O	O=	59.9	11.9	27.2	65.1
	O-H	32.1	70.0	59.7	28.1
	O(H <sub>2</sub> O)	8.0	18.1	13.1	6.8
Cr	Cr	13.0	16.3	11.2	7.9
	Cr <sub>2</sub> O <sub>3</sub>	67.3	48.4	68.9	80.1
	Cr-OH	19.7	35.3	19.9	12.1
Fe	Fe	9.4	5.6	5.1	0.6
	FeO	9.6	16.3	4.5	0.7
	Fe <sub>2</sub> O <sub>3</sub>	59.8	51.5	76.0	81.7
	Fe-OOH	17.4	12.1	10.7	15.7
	Fe Sat	3.8	14.6	3.7	1.3
Cr:Fe Ratio		1:2.10	1:1.71	1:1.82	1:5.88



# XPS Results - 1

## ● Electropolishing:

- $O_2$  in oxide form ↓
- $O_2$  in hydroxide form ↑↑
- Cr as hydroxide ↑
- Fe as metallic,  $Fe_2O_3$ , and hydroxide ↓
- Fe as FeO ↑

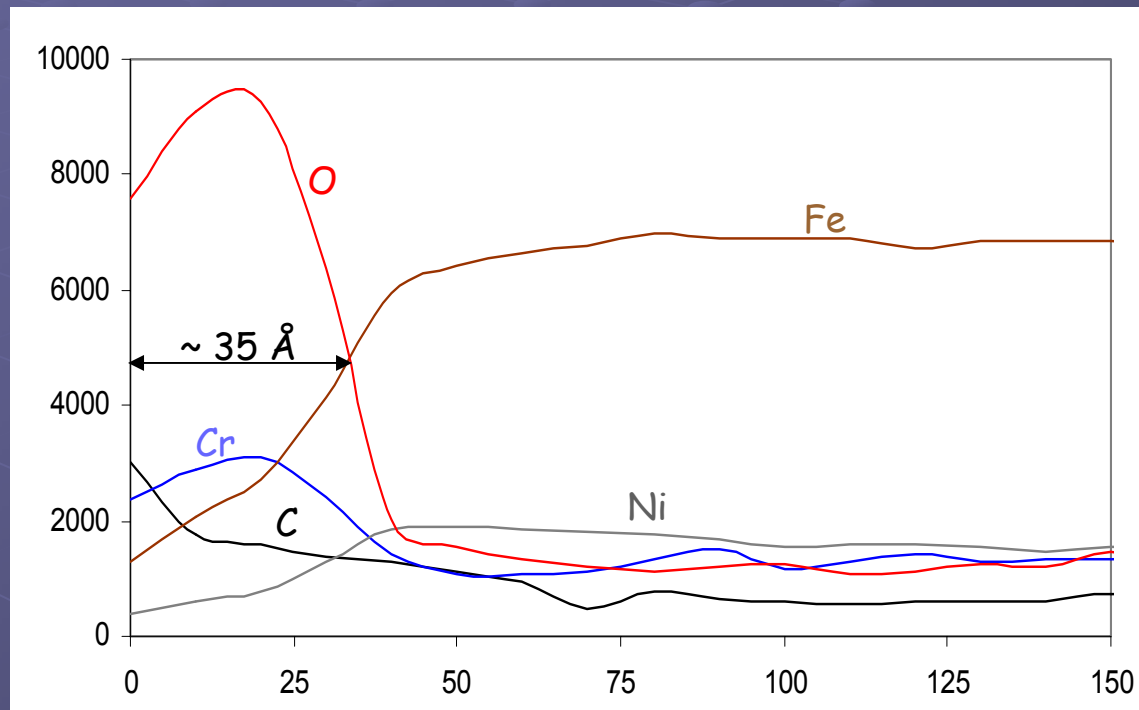


# XPS Results - 2

## ● Cr:Fe ratio

- Bulk: 1:3.63
- After mechanical polishing: 1:2.10
- After electropolishing: 1:1.71
- Enrichment of Cr on the surface enhanced by electropolishing
- Effect of plasma treatment:
  - MP + Plasma treatment: 1:5.88
  - EP + Plasma treatment: 1:1.82

# Auger Electron Spectroscopy



*AES depth profile of electropolished 316L stainless steel*



# Auger Electron Spectroscopy - 2

Element	MP	EP	EP+ Plasma	MP+ Plasma
O	63	66	69	70
Cr	16	20	10	10
Fe	18	10	16	18
Ni	3	3	5	2
Thickness	~ 40 Å	~ 35 Å	~ 35 Å	~ 75 Å

- Ni concentration on surface significantly lower than bulk concentration of 13.78%
- Electropolishing enhances surface Cr concentration
- Surface oxide layer thickness ~ same, except for MP + Plasma



# Conclusions

- AES & XPS results consistent
- Electropolishing enriches Cr concentration in the surface oxide layer
- "Surface oxide layer" is a combination of Fe and Cr oxides and hydroxides
- Surface concentration of Ni ~ 3% - much lower than bulk concentration of 13.78%
- Plasma treatment reduces Cr:Fe ratio; does not improve surface passivation