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X-ray photoelectron spectroscopy investigation of X60 steel

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Raouaa Hannachi ; Deborah Biggio ; Bernhard Elsener ; Marzia Fantauzzi ; Antonella Rossi 



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X-ray photoelectron spectroscopy investigation of X60 steel

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Raouaa Hannachi, Deborah Biggio, Bernhard Elsener, Marzia Fantauzzi, and Antonella Rossi

AFFILIATIONS

Dipartimento di Scienze Chimiche e Geologiche, Università di Cagliari, Cittadella Universitaria, Monserrato, Cagliari 09042, Italy

Note: This paper is part of the 2024 Special Topic Collection on Materials for Energy and the Environment.

ABSTRACT

XPS is used to characterize the surface of commercial X60 pipeline steel after chemical cleaning with a sequence of five solvents and following mechanical polishing. This steel is used as material for pipeline construction aiming to green hydrogen transportation.

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Accession #: 01976 and 01977

Technique: XPS

Specimen: X60 steel cleaned with five solvents (cleaned X60 steel) and mechanically polished X60 steel (MP X60 steel)

Instrument: Thermo Scientific Theta Probe

Major Elements in Spectra: Fe, O, and C

Minor Elements in Spectra: None

Published Spectra: 8

Spectral Category: Comparison

INTRODUCTION

API 5L grade X60 steel (standardized by American Petroleum Institute) has been used since long time as steel for natural gas and petroleum pipelines and in oil and gas applications. Recently, the material has gained interest for hydrogen storage and transportation (Ref. 1). The surface chemistry of X60 steel plays an important role when performing electrochemical hydrogen permeation tests (Ref. 2) because it might affect the hydrogen production and detection kinetics. In this work, x-ray photoelectron spectroscopy was used for characterizing the surface of X60 steel after a thorough cleaning procedure and following mechanical polishing. No significant changes in the chemical state of the elements are detected between the samples upon the different sample preparation procedures. The mechanical polishing reduces the thickness of the natural oxide layer present on the steel surface.

SPECIMEN DESCRIPTION (ACCESSION # 01976 and 01977)

Specimen: X60 steel, thickness 2 mm, diameter 5 cm; cleaned (# 01976) and mechanically polished (# 01977)

CAS Registry #: Unknown

Specimen Characteristics: Homogeneous; solid; polycrystalline; conductor; metal;

Chemical Name: API 5L grade steel X60

Source: RINA Consulting—CSM S.p.A.

Composition: Fe, Mn < 1.4%, Cr, Mo, Ni, Cu, C < 0.28%, and Si < 0.45% (Ref. 3)

Form: X60 steel disk 50 × 2 mm² (diameter × thickness)

Structure: XRD results performed by Rina consulting CSM S.p.A. showed that X60 steel has body centered cubic (BCC) structure. SEM cross-sectional analysis of X60 steel (etched with 2% Nital solution for 5 s) conducted in the Metallography Laboratory of Rina consulting CSM S.p.A. showed that X60 steel has tempered martensite and bainite microstructures with the distribution of precipitated carbides.

History and Significance: The X60 steel was cut from a pipeline tube to obtain disks of 2 mm thickness and a diameter of 50 mm. The surface roughness determined by a SV-3200 surface roughness tester was 0.3–0.4 μm.

As Received Condition: The “as received” X60 steel after cutting is covered with an oil/grease film.

Analyzed Region: Center and at least two other analysis points of the sample surface

Ex Situ Preparation/Mounting: Two different procedures were applied:

Five solvents' cleaning (# 01976): The as received samples were cleaned by following the described procedure in ultrasonic bath at 40 °C: butan-2-one (5 min), acetone (5 min), ethanol (5 min), petroleum ether (5 min), and double distilled water [specific conductivity = 1.5(0.1) $\mu\text{S}/\text{cm}$] (10 min). After the cleaning procedure with five solvents, the sample was dried under an argon stream and analyzed by XPS. The time between the drying step and the sample insertion into the fast entry air lock of the spectrometer is less than 1 min.

Five solvents' cleaning and mechanical polishing (# 01977): XPS characterization was also carried out on the X60 sample following cleaning with five solvents and mechanical polishing using SiC 4000-mesh paper in the presence of ethanol as lubricant for 3 min and 30 s. After grinding, the samples were immersed in ultrasonic bath at 40 °C in ethanol for 5 min and in double distilled water for 10 min. Also, in this case, the samples were dried under an argon stream. The time between the drying step and the sample insertion into the fast entry air lock of the spectrometer was less than 1 min.

In Situ Preparation: None

Charge Control: No charge control

Temp. During Analysis: 300 K

Pressure During Analysis: $<1 \times 10^{-7}$ Pa

Preanalysis Beam Exposure: 0 s

INSTRUMENT DESCRIPTION

Manufacturer and Model: Thermo Scientific Theta Probe

Analyzer Type: Spherical sector

Detector: Other, multichannel plate detector

Number of Detector Elements: 128

INSTRUMENT PARAMETERS COMMON TO ALL SPECTRA

Spectrometer

Analyzer Mode: Constant pass energy

Throughput ($T = E^N$): The energy dependence can be determined

by the following equation: $\frac{A}{E_p} = \left(\frac{a^2}{(a^2 + R^2)}\right)^b$, where a and b are constants, E_p is the pass energy, A is the peak area, and R is the retard ratio equal to E/E_p , where E is the kinetic energy. Three spectral regions [Cu $2p$ (925–940 eV), Cu $3p$ (68–82 eV), and Cu $L_{3M_{4,5}M_{4,5}}$ (561–577 eV)] are recorded on a sputter cleaned copper sample at different pass energies (10, 20, 50, 100, 125, 150, 200, 300, and 400 eV). The values of a and b are then determined to be 26.66 and 1.11, respectively, by a linear least square fit of the data applying the equation described above (Ref. 4).

Excitation Source Window: None

Excitation Source: Al K_{α} monochromatic

Source Energy: 1486.6 eV

Source Strength: 100 W

Source Beam Size: Nominal spot size: $400 \times 400 \mu\text{m}^2$ spot size measured by analyzing Au/Si fresh cleaved sample using line scan measurement resulted to be equal to $355.8 \times 219.2 \mu\text{m}^2$.

Signal Mode: Pulse single channel

Geometry

Incident Angle: 30°

Source-to-Analyzer Angle: 67.38°

Emission Angle: 53°

Specimen Azimuthal Angle: 70°

Acceptance Angle from Analyzer Axis: 60°

Analyzer Angular Acceptance Width: 30° \times 30°

Ion Gun

Manufacturer and Model: Thermo Scientific EX050

Energy: 3000 eV

Current: 0.002 mA

Current Measurement Method: Biased stage

Sputtering Species and Charge: Ar⁺

Spot Size (unrastered): 200 μm

Raster Size: 3000 \times 3000 μm^2

Incident Angle: 45.00°

Polar Angle: 58.43°

Azimuthal Angle: 24.20°

Comment: Sputtering of Ag, Au, and Cu samples was performed before checking the linearity of the binding energy scale.

DATA ANALYSIS METHOD

Energy Scale Correction: Linearity of the energy scale was performed recording the binding energy of Cu $2p_{3/2}$, Ag $3d_{5/2}$, and Au $4f_{7/2}$ signals on sputter cleaned high purity copper, silver, and gold foils (Goodfellow, Cambridge, UK). The binding energy values were compared with those reported in ISO 15472:2010. The C 1s of aliphatic carbon at 285.0 eV was taken as internal reference.

Recommended Energy Scale Shift: -0.16 eV for the five solvents' cleaned sample (# 01976) and -0.1 eV for the mechanically polished sample (# 01977).

Peak Shape and Background Method: Spectra were processed using CasaXPS software (V.2.3.25PR1.0) (Ref. 5). Shirley background subtraction was applied before curve fitting; the curve-fitting was carried out using mixed Gaussian - Lorentzian product function as line shape. In the case of Fe $2p_{3/2}$, the metallic component was fitted adding the tail for taking into account the asymmetry of the signal (Ref. 6).

Quantitation Method: N/A

ACKNOWLEDGMENTS

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Raouaa Hannachi: Data curation (equal); Investigation (equal); Writing – original draft (equal); Writing – review & editing (equal). **Deborah Biggio:** Data curation (equal); Investigation (equal); Writing – review & editing (equal). **Bernhard Elsener:** Conceptualization (equal); Data curation (equal); Investigation (equal); Methodology (equal); Writing – review & editing (equal). **Marzia Fantauzzi:** Conceptualization (equal); Data curation (equal); Investigation (equal); Methodology (equal); Writing – review & editing (equal). **Antonella Rossi:** Conceptualization (equal); Funding acquisition (equal); Investigation (equal); Methodology (equal); Project administration (equal); Supervision (equal); Writing – review & editing (equal).

DATA AVAILABILITY

The data that support the findings of this study are available within the article and its [supplementary material](#).

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SPECTRAL FEATURES TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01976-02	Fe 2p _{3/2}	Fe 2p high resolution spectrum
01976-02	Fe 2p _{3/2}	706.9	1.2	3 770.8	10.77	...	1-Fe (0)
01976-02	Fe 2p _{3/2}	710.6	2.3	17 332.5	19.16	...	2-iron oxide Fe ₂ O ₃
01976-02	Fe 2p _{3/2}	712.4	3.1	12 896.6	19.16	...	3-iron oxy hydroxide
01976-02	Fe 2p _{1/2}
01976-02	Fe 2p _{1/2}	720.0	4-Fe (0)
01976-02	Fe 2p _{1/2}	724.2	5-iron oxide Fe ₂ O ₃
01976-02	Fe 2p _{1/2}	725.9	6-iron oxy hydroxide
01976-03	C 1s	C 1s high resolution spectrum
01976-03	C 1s	285.0	1.5	6 770.3	1.89	...	1-aliphatic carbon
01976-03	C 1s	286.7	1.5	2 274.8	1.89	...	2-alcohol C—OH
01976-03	C 1s	288.1	1.5	1 105.8	1.89	...	3-carbonyl C=O
01976-03	C 1s	289.2	1.5	930.4	1.89	...	4-CO ₃ ²⁻ and/or carboxylic acid —COOH
01976-04	O 1s	O 1s high resolution spectrum
01976-04	O 1s	530.1	1.4	27 482.1	5.85	...	1-oxide
01976-04	O 1s	531.6	1.4	14 238.8	5.85	...	2-hydroxide
01976-04	O 1s	532.8	1.4	4 963.5	5.85	...	3-CO ₃ ²⁻ and/or organic contamination
01976-04	O 1s	534.1	1.4	2 571.9	5.85	...	4-adsorbed water
01977-02	Fe 2p _{3/2}	Fe 2p high resolution spectrum
01977-02	Fe 2p _{3/2}	706.8	1.3	5 940.6	10.77	...	1-Fe (0)
01977-02	Fe 2p _{3/2}	710.5	2.5	17 603.0	19.16	...	2-iron oxide Fe ₂ O ₃
01977-02	Fe 2p _{3/2}	712.4	3.3	10 943.4	19.16	...	3-iron oxy hydroxide
01977-02	Fe 2p _{1/2}
01977-02	Fe 2p _{1/2}	719.9	4-Fe (0)
01977-02	Fe 2p _{1/2}	724.1	5-iron oxide Fe ₂ O ₃
01977-02	Fe 2p _{1/2}	725.9	6-iron oxy hydroxide
01977-03	C 1s	C 1s high resolution spectrum
01977-03	C 1s	285.0	1.4	8 069.5	1.89	...	1-aliphatic carbon
01977-03	C 1s	286.6	1.4	1 722.0	1.89	...	2-alcohol C—OH
01977-03	C 1s	287.8	1.4	395.8	1.89	...	3-carbonyl C=O
01977-03	C 1s	289.1	1.4	894.3	1.89	...	4-CO ₃ ²⁻ and/or carboxylic acid —COOH
01977-04	O 1s	O 1s high resolution spectrum
01977-04	O 1s	530.1	1.4	24 447.5	5.85	...	1-oxide
01977-04	O 1s	531.5	1.4	10 460.7	5.85	...	2-hydroxide

27 November 2024 09:15:31

(Continued.)

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
01977-04	O 1s	532.5	1.4	4 882.0	5.85	...	3-CO ₃ ²⁻ and/or organic contamination
01977-04	O 1s	533.7	1.4	2 260.2	5.85	...	4-Adsorbed water

Comment to Spectral Features Table: The oxygen and carbon components listed in the table are due to the oxo-hydroxide layer and to surface contamination. The composition is not given because the assumption of homogeneity is not fulfilled.

Footnote to Spectrum # 01976-02: The Fe 2p spectrum consists of a Fe 2p_{3/2} and Fe 2p_{1/2} doublet due spin-orbit coupling. The Fe 2p_{3/2} signal shows three peaks located at 706.8, 710.5, and 712.4 eV that correspond to Fe (0) (Ref. 6) Fe₂O₃ and FeOOH, respectively (Ref. 6–10). The Fe 2p_{1/2} signal presents three peaks located at 720.0, 724.2, and 725.9 eV that are due to the presence of Fe (0), Fe₂O₃, and FeOOH, respectively.

Footnote to Spectrum # 01976-03: The presence of carbon is attributed to the organic contamination layer and, consequently, it is not considered in the steel composition. In the C 1s spectrum, peaks 1, 2, 3, and 4 correspond to C 1s aliphatic (BE = 285.0 eV), C—OH (BE = 286.7 eV) C=O (BE = 288.1 eV), and COOH (BE = 289.2 eV), respectively.

Footnote to Spectrum # 01976-04: The O 1s spectrum shows four peaks (1, 2, 3, and 4) that correspond to oxide (BE = 530.0 eV), hydroxide (BE = 531.4 eV), oxygen in CO₃²⁻ and/or organic contamination (BE = 532.2 eV), and adsorbed water (BE = 533.6 eV).

Footnote to Spectrum # 01977-02: The Fe 2p spectrum of MP X60 steel consists of a Fe 2p_{3/2} and Fe 2p_{1/2} doublet due spin-orbit coupling. Also in this case, Fe 2p_{3/2} signal shows three peaks located at 706.8, 710.5, and 712.4 eV that are assigned to Fe (0), Fe₂O₃, and FeOOH, respectively. The Fe 2p_{1/2} signal consists of three peaks located at 719.9, 724.1, and 725.9 eV due to Fe (0), Fe₂O₃, and FeOOH, respectively. No differences in the chemical state of iron are observed for the cleaned and MP X60 samples.

Footnote to Spectrum # 01977-03: The C 1s spectrum of the MP X60 steel presents four peaks 1, 2, 3, and 4 that correspond to C 1s aliphatic (BE = 285.0 eV), C—OH (BE = 286.6 eV), C=O (BE = 287.8 eV), and COOH (BE = 289.1 eV), respectively. When comparing the cleaned X60 steel to the MP X60 steel, it can be observed that the measured binding energies are equal within experimental uncertainty (0.1 eV).

Footnote to Spectrum # 01977-04: The O 1s spectrum of the MP X60 steel shows four peaks (1, 2, 3, and 4) that correspond to oxide (BE = 530.0 eV), hydroxide (BE = 531.5 eV), oxygen in organic compounds from CO₃²⁻ and/or organic contamination layer (BE = 532.5 eV), and adsorbed water (BE = 533.7 eV), respectively.

No significant difference in the chemical state of the elements is detected between the cleaned and mechanically polished samples.

ANALYZER CALIBRATION TABLE

Spectrum ID #	Element/Transition	Peak Energy (eV)	Peak Width FWHM (eV)	Peak Area (eV × counts)	Sensitivity Factor	Concentration (at. %)	Peak Assignment
...	Au 4f _{7/2}	83.95	1.04	191 600	Gold metal
...	Ag 3d _{5/2}	368.26	0.96	197 000	Silver metal
...	Cu 2p _{3/2}	932.61	1.24	143 000	Copper metal

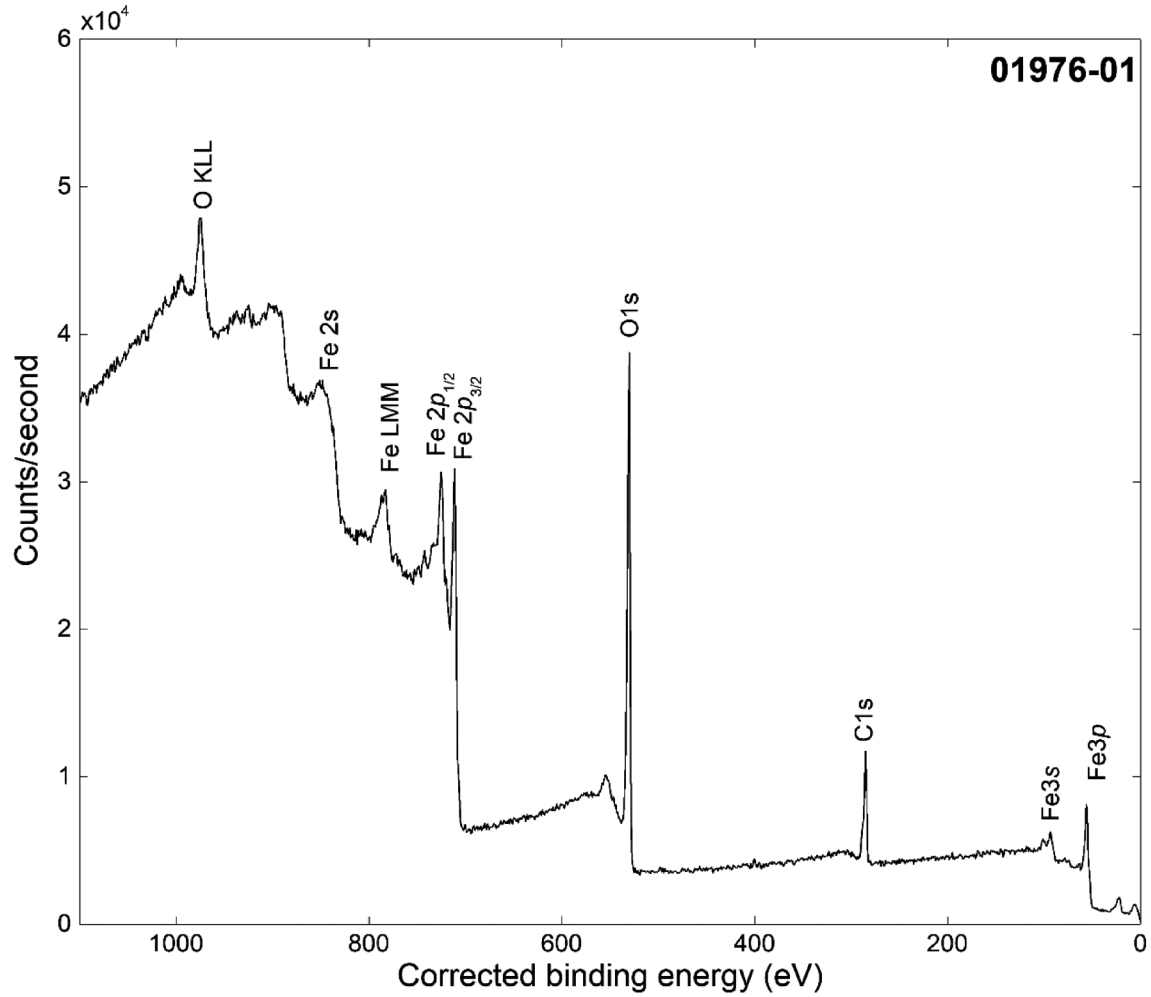
Comment to Analyzer Calibration Table: The spectra were acquired after Ar⁺ ion etching.

GUIDE TO FIGURES

Spectrum ID #	Element/Transition	Voltage Shift ^a	Multiplier	Baseline	Comment #
01976-01	Survey	0.16	1	0	...
01976-02	Fe 2p	0.16	1	0	...
01976-03	C 1s	0.16	1	0	...
01976-04	O 1s	0.16	1	0	...
01977-01	Survey	0.1	1	0	...
01977-02	Fe 2p	0.1	1	0	...
01977-03	C 1s	0.1	1	0	...
01977-04	O 1s	0.1	1	0	...

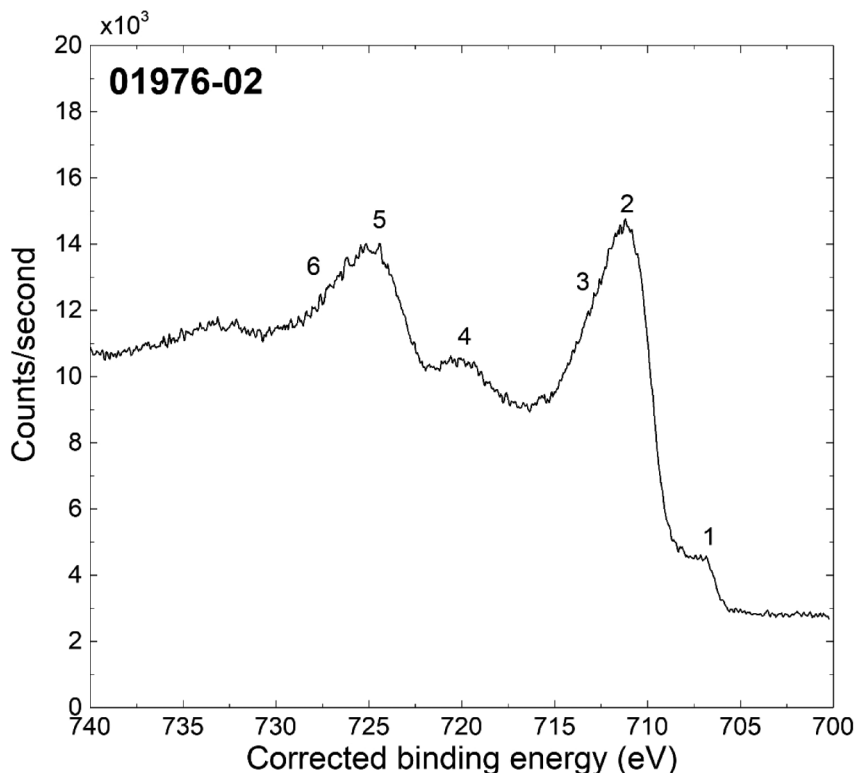
^aVoltage shift of the archived (as-measured) spectrum relative to the printed figure. The figure reflects the recommended energy scale correction due to a calibration correction, sample charging, flood gun, or other phenomenon.

27 November 2024 09:15:31



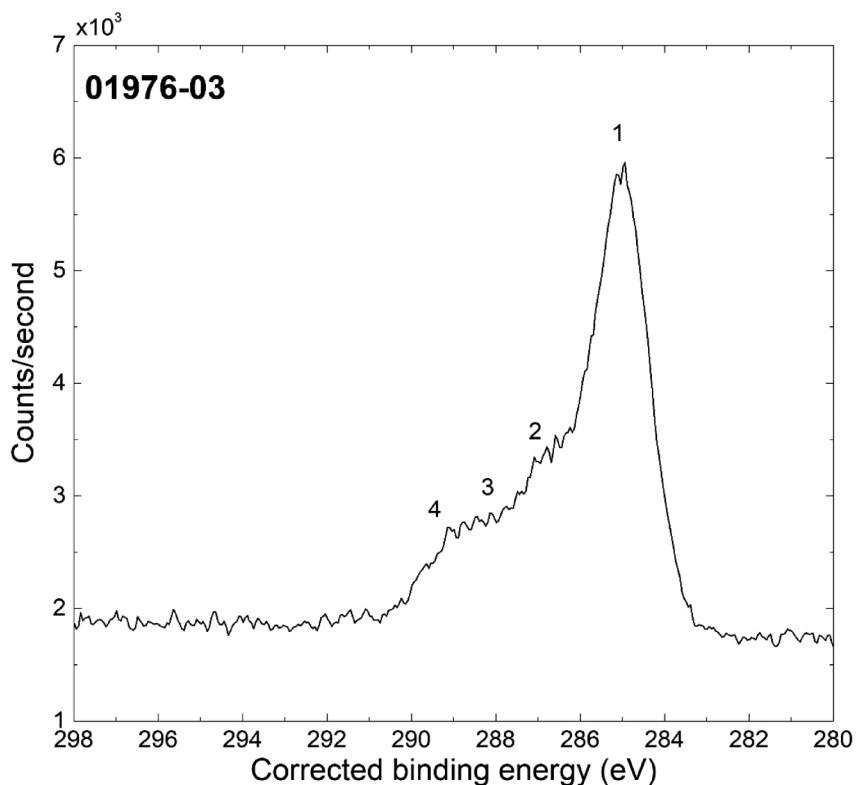
27 November 2024 09:15:31

Accession #:	01976-01
Specimen:	Cleaned X60 steel
Technique:	XPS
Spectral Region:	Survey
Instrument:	Thermo Scientific Theta Probe
Excitation Source:	Al K_{α} monochromatic
Source Energy:	1486.6 eV
Source Strength:	100 W
Source Size:	0.4 × 0.4 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	30°
Emission Angle:	53°
Analyzer Pass Energy:	200 eV
Instrument Resolution:	1.5 eV
Total Signal Accumulation Time:	612 s
Total Elapsed Time:	985 s
Number of Scans:	9



- Accession #: [01976-02](#)
- Specimen: Cleaned X60 steel
- Technique: XPS
- Spectral Region: Fe 2p

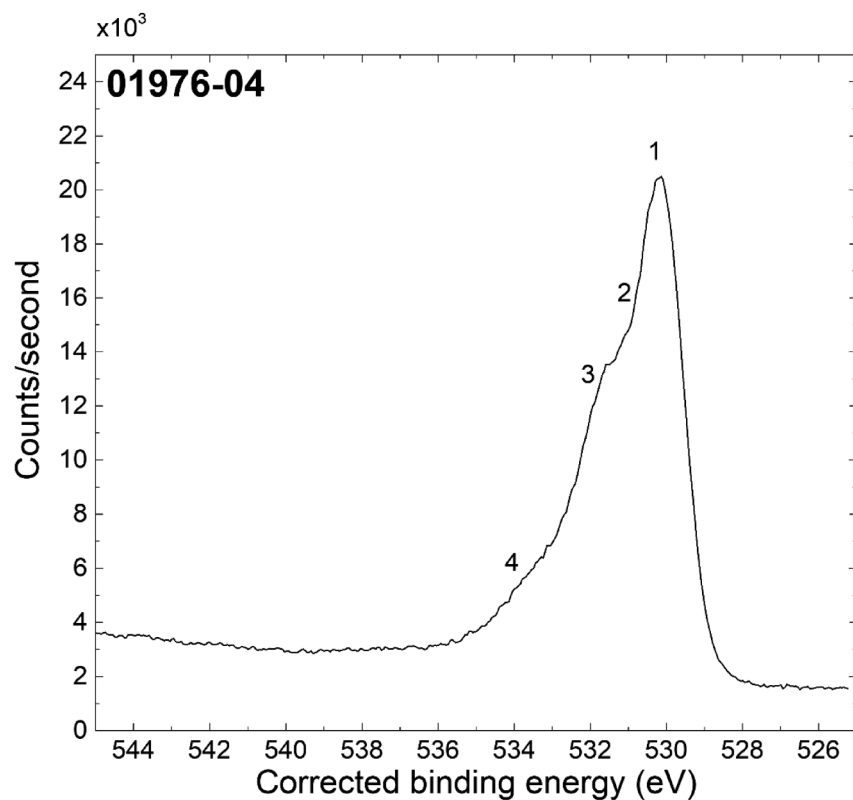
Instrument: Thermo Scientific Theta Probe
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 100 W
 Source Size: $0.4 \times 0.4 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 30°
 Emission Angle: 53°
 Analyzer Pass Energy: 100 eV
 Instrument Resolution: 0.96 eV
 Total Signal Accumulation Time: 360 s
 Total Elapsed Time: 647 s
 Number of Scans: 9
 Comments: See the table's footnotes



- Accession #: [01976-03](#)
- Specimen: Cleaned X60 steel
- Technique: XPS
- Spectral Region: C 1s

Instrument: Thermo Scientific Theta Probe
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 100 W
 Source Size: $0.4 \times 0.4 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 30°
 Emission Angle: 53°
 Analyzer Pass Energy: 100 eV
 Instrument Resolution: 0.96 eV
 Total Signal Accumulation Time: 171 s
 Total Elapsed Time: 383 s
 Number of Scans: 9
 Comments: See the table's footnotes

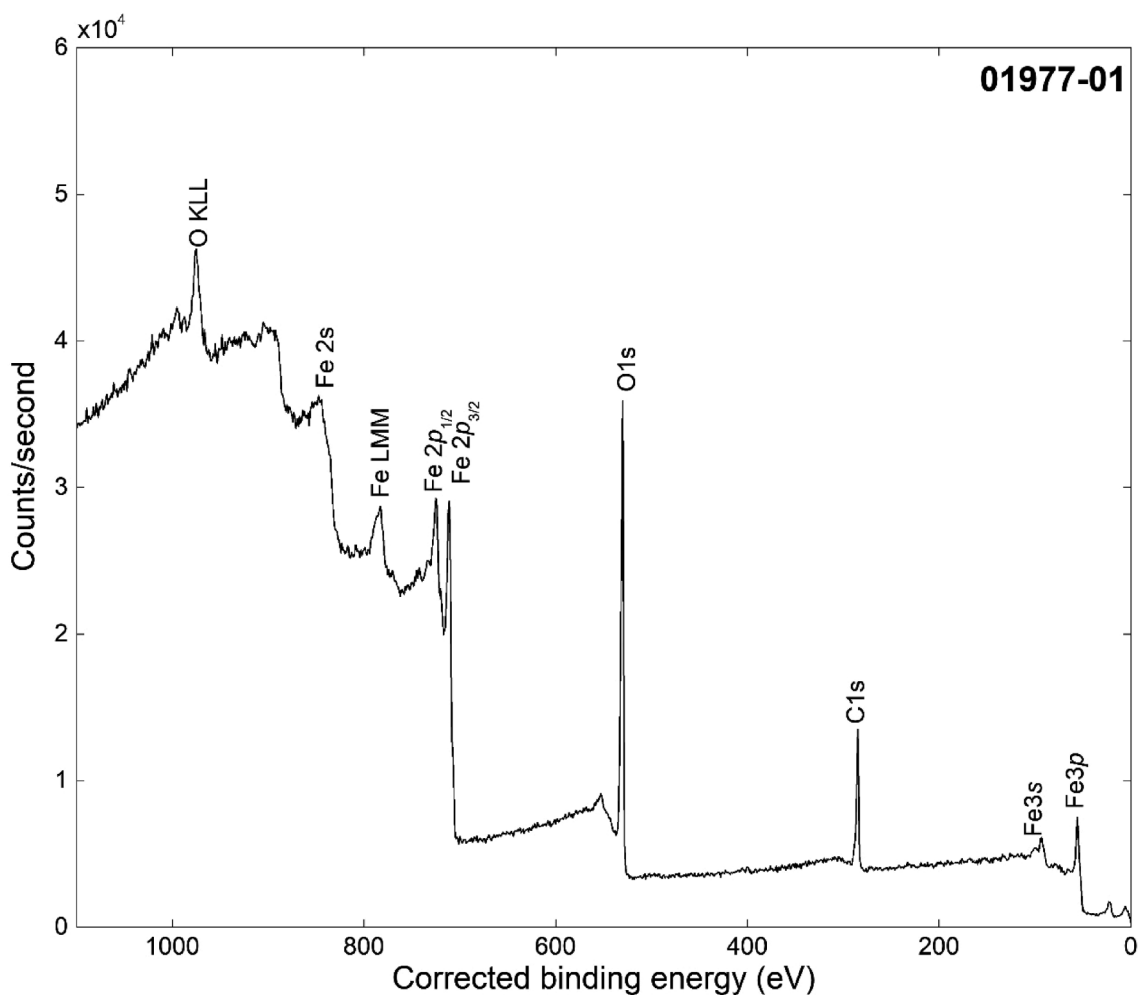
27 November 2024 09:15:31



- Accession #: [01976-04](#)
- Specimen: Cleaned X60 steel
- Technique: XPS
- Spectral Region: O 1s

Instrument: Thermo Scientific Theta Probe
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 100 W
 Source Size: $0.4 \times 0.4 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 30°
 Emission Angle: 53°
 Analyzer Pass Energy: 100 eV
 Instrument Resolution: 0.96 eV
 Total Signal Accumulation Time: 180 s
 Total Elapsed Time: 389 s
 Number of Scans: 9
 Comments: See the table's footnotes

27 November 2024 09:15:31

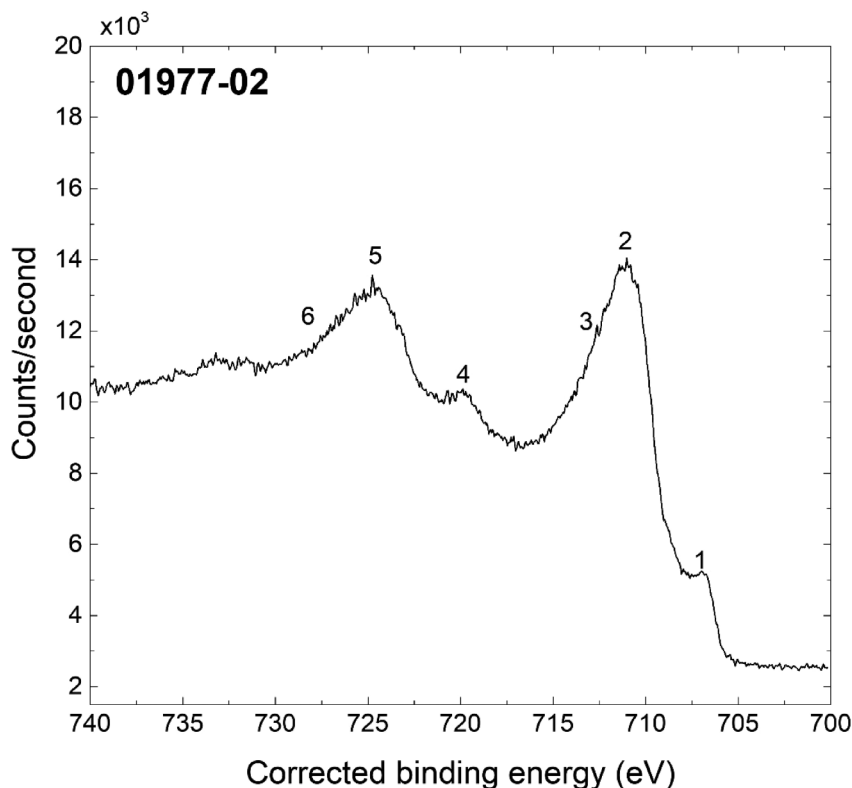


27 November 2024 09:15:31

Accession #:

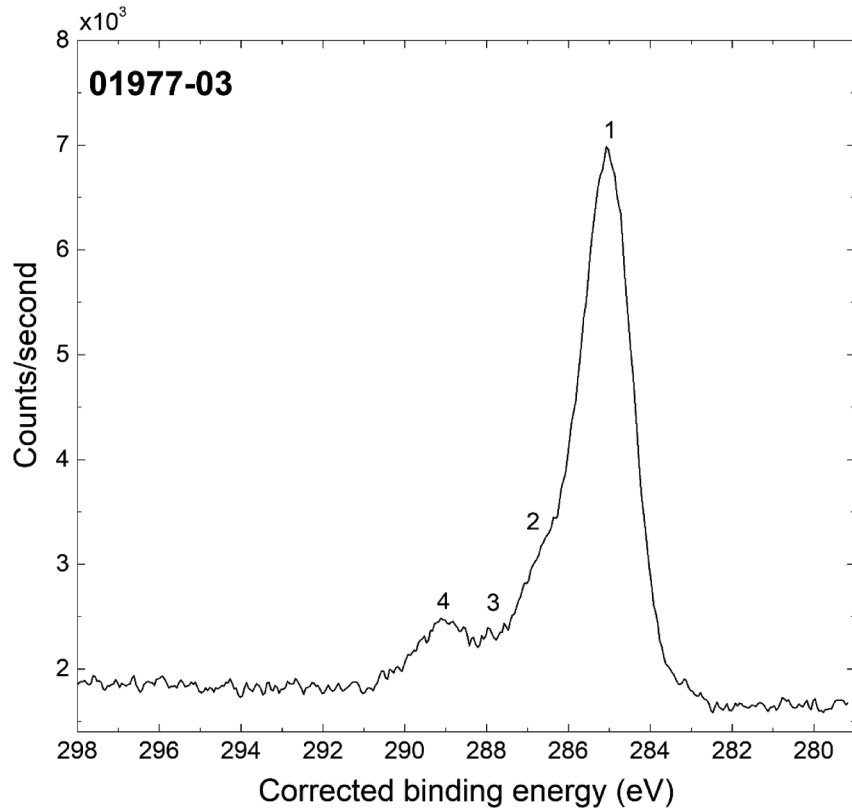
01977-01

■ Specimen:	MP X60 steel
■ Technique:	XPS
■ Spectral Region:	Survey
Instrument:	Thermo Scientific Theta Probe
Excitation Source:	Al K_{α} monochromatic
Source Energy:	1486.6 eV
Source Strength:	100 W
Source Size:	0.4 × 0.4 mm ²
Analyzer Type:	Spherical sector analyzer
Incident Angle:	30°
Emission Angle:	53°
Analyzer Pass Energy:	200 eV
Instrument Resolution:	1.5 eV
Total Signal Accumulation Time:	612 s
Total Elapsed Time:	985 s
Number of Scans:	9



- Accession #: [01977-02](#)
- Specimen: MP X60 steel
- Technique: XPS
- Spectral Region: Fe 2p

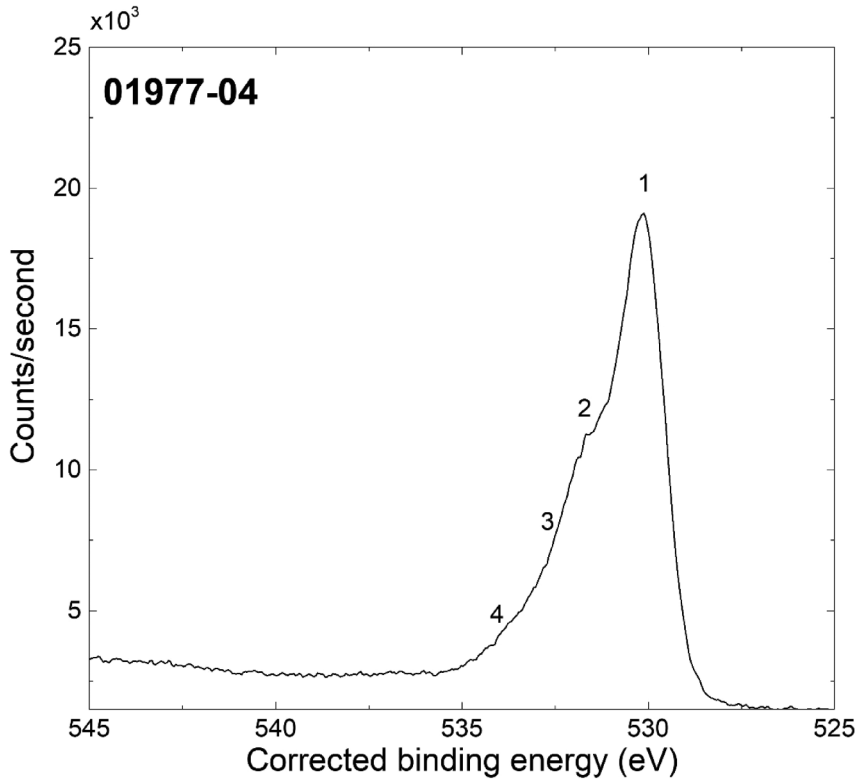
Instrument: Thermo Scientific Theta Probe
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 100 W
 Source Size: $0.4 \times 0.4 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 30°
 Emission Angle: 53°
 Analyzer Pass Energy: 100 eV
 Instrument Resolution: 0.96 eV
 Total Signal Accumulation Time: 360 s
 Total Elapsed Time: 647 s
 Number of Scans: 9
 Comments: See the table's footnotes



- Accession #: [01977-03](#)
- Specimen: MP X60 steel
- Technique: XPS
- Spectral Region: C 1s

Instrument: Thermo Scientific Theta Probe
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 100 W
 Source Size: $0.4 \times 0.4 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 30°
 Emission Angle: 53°
 Analyzer Pass Energy: 100 eV
 Instrument Resolution: 0.96 eV
 Total Signal Accumulation Time: 171 s
 Total Elapsed Time: 383 s
 Number of Scans: 9
 Comments: See the table's footnotes

27 November 2024 09:15:31



- Accession #: [01977-04](#)
- Specimen: MP X60 steel
- Technique: XPS
- Spectral Region: O 1s

Instrument: Thermo Scientific Theta Probe
 Excitation Source: Al K_{α} monochromatic
 Source Energy: 1486.6 eV
 Source Strength: 100 W
 Source Size: $0.4 \times 0.4 \text{ mm}^2$
 Analyzer Type: Spherical sector
 Incident Angle: 30°
 Emission Angle: 53°
 Analyzer Pass Energy: 100 eV
 Instrument Resolution: 0.96 eV
 Total Signal Accumulation Time: 180 s
 Total Elapsed Time: 389 s
 Number of Scans: 9
 Comments: See the table's footnotes

27 November 2024 09:15:31