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Evaluation of the Residue from Microset on Various Metal Surfaces

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Evaluation of the Residue from Microset Cure Elastomer on Metals

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Abstract

Fast-curing impression materials are sometimes used to cast negative-mold replications of physical defects on material surfaces. The negative-mold impressions can then be used for further measurements to record the nature of the defect. These impression materials have been designed to cure quickly, and with very low adhesion, so that they can be easily removed from the surface leaving little residual contamination. Unfortunately, some contaminant is retained by the substrate material. This investigation seeks to identify the composition and quantity of the remaining material upon removal of Microset Synthetic Rubber Replicating Compound from several material surfaces. Coe-Flex was used as a relative comparison to Microset. On fifteen different substrate materials the Microset leaves no visible trace of contaminant, however, X-ray photoelectron spectroscopy shows evidence of a thin silicone-based contaminant film of approximately 2 nm thickness.

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NOMENCLATURE

IPA	isopropanol
XPS	X-ray photoelectron spectroscopy
SNL	Sandia National Laboratories
RMS	root mean squared
Microset	Microset Synthetic Rubber Replicating Compount
Coe-Flex	Coe-Flex Polysulfide Impression Material
MSDS	Material safety data sheet

1. INTRODUCTION

Fast-curing impression materials are sometimes used to cast negative-mold replications of physical defects, scratches, dents, etc., on a variety of material surfaces. The negative-mold impressions can then be used for further measurements to determine quantifiable properties of the defect, such as length and depth of a scratch. These impression materials have been designed to cure quickly, within minutes or seconds, and with very low adhesion so that they can be easily removed from the surface leaving little residual contamination. Due to the low adhesion of the materials, they can also be effectively removed from high aspect ratio defects, such as a very deep scratch. Unfortunately, these molds are not 100% removed when peeled away from the material surface. Some residual contaminant is retained by the substrate material. This investigation seeks to identify the composition and quantity of the remaining material upon removal of Microset Synthetic Rubber Replicating Compound from several material surfaces. Coe-Flex has been used on some substrates as a relative comparison to Microset.

XPS was used to determine the composition of the residual contaminant film. Intensity data from XPS was also used to provide approximate thicknesses of the residual film. Ellipsometry was used to corroborate thickness estimations.

The Microset replicating compound was applied to fifteen different substrate materials followed by removal of the cured impression. The Microset leaves no visible trace of contaminant on the substrate surface, however, XPS showed a silicone-based contaminant film of approximately 2 nm thickness on all samples. Coe-Flex tends to leave a thicker residual film, which contains copper and chlorine as contaminant species.

A simple cleaning procedure was used to reduce the level of contaminant at the surface. The various surfaces were wiped with isopropanol following initial XPS characterization. The IPA wipe reduces the contaminant levels on nearly all substrates.

2. METHODS

The basic procedure for this study involved:

1. Cleaning the substrate.
2. Applying Microset (or Coe-Flex).
3. Allowing time for replicate curing.
4. Marking the location of the Microset (or Coe-Flex) followed by removal of the replicate by peeling it away from the surface.
5. XPS analyses was performed on three locations of the pristine substrate surface as well as three locations within the region where Microset (or Coe-Flex) had been applied.
6. Samples were then removed from the analysis chamber and were wiped with a Kimwipe soaked in isopropanol
7. Three locations in the Microset (or Coe-Flex) region were then reanalyzed.

2.1. Samples

Fifteen materials were used for the study as shown in Table 1. One inch squares of each of the different materials were cut from the material stock. Samples were labeled on backside for identification. The surfaces of samples A-F were polished to a one micron finish.

Table 1. Sample identifications.

I.D.	Material
A	304
B	304L
C	316
D	316L
E	21-6-9
F	Ti-Al alloy
G	Ti / 6% Al / 4% V
H	Al 6061-T6 Bare
I	Al 7075-T6 Bare
J	Al 6061-T6 Anodized
K	Al 7075-T6 Anodized
L	Al 6061-T6 Alodine 1997
M	Al 7075-T6 Alodine 1997
N	Al 6061-T6 Alodine 047
O	Al 6061-T6 Alodine-new 2004

Microset 101RT Synthetic Rubber Replicating compound was used from Microset Products Ltd., 261 Weddington Road, Nuneaton, Warwickshire, UK. Coe-Flex was used from GC America, Inc. 3737 West 127th St., Alsip, IL, 60803.

Microset 101RT Synthetic Rubber Replicating Compound was used with an application gun and static mixers. The material was used before its expiration date. The MSDS for Micrsoet is

vague but discusses the presence of siloxanes, silicones, glycol, silica, carbon, and complexed platinum. The density of the material is given as 1.1 g/cm^3.

Coe-Flex polysulfide impression material Type 2, medium-bodied consistency, regular type, was used as a control. The Coe-Flex requires mixing of the base and catalyst from two separate tubes. According the to the MSDS for the Coe-Flex, the catalyst material contains 10-30% Cupric Hydroxide, 10-35% TiO_2, 1-5% SiO_2, and 30-50% chlorinated fatty acid ester. The MSDS for the Coe-Flex base material only discusses the fillers, TiO_2 10-25% and SiO_2 1-5%. The density of the Coe-Flex base is given as 1.3-1.6 g/cm^3.

2.2. Cleaning and Curing

The fifteen samples were cleaned by sonication in a solution of Brulin (815 GD-NF) GD detergent.

A small aliquot of Microset was applied to the sample using the application gun. Microset paper was placed on top of the drop of Microset, and finger pressure was used to give a thin (1 mm) impression on the surface. Microset films were allowed to cure for two days. The location of the Microset was scratched into the metal surface. The Microset film was then peeled away from the substrate. Regions of the substrate were left bare to be used as the representative cleaned surface for XPS analysis.

A similar procedure was used for Coe-Flex, however, Coe-Flex required mixing of the base and catalyst in a separate container prior to application. The ratio is 2 parts base to 1 part catalyst (by volume). A spatula was used to transfer mixed Coe-Flex to the sample, followed by pressing on the un-cured Coe-Flex to give a thin mold on the surface. Coe-Flex was also allowed to cure for two days at room temperature. For XPS analysis, Coe-Flex was used on samples A, I, and J to give relative comparisons to Microset Coe-Flex was not used on all samples due to time constraints of the study.

2.3. Analysis Methods

Profilometry (Veeco Dektak 150) was performed to determine surface roughness in several regions on each sample. Six segments were analyzed at various locations on each sample as shown in Figure 1.

Ellipsometry (Nanofilm EP3 with EP3 View software) was used to determine thin film thicknesses. More details on ellipsometry are given below.

XPS was performed using a Kratos Axis Ultra DLD instrument with a monochromatic Al kα (1486.6 eV) source. Only full survey spectra were collected with an analyzer pass energy of 160 and a step size of 1 eV. Three locations were analyzed in each region on all samples to determine the average surface compositions. This gave a total of 9 analyses for each sample: 3 spectra of the cleaned surface, 3 spectra of the residual contaminant film, and 3 spectra of the residual contaminant film after IPA wiping.

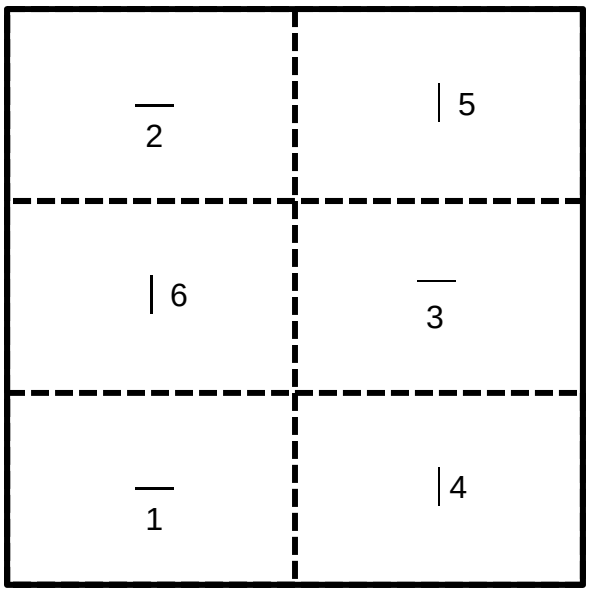


Figure 1. Profilometry was performed on each sample in one micron segments at six locations as indicated. Three measurements were performed perpendicular to the other three measurements.

3. RESULTS

3.1. Profilometry

Profilometry was performed to determine the approximate roughness of the samples. Roughness can impact the XPS measurements by influencing the measured atomic concentrations as well as the thickness calculations. Surface roughness was also measured to evaluate any correlation between surface roughness and the amount of residual contamination left by Microset and/or Coe-Flex. Samples A-F were relatively smooth compared to the aluminum samples which had RMS roughness values which were 1-2 orders of magnitude greater. Despite the variations in roughness, no clear correlation could be made between RMS roughness and the amount of residual contaminant from Microset.

Table 2. Roughness approximations based on profilometry.

	RMS Roughness (nm)
A	12 ± 4
B	15 ± 4
C	12 ± 3
D	No data
E	9 ± 1
F	No data
G	160 ± 50
H	2900 ± 400
I	100 ± 10
J	500 ± 160
K	270 ± 70
L	430 ± 200
M	670 ± 170
N	3100 ± 200
O	3400 ± 600

3.2. XPS

XPS was performed on cured Microset and Coe-Flex materials to determine their elemental constituents. The relative atomic concentrations of the identifiable species are shown in Table 3.

Table 3. Relative atomic concentrations of Microset and Coe-Flex as measured by XPS.

	[C]	[Si]	[O]	[Cl]	[S]	[Cu]	[Na]	[Ti]	[N]
Microset	50	30	20						
Coe-Flex	70	4	10	10	2	1	< 1	<1	<1

Residual contaminant films from Microset contain C, O, and Si with no other elemental components. Residual contaminant films from Coe-Flex contain C, Cu, Si, O, S, Cl, N, and Na. Only Cu and Cl are shown in Figures 2-15, since the other constituents were either low in concentration (<1%), were not observed consistently among samples, and/or were not affected significantly by isopropanol wiping.

XPS only evaluates to a depth of 5-10 nm for most materials and the presence of adventitious contamination, surface segregation, or surface layers can give elemental compositions which may differ from that of the bulk material (bulk compositions summarized in the Appendix). All surfaces which have been exposed to air, which includes all of the materials analyzed in this study, will have an adventitious contaminant layer of adsorbed species composed of oxygen and carbon. Only the major elemental constituents, or elements which have shown significant differences between sample preparations, are shown in Figures 2-15. The concentrations are only for the near-surface region evaluated by XPS and do not necessarily relate to the composition of the bulk substrate. For this study the elemental constituents and exact compositions are not important. The most important observations are the relative changes in elemental concentrations from the as-cleaned surface compared to the compositions obtained with the presence of the residual contaminant films, as well as the changes observed after wiping the surface with isopropanol (IPA).

XPS results for residual films on the various metal surfaces show an increase in the apparent concentrations of the residual film components (i.e. Si, C, Cu, Cl, etc.). The presence of the overlayer results in a decrease in the apparent concentration of constituents of the substrate material (i.e. Al, Fe, Ti, etc.).

3.2.1. *XPS Results for Sample A - 304*

Carbon increases after removal of Microset and Coe-Flex impression materials, relative to the initial cleaned surface (~15%), due to the presence of residual films. The increase in carbon is greater for Coe-Flex than for Microset. The Microset residual film results in a large increase in silicon, however, the silicon can be effectively reduced by wiping the surface with isopropanol (IPA). Coe-Flex leaves residual Cl and Cu on the surface. The Cu and Cl can be partially removed with IPA wiping, however, the carbonaceous component of the film remains virtually unchanged. Cr and Fe concentrations are attenuated by the overlaying residual films, but IPA wiping partially removes the overlayer leading to an apparent increase in their atomic concentrations. Attenuation of the substrate signals can be used for thickness estimations.

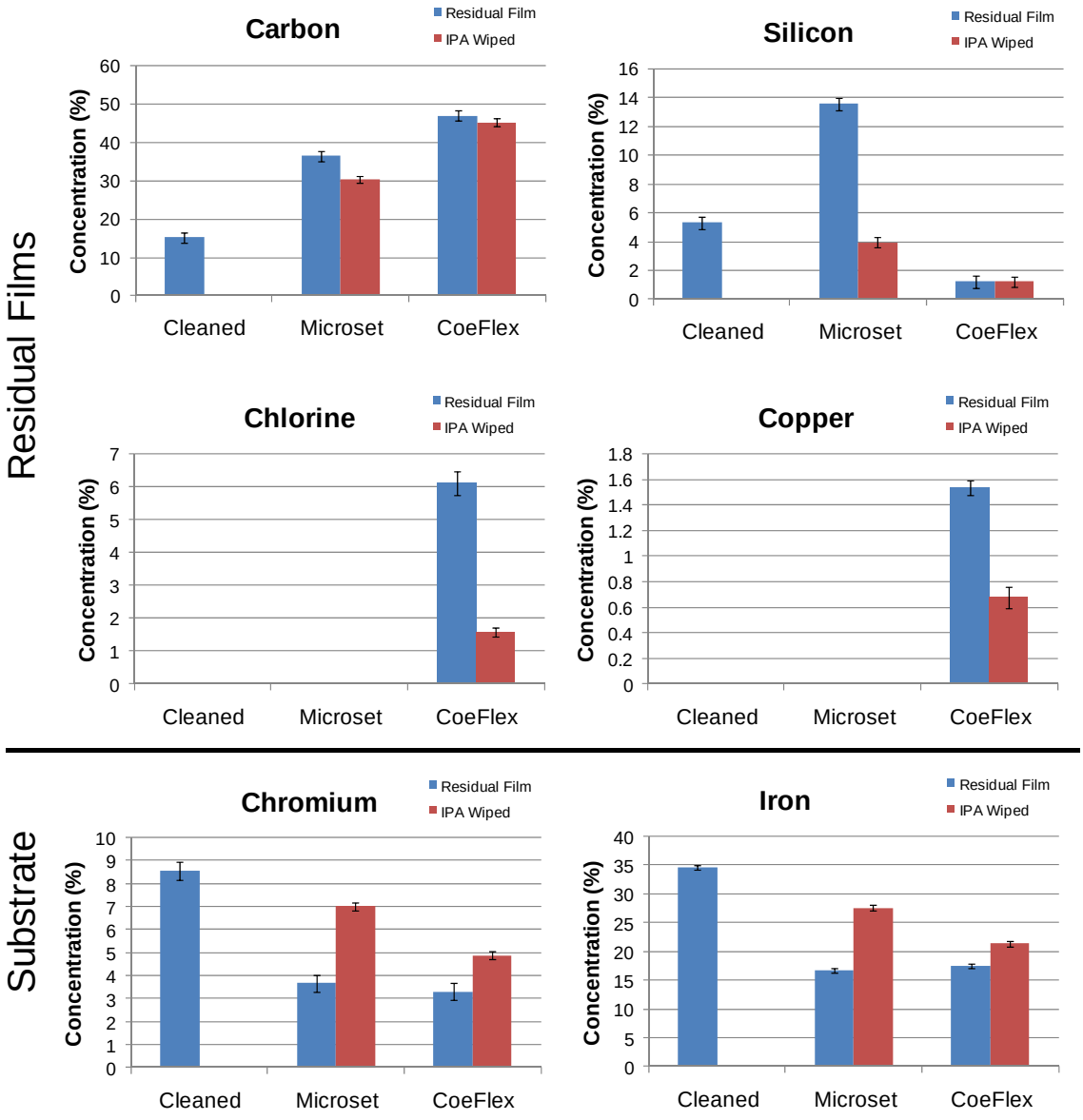


Figure 2. Sample A, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, Coe-Flex residual film, and the residual films after wiping the surface with isopropanol.

3.2.2. *XPS Results for Sample B – 304L*

Carbon and silicon increase due to the Microset residual film, relative to the initial cleaned surface. The silicon can be effectively reduced by wiping the surface with isopropanol (IPA), however the carbon level remains high. The remaining high level of carbon does not necessarily relate to residual Microset, it may also be due to other carbonaceous contaminants introduced by the IPA wiping process (possibly including IPA itself).

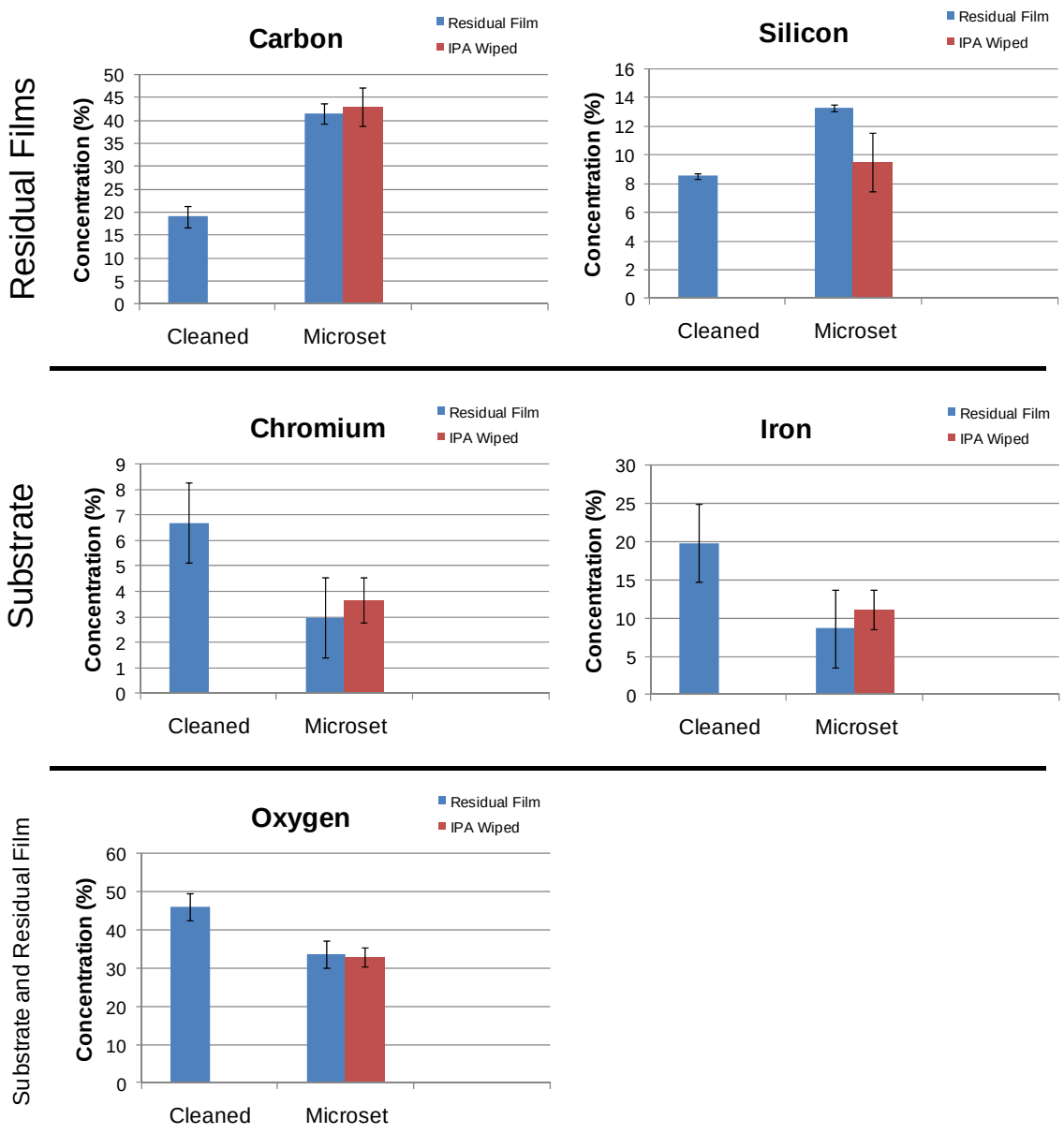


Figure 3. Sample B, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.3. *XPS Results for Sample C - 316*

Results for Sample C are very similar to those observed from Sample B.

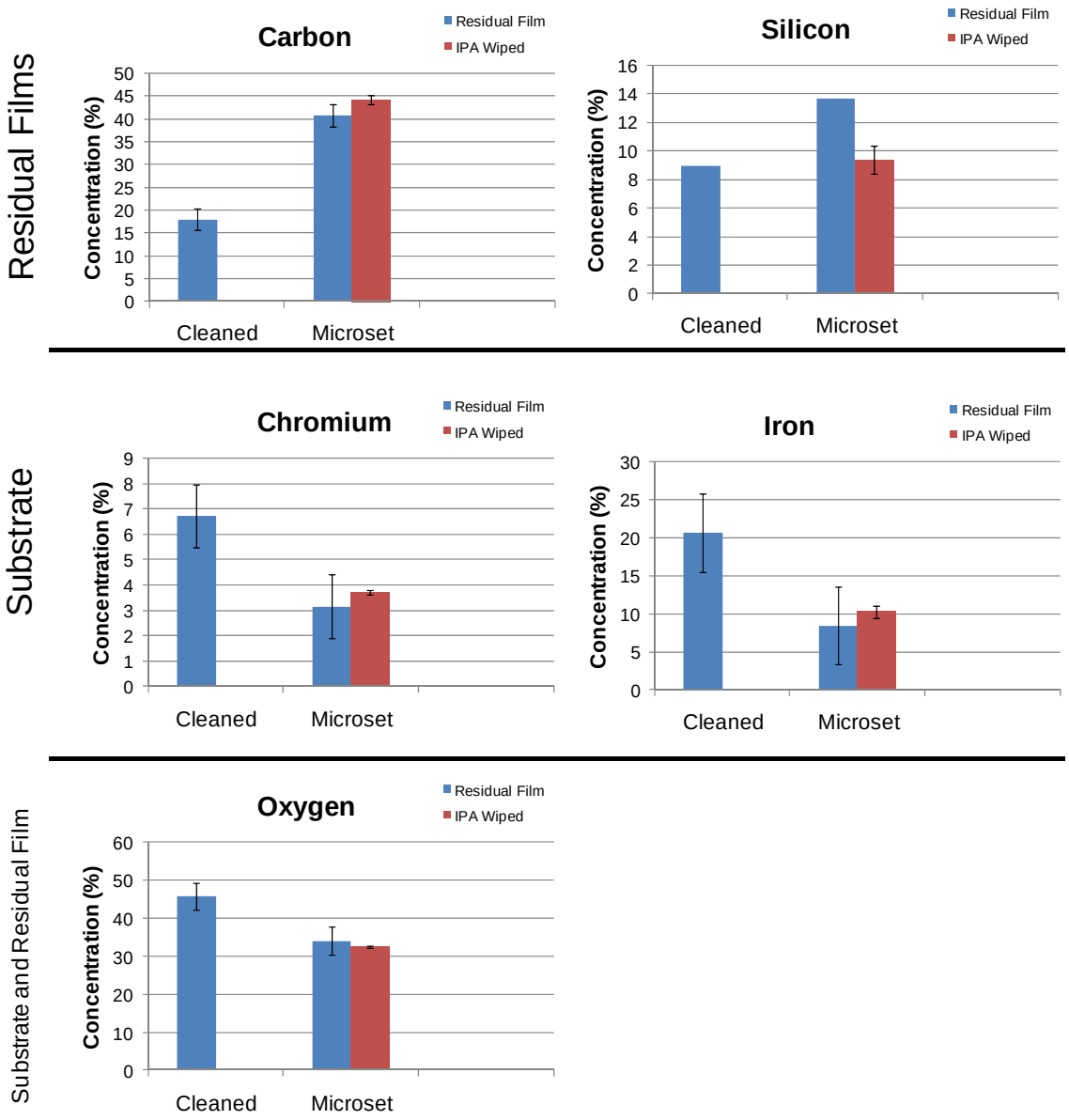


Figure 4. Sample C, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.4. *XPS Results for Sample D – 316L*

Carbon and silicon increase due to the Microset residual film, relative to the initial cleaned surface. The silicon can be effectively reduced by wiping the surface with isopropanol (IPA), however the carbon level remains high. The increase in substrate signals after IPA wiping is greater for this sample than was observed for Sample B and C.

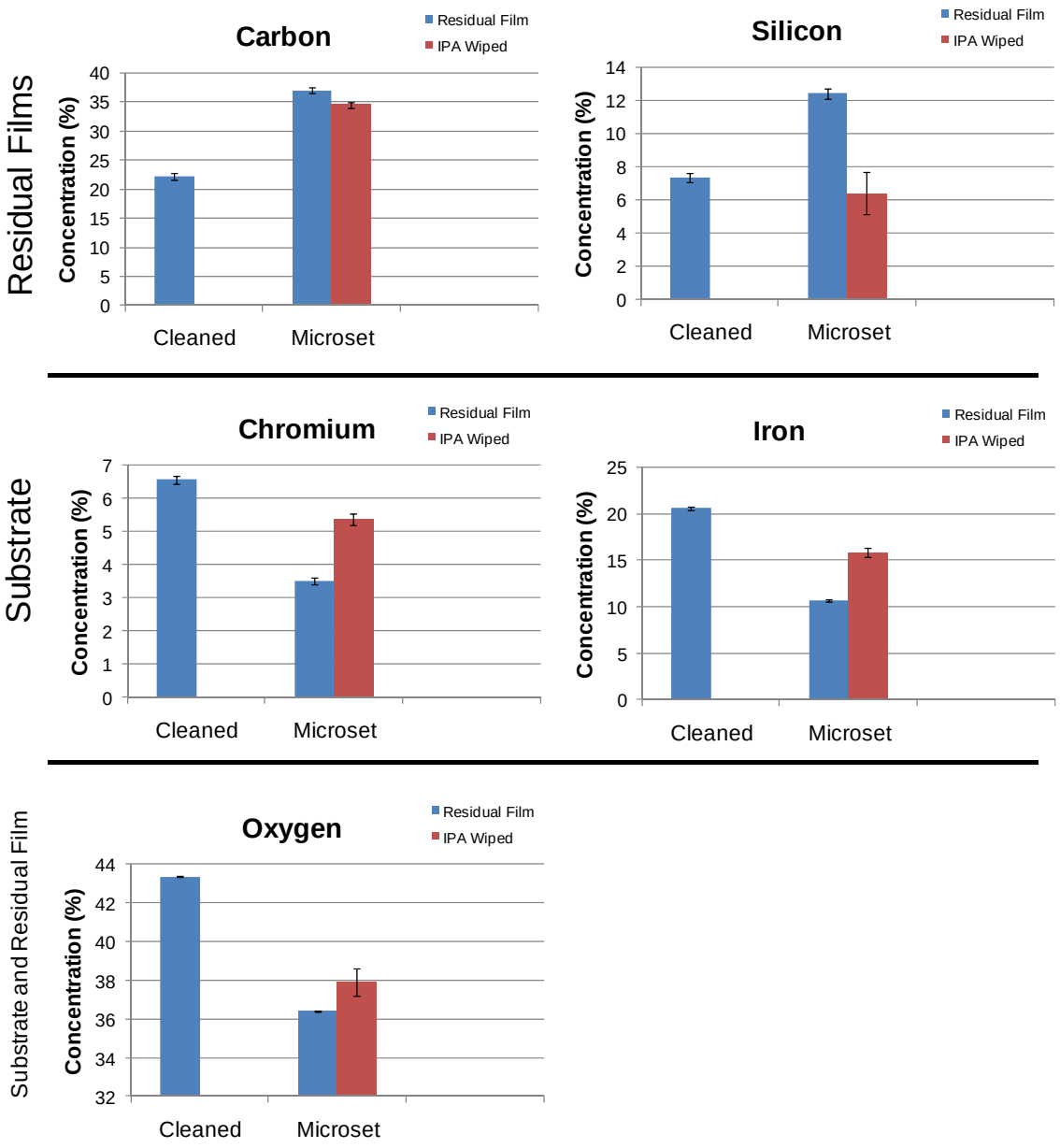


Figure 5. Sample D, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.5. *XPS Results for Sample E – 21-6-9*

Carbon and silicon increase due to the Microset residual film, relative to the initial cleaned surface. The silicon can be effectively reduced by wiping the surface with isopropanol (IPA), however the carbon level remains high. The remaining high level of carbon does not necessarily relate to residual Microset, it may also be due to other carbonaceous contaminants introduced by the IPA wiping process (possibly including IPA itself).

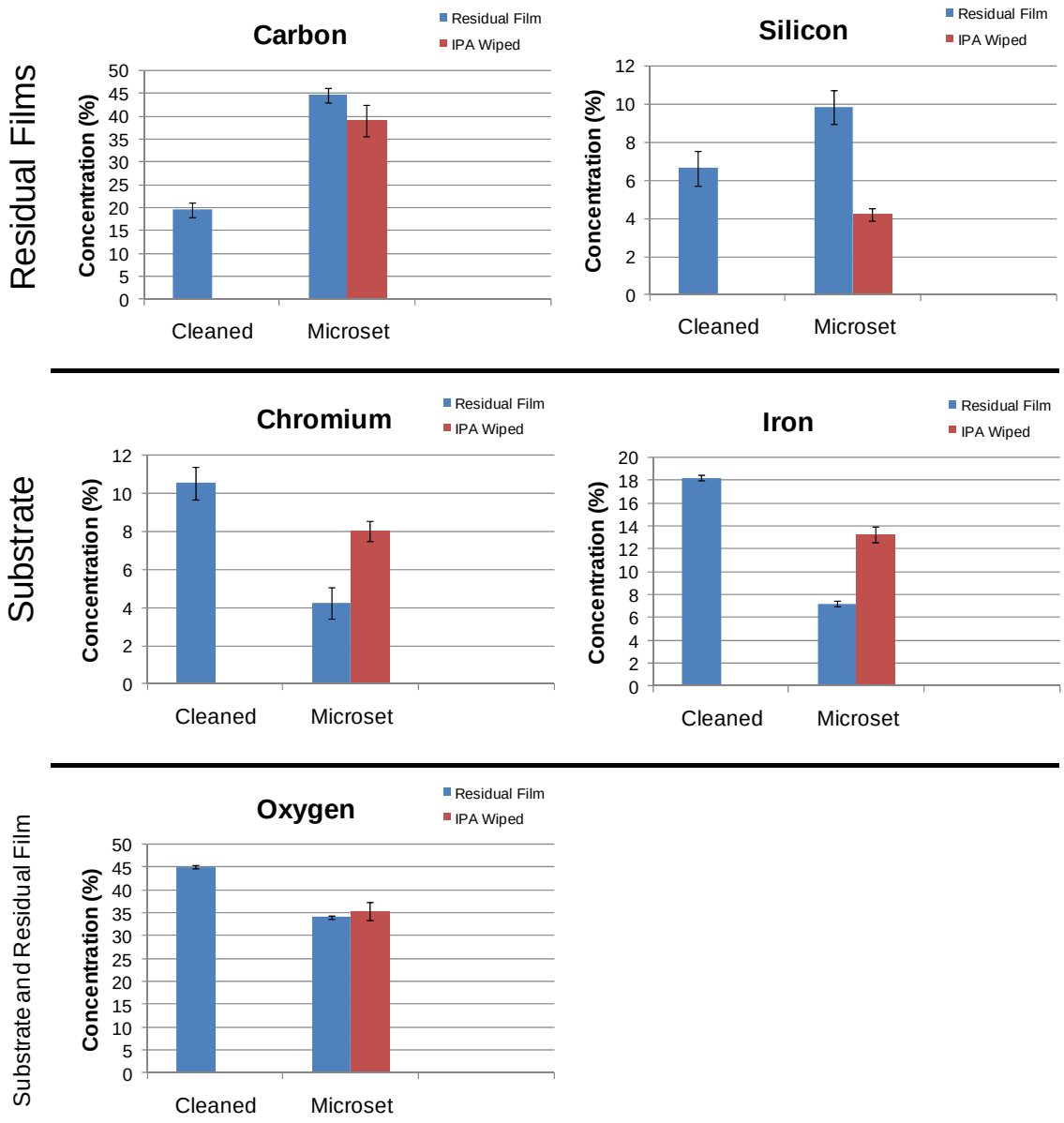


Figure 6. Sample E, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.6. *XPS Results for Sample F – Ti/Al alloy*

The Microset residual film results in a dramatic increase in silicon content, however, it can be very effectively removed by the IPA wiping procedure.

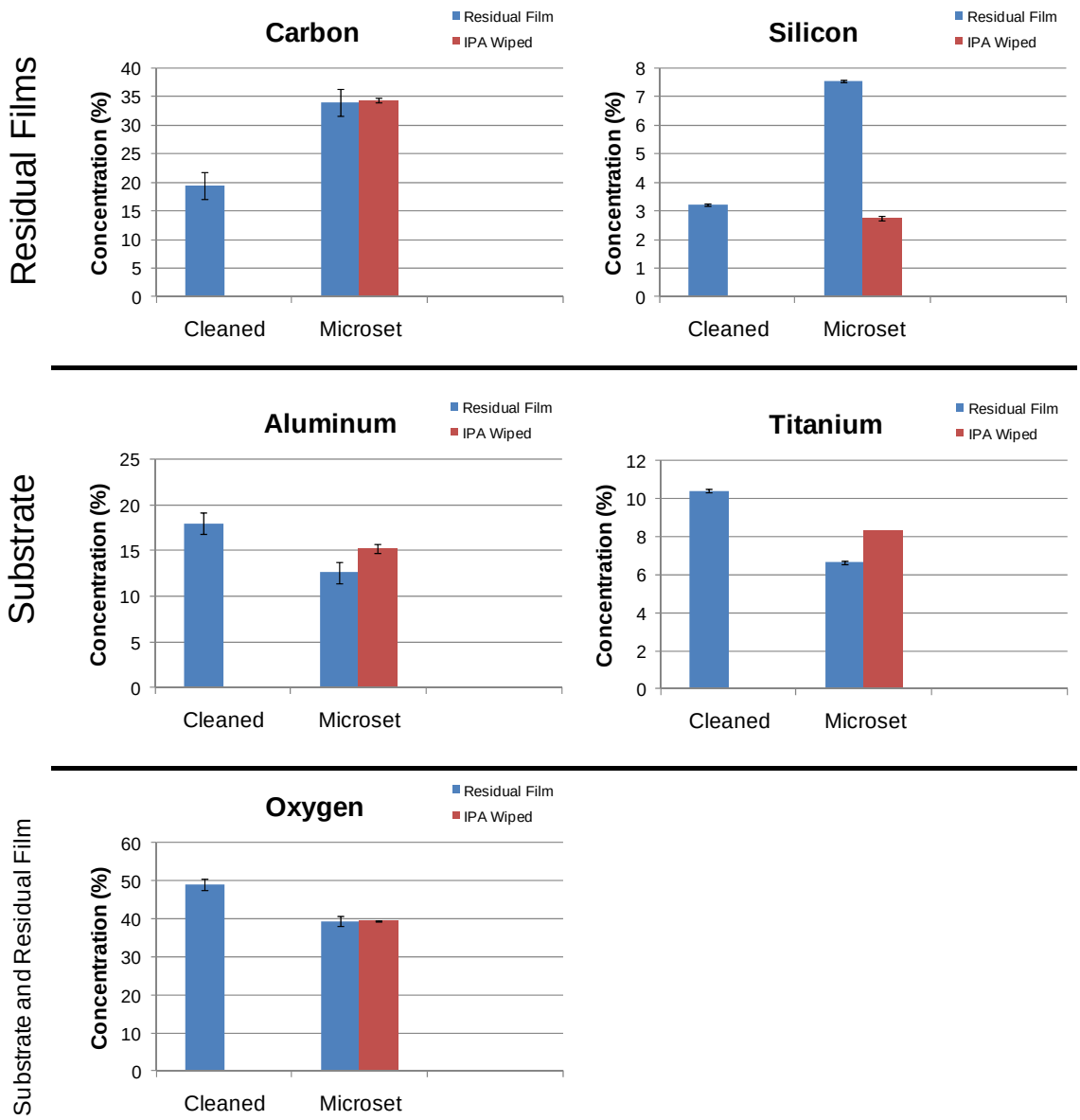


Figure 7. Sample F, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.7. *XPS Results for Sample G – Ti/Al/V Alloy*

Carbon and silicon increase due to the Microset residual film, relative to the initial cleaned surface. The silicon can be reduced by IPA wiping, but not as effectively as has been seen for previous samples A-F.

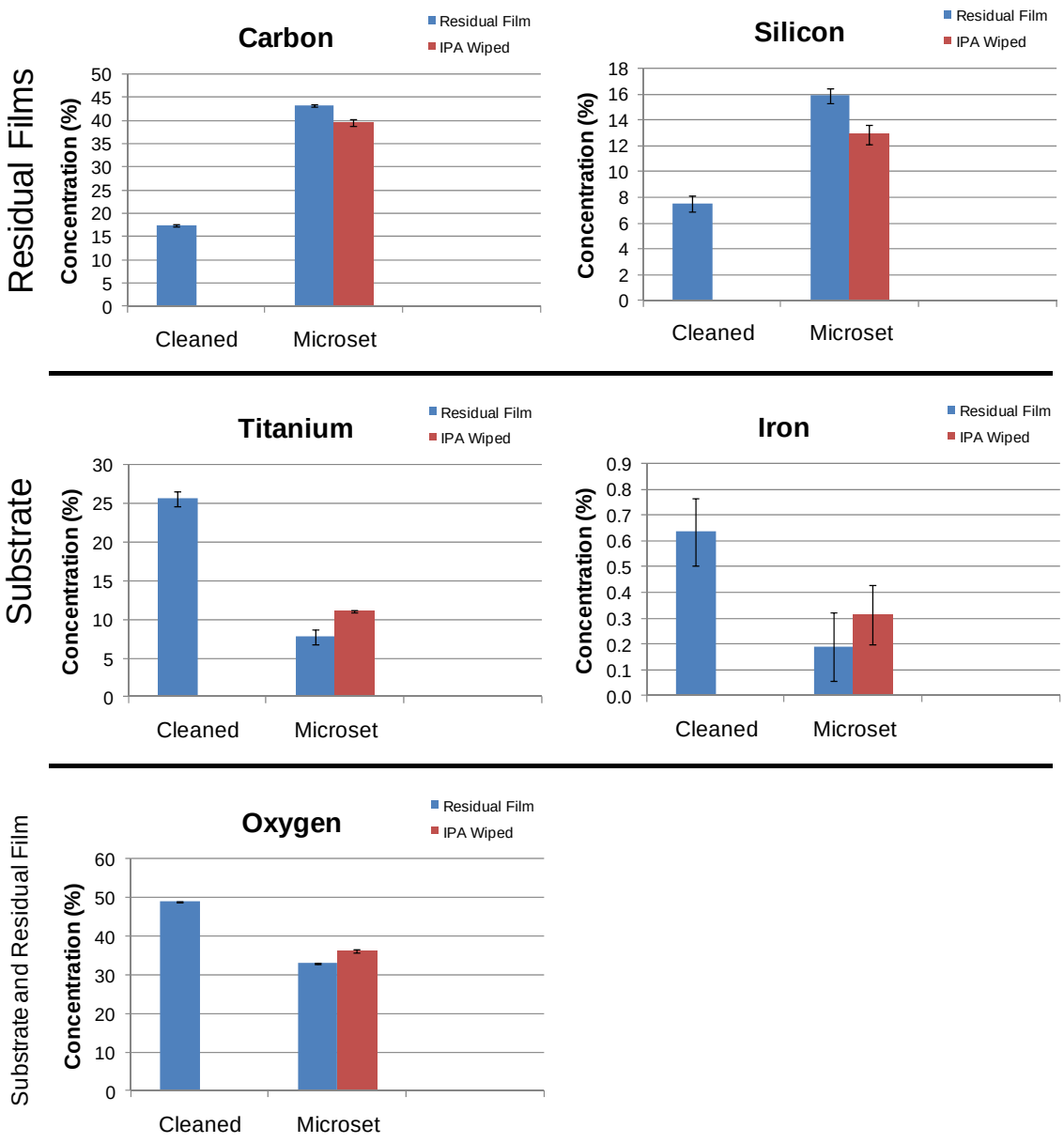


Figure 8. Sample G, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.8. *XPS Results for Sample H – Al 6061-T6*

Carbon and silicon increase due to the Microset residual film, relative to the initial cleaned surface. The silicon can be reduced by IPA wiping, but not as effectively as has been seen for previous samples A-F.

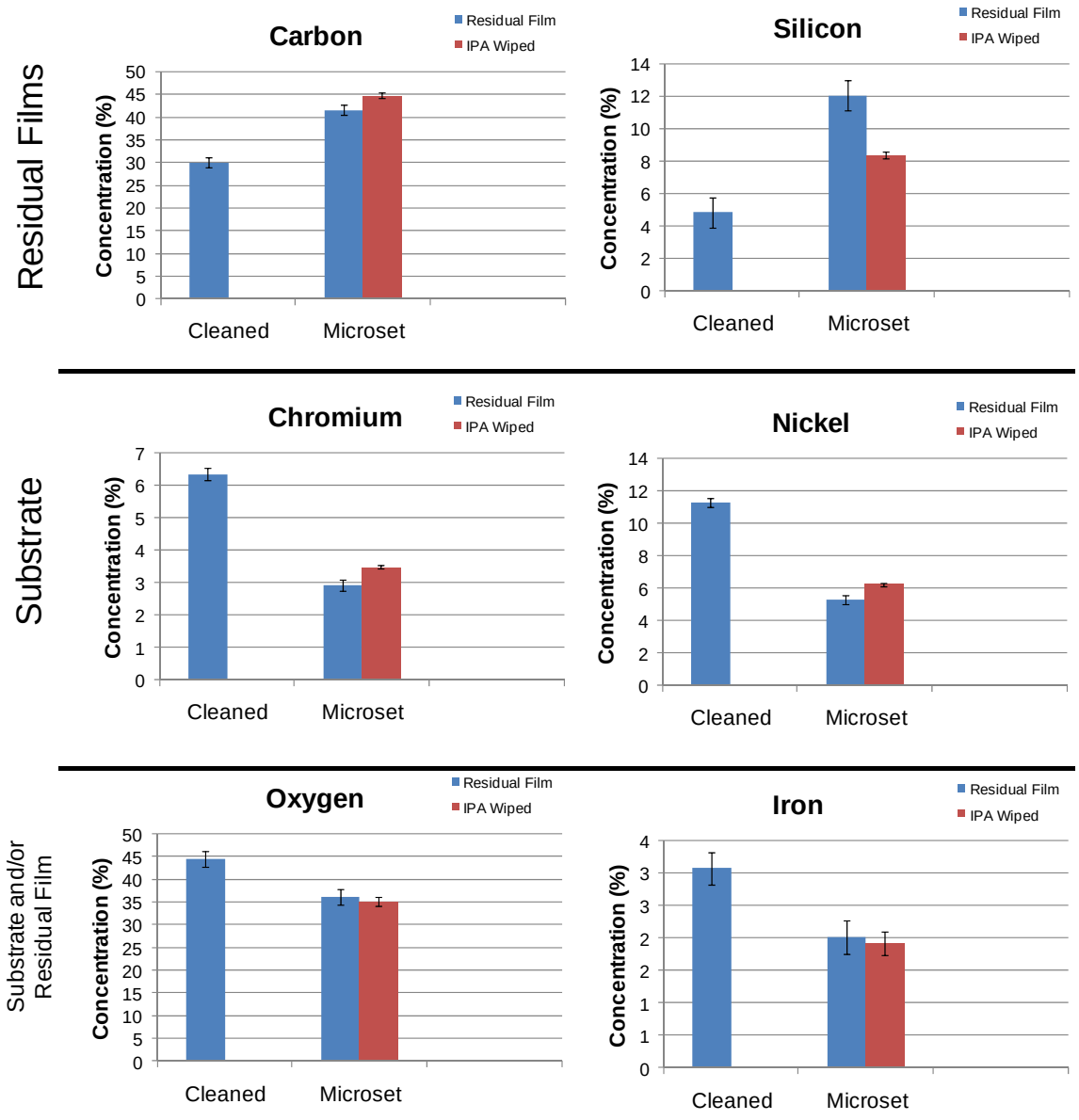


Figure 9. Sample H, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.9. XPS Results for Sample I – Al 7075-T6

Carbon increases due to residual Microset and Coe-Flex films. The carbon increase is substantial for the Coe-Flex film. The silicon from the substrate is masked by the thick carbonaceous film from Coe-Flex. IPA wiping is effective at removing the Coe-Flex film, however, Cu and Cl remain. Coe-Flex also leaves residual N and Na, but these elements have not been included since they are at low concentrations and do not appear to be affected by the IPA wiping.

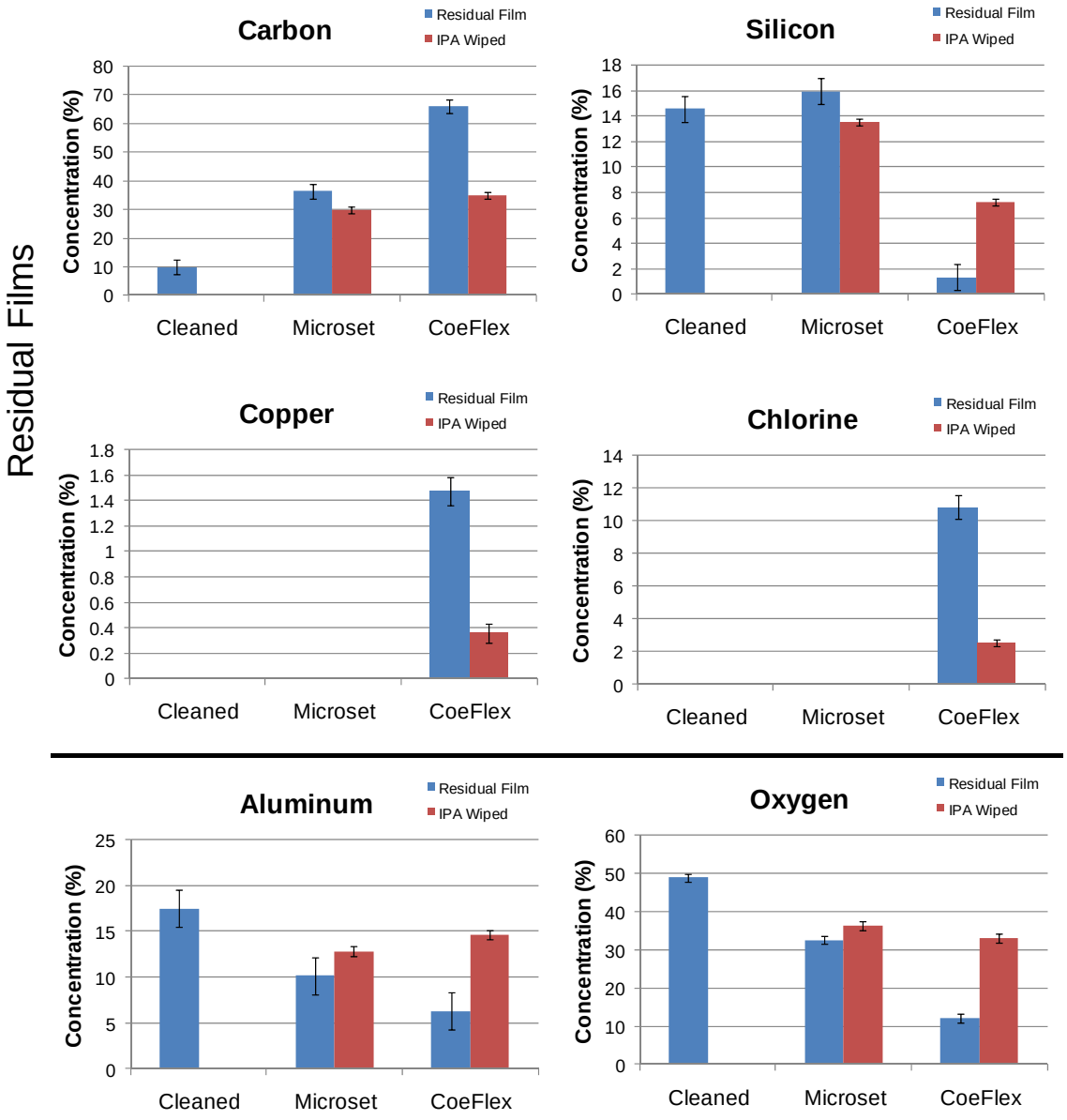


Figure 10. Sample I, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, Coe-Flex residual film, and the residual films after wiping the surface with isopropanol.

3.2.10. XPS Results for Sample J – Al 6061-T6 anodized

Very similar results are observed for Sample J and Sample I. These two samples show that IPA wiping is about equally effective at removing Microset and Coe-Flex films. However, IPA wiping does not remove all of the Cu and Cl remaining from the Coe-Flex residual film.

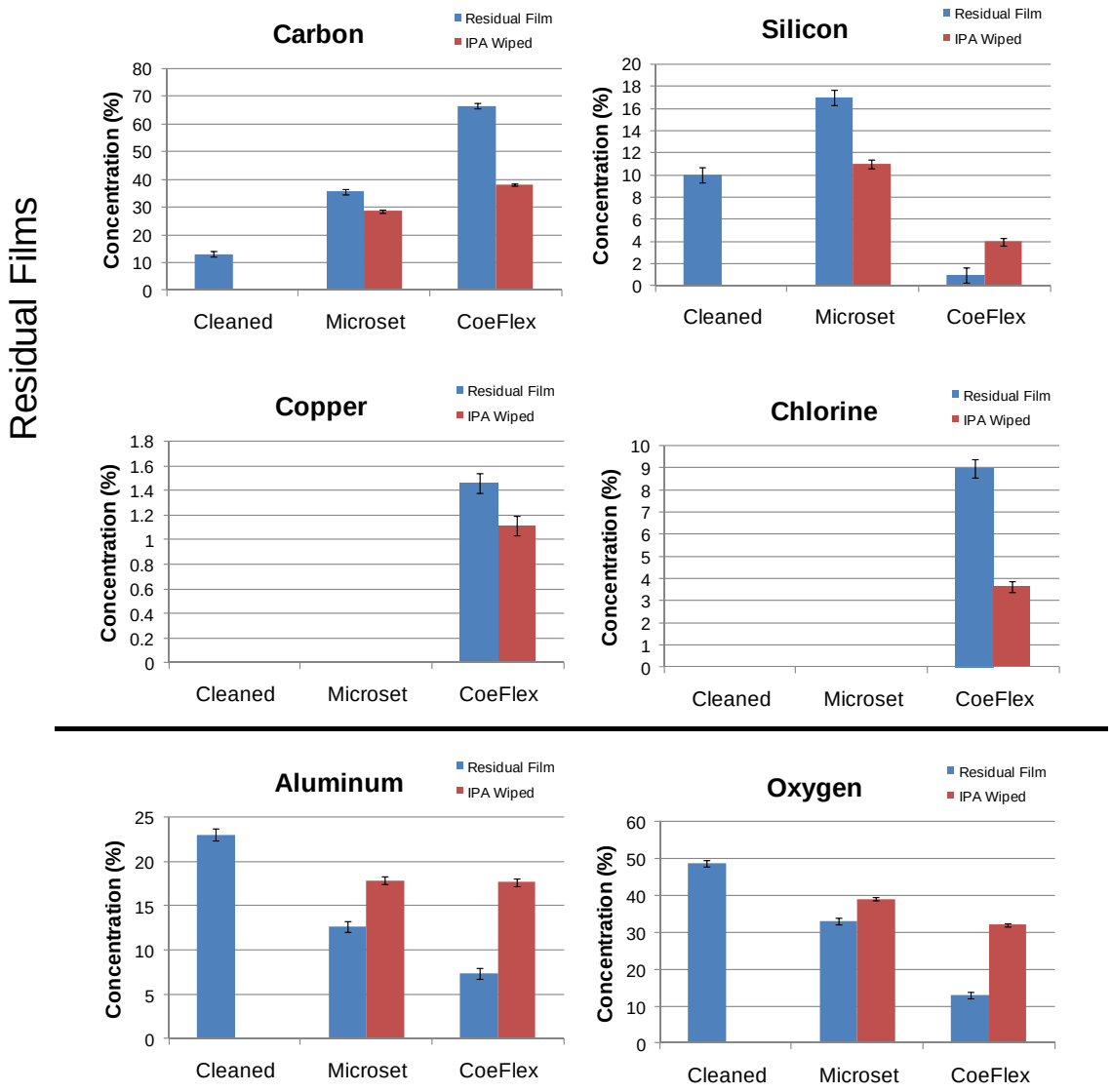


Figure 11. Sample J, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, Coe-Flex residual film, and the residual films after wiping the surface with isopropanol.

3.2.11. XPS Results for Sample K – Al 7075-T6 anodized

Carbon and silicon increase due to the Microset residual film. IPA wiping removes some of the residual film.

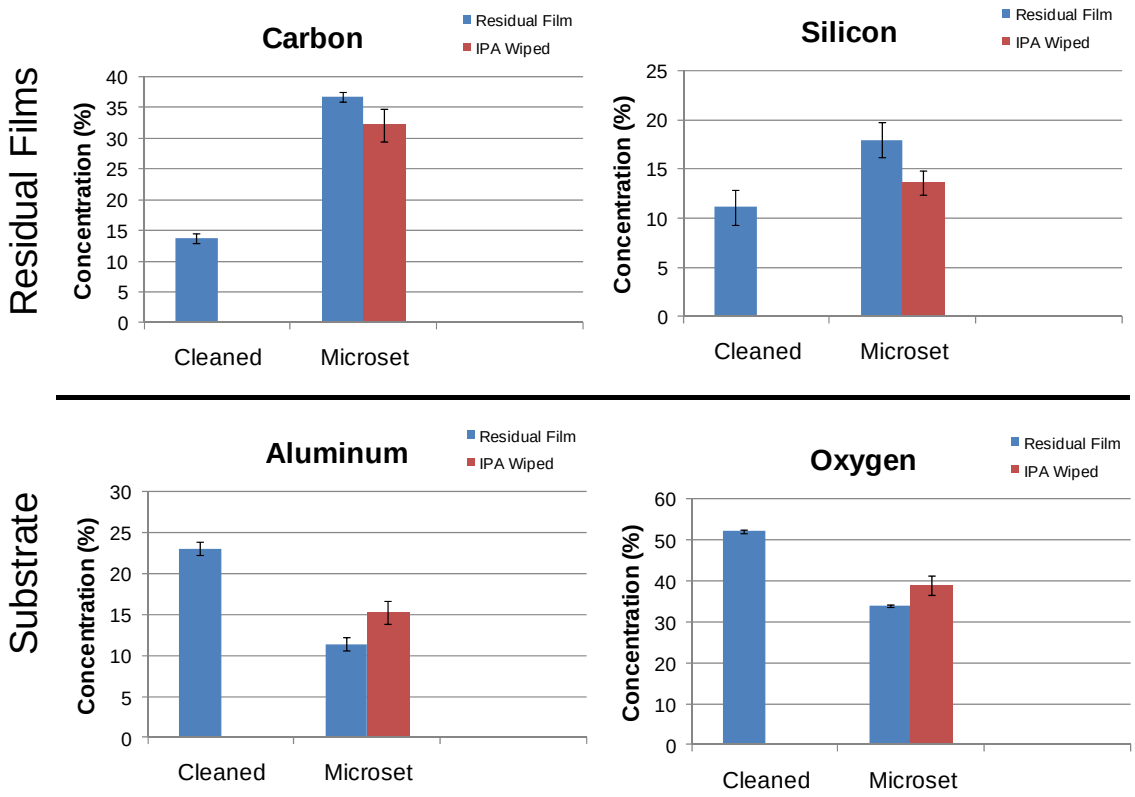


Figure 12. Sample K, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.12. *XPS Results for Sample L – Al 6061-T6 alodined 1997*

Carbon and silicon increase due to the Microset residual film. IPA wiping appears to be the most effective at removing the residual film for this sample.

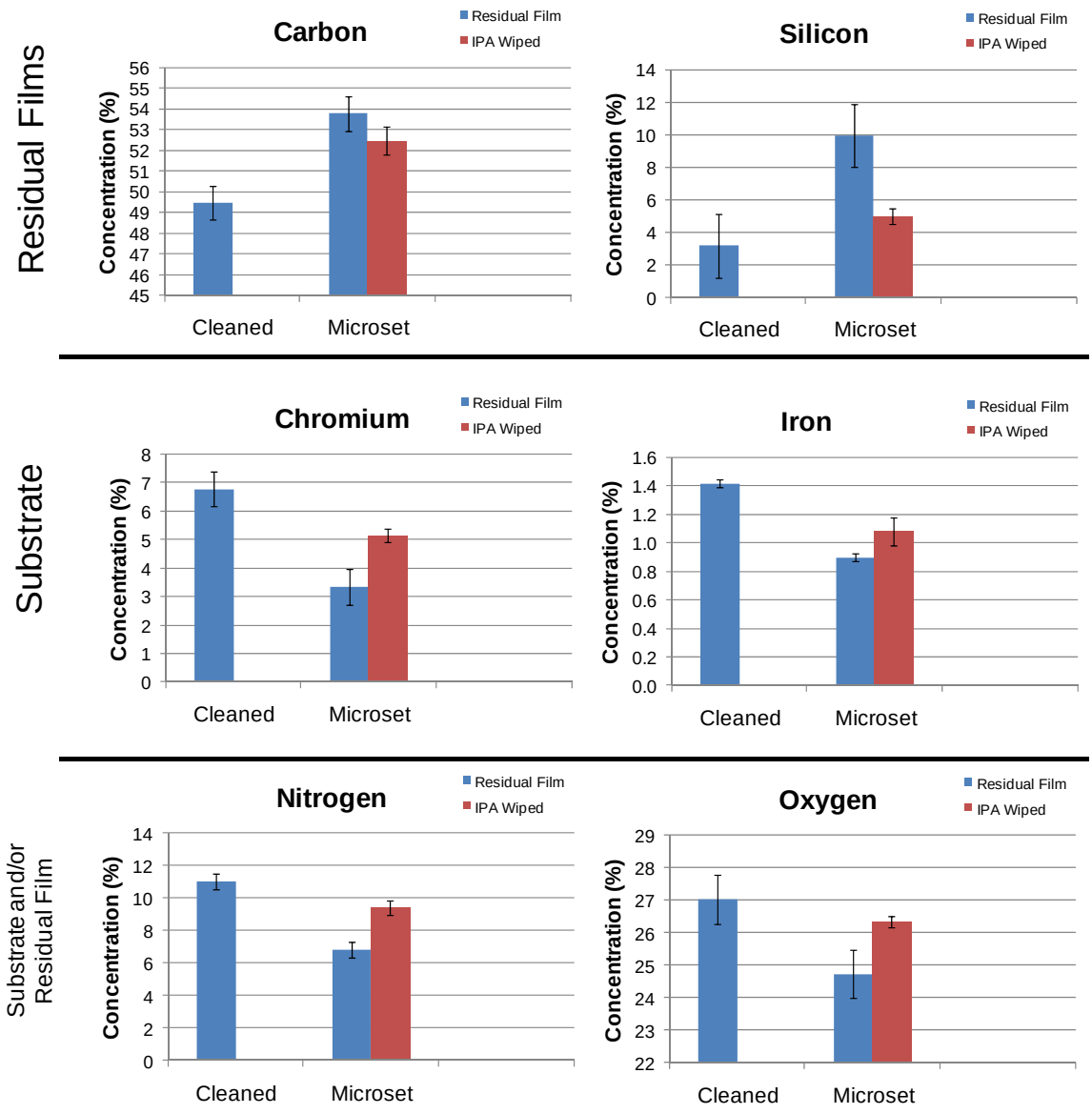


Figure 13. Sample L, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.13. *XPS Results for Sample M – Al 7075-T6 alodined 1997*

Carbon and silicon increase due to the Microset residual film, relative to the initial cleaned surface. The silicon can be effectively reduced by wiping the surface with isopropanol (IPA), however the carbon level remains high. The remaining high level of carbon does not necessarily relate to residual Microset, it may also be due to other carbonaceous contaminants introduced by the IPA wiping process (possibly including IPA itself).

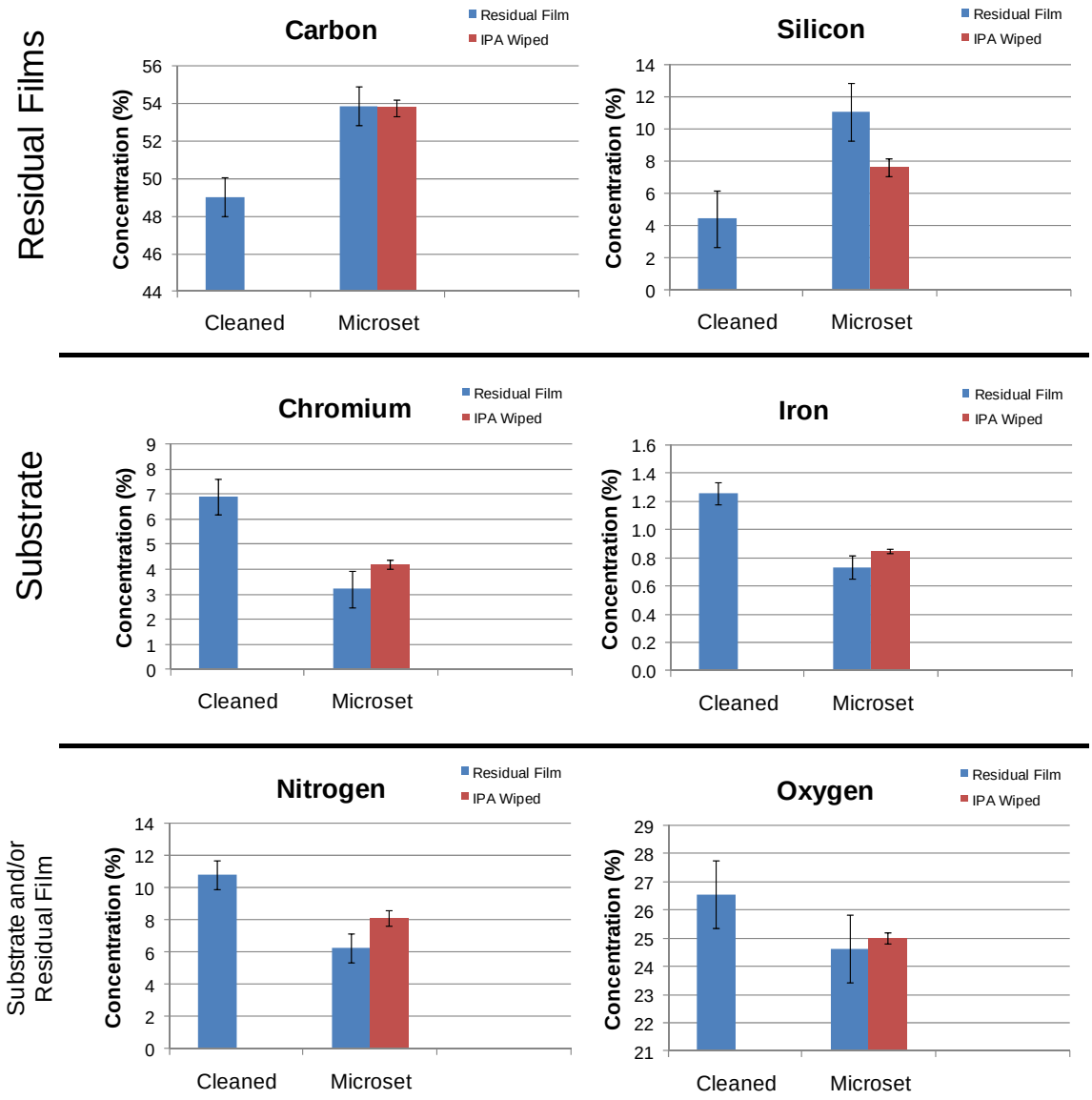


Figure 14. Sample M, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.14. *XPS Results for Sample N – Al 6061-T6 alodined 047*

Carbon and silicon increase due to the Microset residual film, relative to the initial cleaned surface. The silicon can be effectively reduced by wiping the surface with isopropanol (IPA), however the carbon level remains high. The remaining high level of carbon does not necessarily relate to residual Microset, it may also be due to other carbonaceous contaminants introduced by the IPA wiping process (possibly including IPA itself).

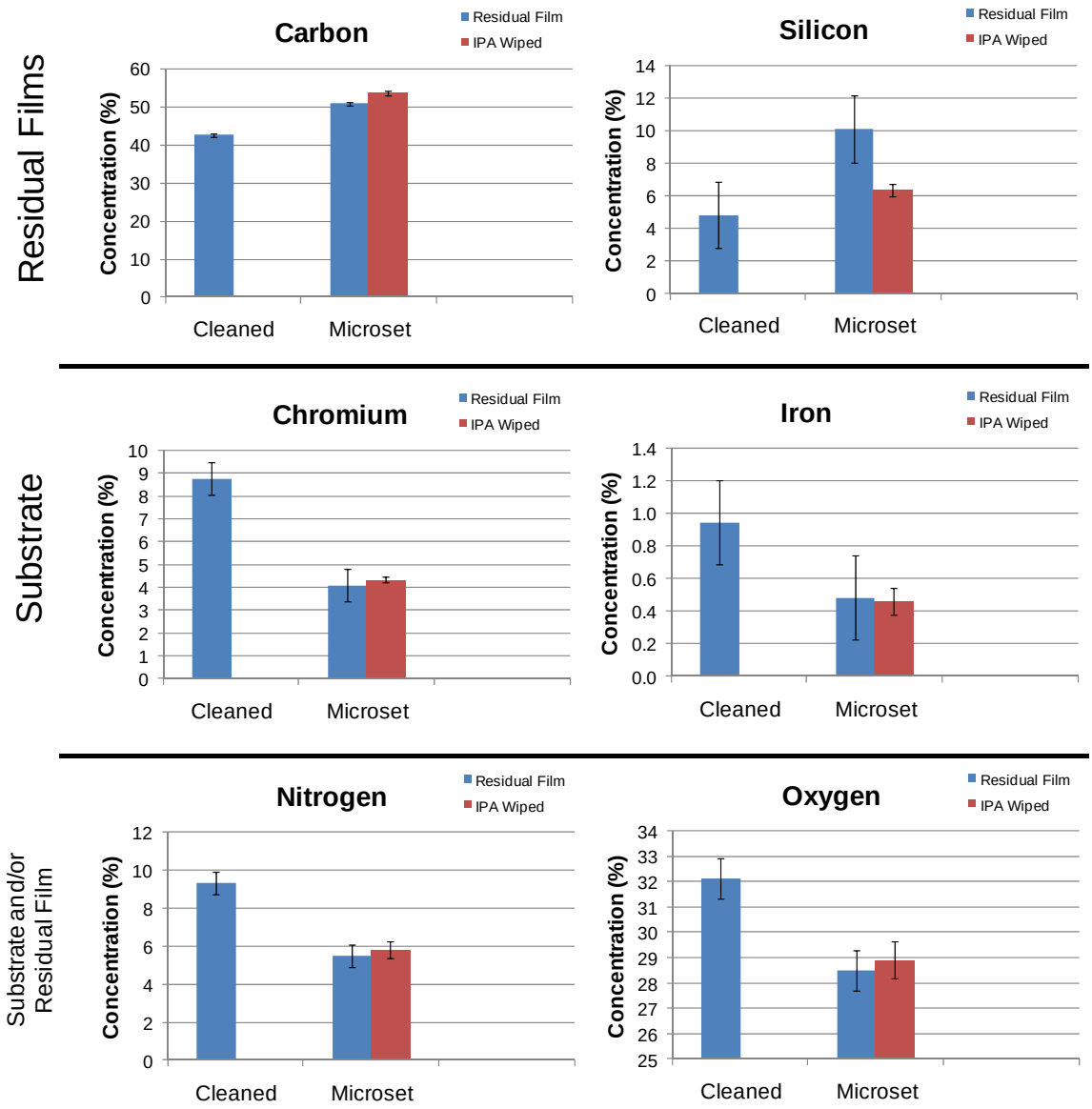


Figure 15. Sample N, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.15. *XPS Results for Sample O – Al 6061 alodined 2004*

Carbon and silicon increase due to the Microset residual film, relative to the initial cleaned surface. The silicon can be effectively reduced by wiping the surface with isopropanol (IPA), however the carbon level remains high. The remaining high level of carbon does not necessarily relate to residual Microset, it may also be due to other carbonaceous contaminants introduced by the IPA wiping process (possibly including IPA itself).

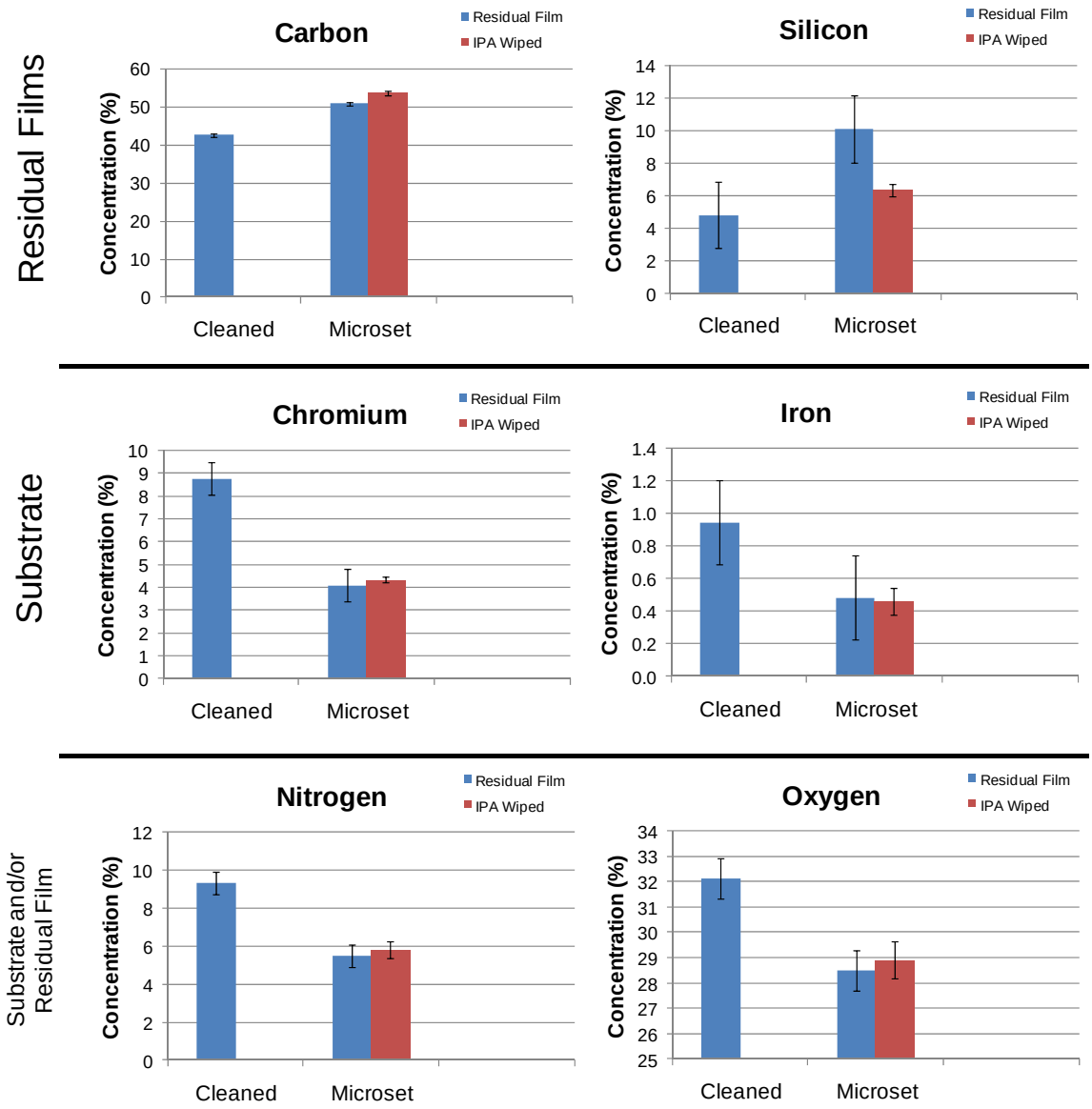


Figure 16. Sample O, relative atomic concentrations of important elemental constituents are shown for the as-cleaned surface, the Microset residual film, and the residual film after wiping the surface with isopropanol.

3.2.16. Thickness Estimations from XPS

Approximate thicknesses of the residual films can be estimated from the XPS data. The equation used to estimate thickness is:

$$\frac{I_{obs}}{I_{ideal}} = \exp\left(\frac{-d}{\lambda sin\theta}\right)$$

I_{obs} = the intensity from the substrate elemental component with the residual film present
I_{ideal} = the intensity of the substrate under ideal/clean conditions
d = thickness of the residual film (nm)
λ = effective attenuation length of the escaping photoelectron (nm)
θ = take-off angle

I_{obs} and I_{ideal} are measured values obtained from the XPS analysis. The take-off angle is 90°. The effective attenuation length is the approximate escape depth for electrons through the overlayer at a specific kinetic energy (KE). Approximate values for λ can be obtained from the literature.

In this analysis there are sources of error which cause the calculation to give only an approximation. Sources of error can arise from measurement of I_{ideal} and λ. Surface roughness can also induce significant error in the measurement and calculation. Here, I_{ideal} was taken as the intensity from the as-cleaned metal surface. This induces some error in the calculation since the as-cleaned metal surface is not perfectly clean. The effective attenuation length was assumed to be 3 nm for all calculations. A value of 3 nm is a relatively good approximation for making calculations which can be relatively compared, but the true value of λ would be different for the Microset and Coe-Flex residual films, as well as for the various elemental signals (Cr, Fe, Al). The estimated thicknesses shown in Table 4 give some degree of the changes in thicknesses that could be anticipated from IPA wiping, and give some relative indication of the difference in thickness between Microset and Coe-Flex residual films.

The Microset residual film is quite thin and substrate signal can be clearly observed via XPS through the thin contaminant film. Given the above disclaimers regarding thickness calculations from XPS, an approximate thickness for the Microset residual film is around 2 nm. It is apparent from the data shown in Table 4, that the isopropanol wiping procedure is effective at removing approximately half of the residual Microset film thickness.

The residual Coe-Flex films appears to be slightly thicker than the Microset films, however, there may be a change in λ which makes this comparison more ambiguous. The isopropanol wiping procedure is also effective at reducing the total thickness of the Coe-Flex residual film.

Table 4. Estimated thicknesses of residual films from XPS measurements.

	Microset residual film thickness (nm)	Microset film after wiping with IPA (nm)	Coe-Flex residual film thickness (nm)	Coe-Flex film after wiping with IPA (nm)	Element used for calculation
A	2.5	0.6	2.8	1.7	Cr
B	2.4	1.8			Cr
C	2.3	1.8			Cr
D	1.9	0.6			Cr
E	2.7	0.8			Cr
F	1.1	0.5			Al
G	3.6	2.1			Fe
H	2.3	1.8			Cr
I	1.6	0.9	3.1	0.5	Al
J	1.8	0.8	3.4	0.8	Al
K	2.1	1.2			Al
L	2.1	0.8			Cr
M	2.3	1.5			Cr
N	2.3	2.1			Cr
O	1.2	1.2			Cr
Avg.	2.1 ± 0.6	1.2 ± 0.6	3.1 ± 0.3	1.0 ± 0.6	

3.3. Ellipsometry

Ellipsometry was performed on residual Microset and Coe-Flex films to obtain a second determination of approximate film thicknesses, Table 5. Different samples were prepared than those used for XPS and only samples B, C, E, and I were evaluated. The refractive index of the residual films was assumed to be 1.45, a reasonable value for most organic films. The presence of inorganic constituents in the residual films may cause the refractive index to be different from this assumed value, however, for these thin films it was not possible to get a direct measurement of refractive index. The refractive index will change the calculated thickness linearly (e.g. if the refractive index is off by 5%, so is the calculated thickness). Delta and psi values were taken at six locations and averaged (lambda = 532 nm, AOI = 70 degrees). The presence of regions with varying thicknesses will not be observed given the ~2 micron resolution of the instrument, thus the values given are average thicknesses.

A cloudy, non-uniform film was visually observable in regions where Coe-Flex was removed from the metal surface on samples B, C, and I. This cloudy film was not observed on sample E. This may be due to the amount of catalyst used in the Coe-Flex and/or the time allowed for curing. The films that appeared visually cloudy gave significantly greater thickness values than the Coe-Flex film which could not be visually observed.

The cloudy Coe-Flex residual films were estimated to be significantly thicker than the residual Microset films. Ellipsometry measured thicknesses of the residual Coe-Flex approximately ten times that of the Microset film thicknesses, for samples B and I. These thickness values are much greater than were determined by XPS, however, both XPS and ellipsomtery thickness measurements are susceptible to error. Since Coe-Flex contains Cu and Cl, and has a greater density than Microset, the ellipsometry thickness calculations for Coe-Flex may be overestimated. Nevertheless, the Coe-Flex films appear to be generally thicker than the Microset films, and the Microset films appears to be around 2 nm thick.

Table 5. Estimated thicknesses of residual films calculated from ellipsometry.

	Delta	Psi	n	k	Thickness (nm)
E	121.80 ± 0.10	32.93 ± 0.03			
E/Coe-Flex	118.43 ± 0.53	33.01 ± 0.05	1.45	0	2.3 ± 1
E/Microset	118.1 ± 0.14	33.22 ± 0.03	1.45	0	2.6 ± 1
B	120.67 ± 0.15	31.36 ± 0.11			
B/Coe-Flex	98.21 ± 5.24	40.32 ± 11.25	1.45	0	22 ± 1
B/Microset	117.91 ± 0.16	31.83 ± 0.10	1.45	0	2 ± 1
C	118.54 ± 0.50	32.74 ± 0.22			
C/Coe-Flex	106.98 ± 2.94	32.95 ± 0.34	1.45	0	8 ± 1
C/Microset	116.01 ± 0.53	32.90 ± 0.10	1.45	0	1.8 ± 1
I	88.43 ± 3.77	36.34 ± 0.69			
I/Coe-Flex	65.56 ± 2.44	37.57 ± 4.24	1.45	0	28 ± 2
I/Microset	87.22 ± 1.79	37.57 ± 0.48	1.45	0	2.4 ± 1

3. CONCLUSIONS

If a 1 m^2 area were covered with Microset, and the Microset were removed leaving a 2 nm thick contaminant film, the resulting volume of material remaining would be 2 µL. For most applications the area covered would be expected to be significantly less, and so the volume of material remaining would also be significantly less. This simple calculation demonstrates the very minimal amount of material that remains from these impression materials, particularly Microset. Figure 17 shows the approximate masses of residual material that could be expected for a 2 nm thick Microset residual film.

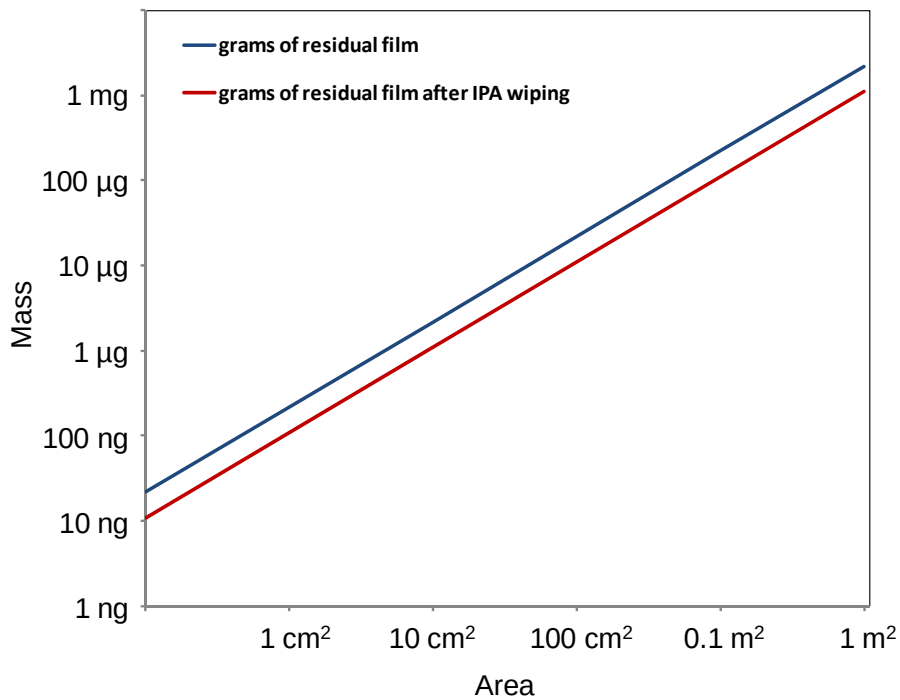


Figure 17. The mass of residual material is plotted as a function of area. The residual material is assumed to have a density of 1.1 g/cm^3 and a thickness of 2 nm. The red line shows the expected mass of material if half of the film thickness was removed by wiping with isopropanol (IPA).

From an ease-of-use perspective, Microset was vastly superior to Coe-Flex. Coe-Flex requires the mixing of a base material and a catalyst. Presumably the percent of cure and cure time would be dependent on the amount of catalyst used, as well as the effectiveness of mixing. The Coe-Flex also has to be applied to the substrate using a transferring instrument, such as a spatula. Conversely, Microset can be applied in a one-shot application using the application gun and the attached static mixer.

The residual film from Microset appears to be a silicone based material comprised of C, Si, and O. ToF-SIMS would be required to prove the silicone-based nature of the contaminant film. A simple isopropanol wipe can effectively remove half of the residual contaminant film thickness.

Other solvents, such as hexane, would be much more effective at removing the silicone based contaminant.

Isopropanol is effective at removing Coe-Flex residual films, which are thicker than Microset residual films, however, copper and chlorine cannot be completely removed. Coe-Flex is more dense than Microset, so the overall mass of remaining contaminant could be expected to be greater for the Coe-Flex impression material.

APPENDIX A: METAL BULK COMPOSITION

Bulk compositions for the various metal alloys are identified in Table A.1 using values found in *Woldman's Engineering Alloys*, 8th edition, edited by John P. Frick, 1994, ASM International, Materials Park, Ohio. The alodine process applied to some of the samples used in this study results in a chromate film on the surface. The chromate film is thick, from an XPS perspective, and the underlying substrate is completely masked from XPS analysis.

Table A. Expected bulk compositions of the metal substrates.

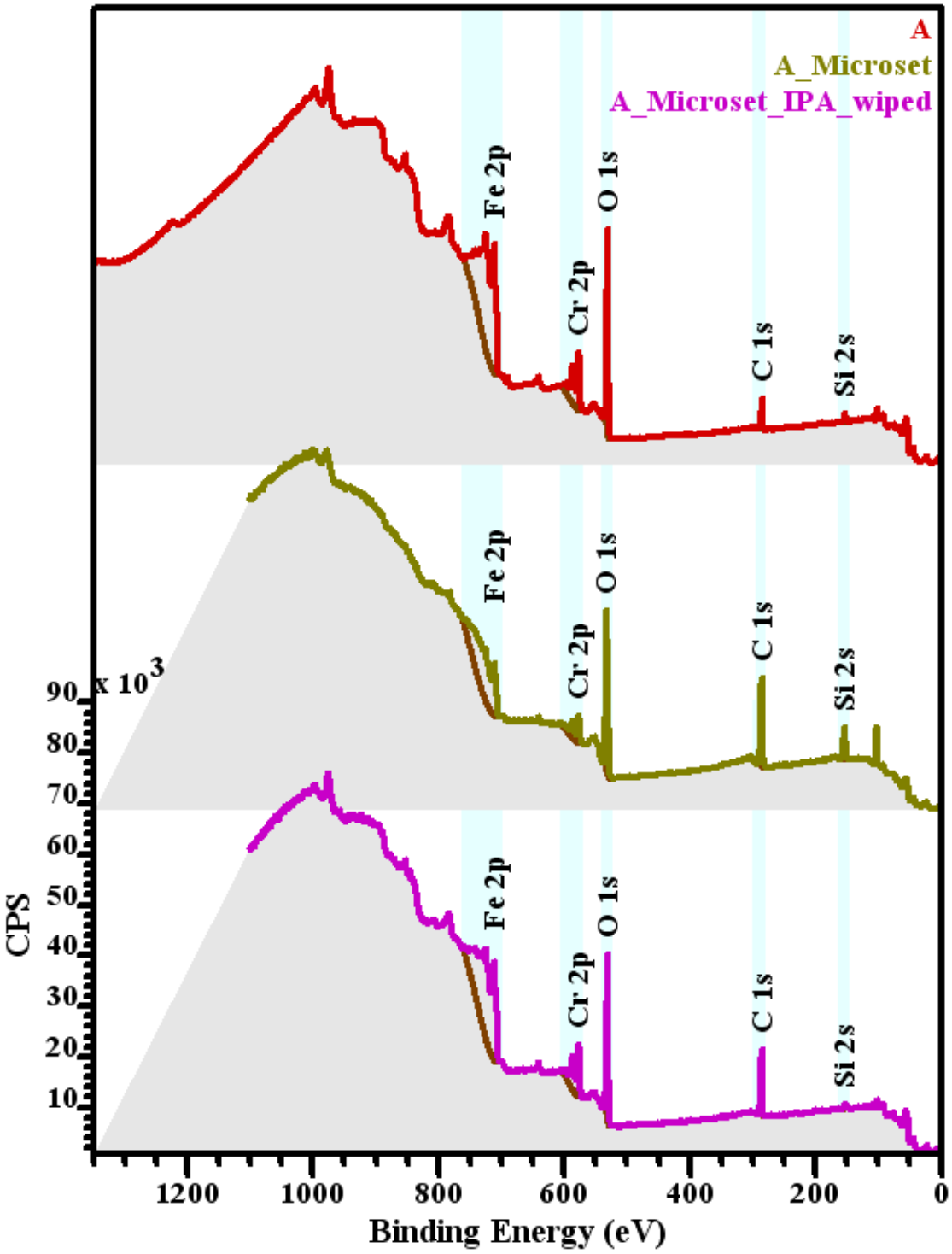
	Si	Fe	Cu	Mn	Mg	Cr	Ni
304	1	rem.		2		18-20	8-10.5
304L	1	rem.		2		18-20	8.0-12.0
316	1	rem.		2		16-18	10.0-14
316L	1	rem.		2		16-18	10.0-14
21-6-9		rem.		8.0-10		19-21.5	5.5-7.5
Ti / 6Al / 4V		0.25					
Al 6061	0.4-0.8	0.7	0.15-0.4	0.15	0.8-1.2	0.04-0.35	
Al 7075	0.4	0.5	1.2-2	0.3	2.1-2.9	0.18-0.28	

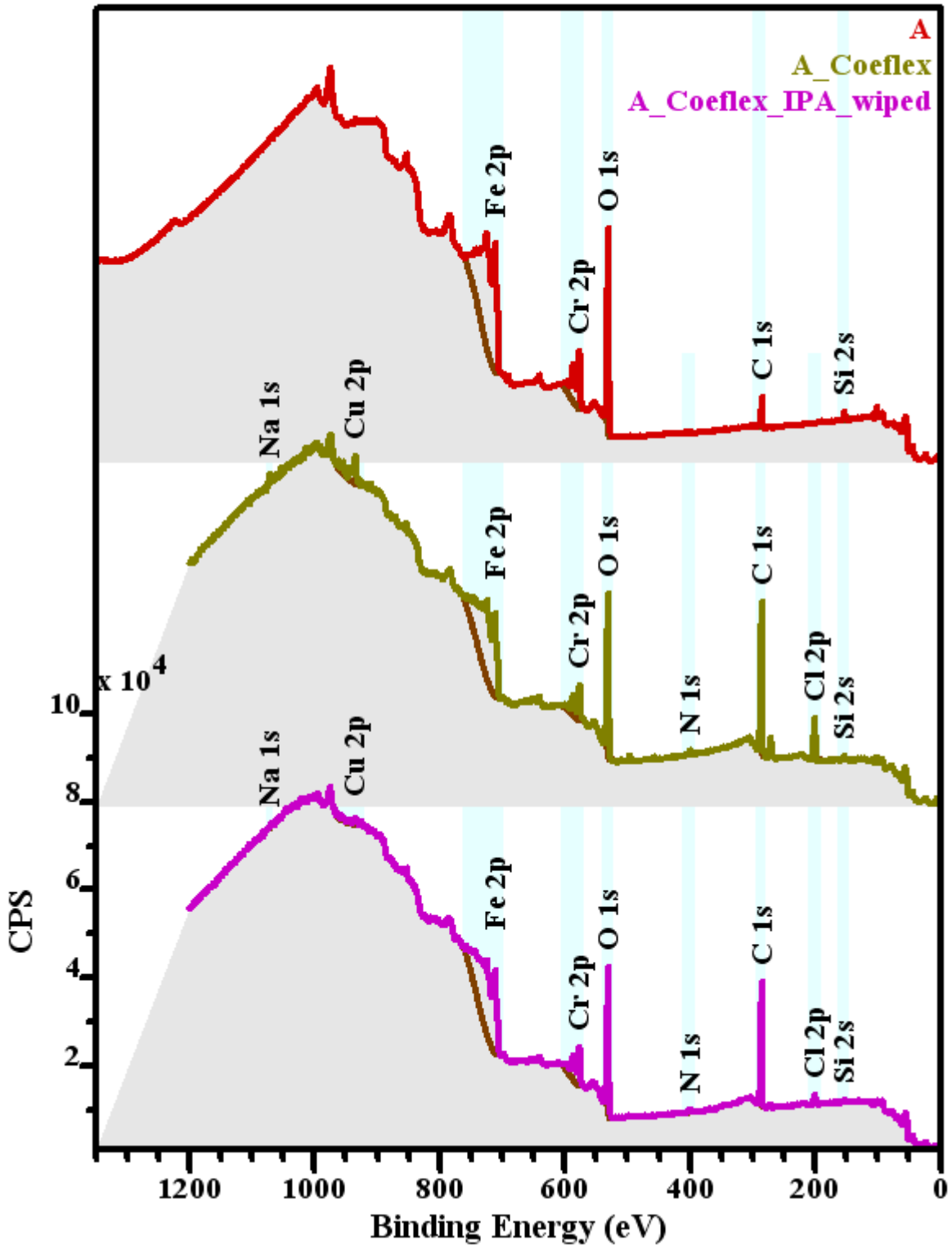
cont.	Zn	V	Ti	Mo	Al	other
304						x
304L						x
316				2.0-3		x
316L				2.0-3		x
21-6-9						x
Ti / 6Al / 4V		3.5-4.5	rem.		5.5-6.75	x
Al 6061	0.25		0.15		rem.	x
Al 7075	5.1-6.1		0.2		rem.	x

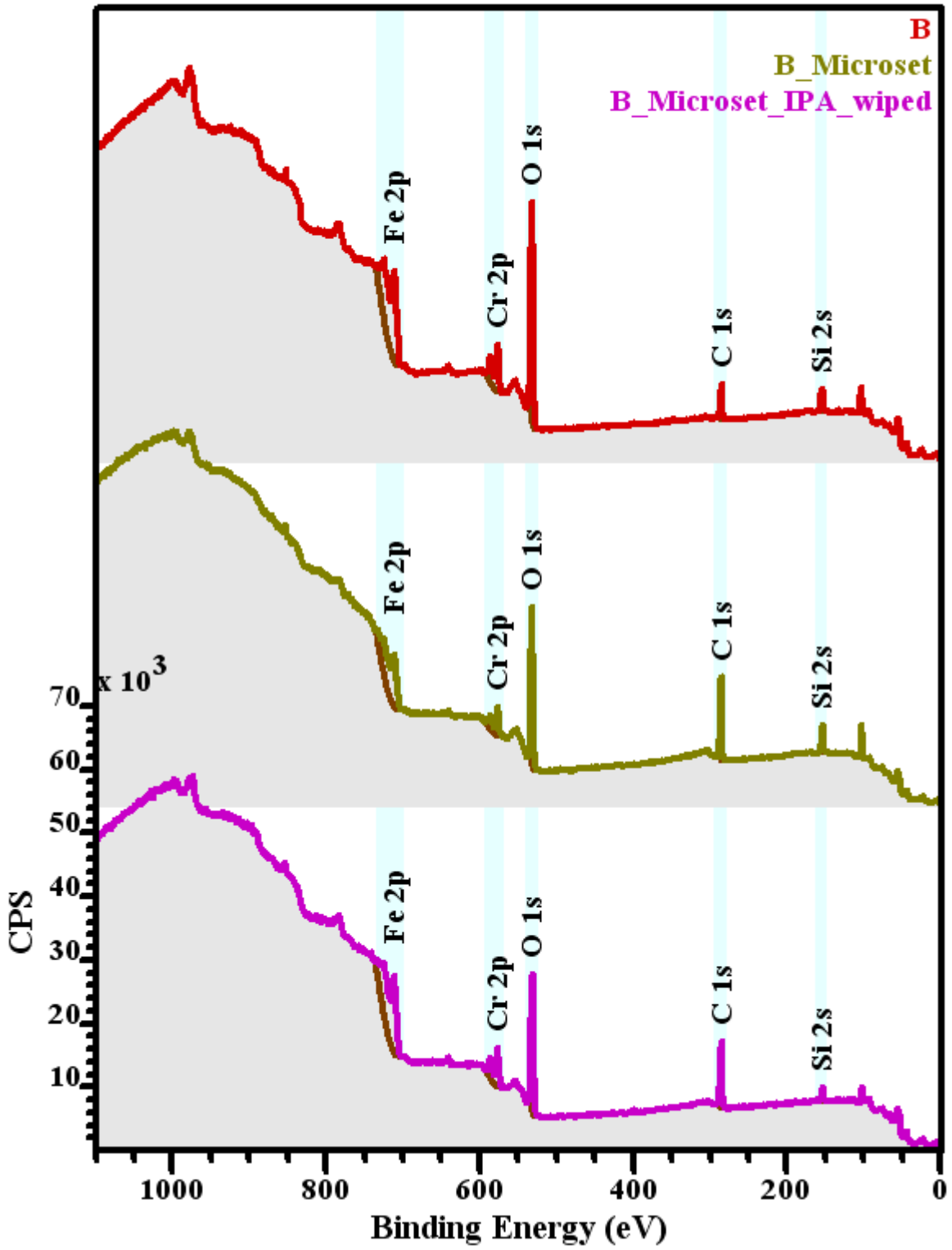
.

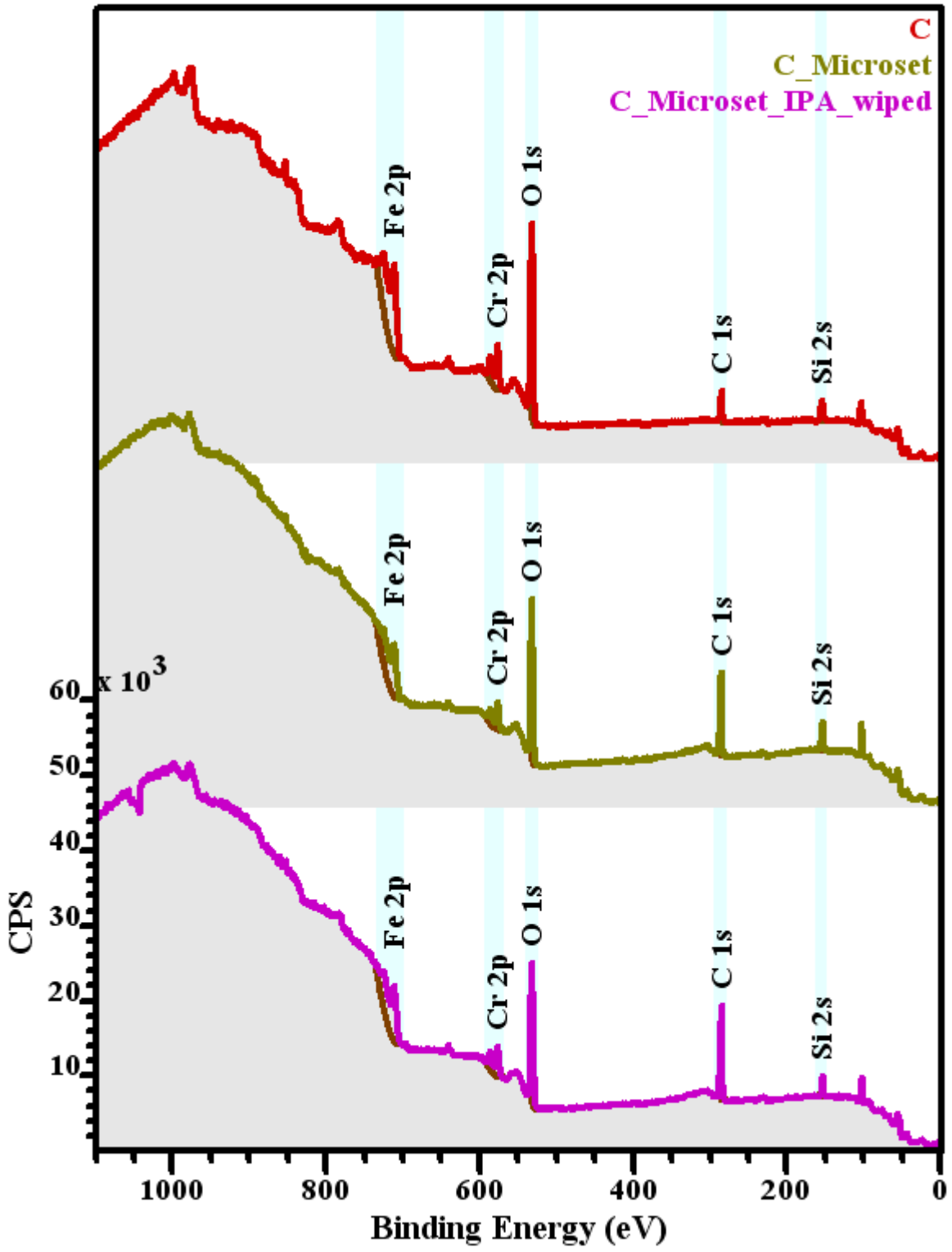
APPENDIX B: XPS SPECTRA

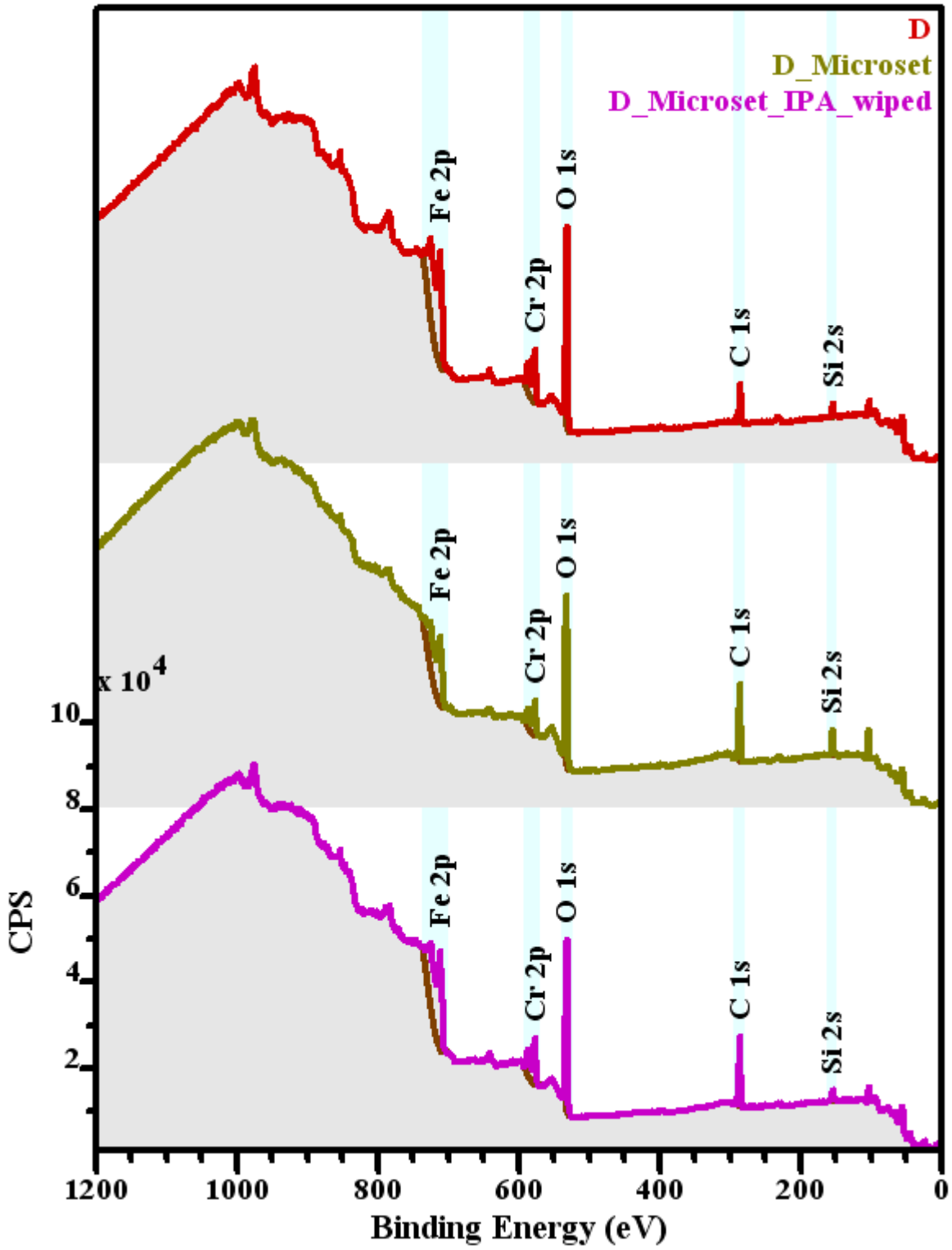
XPS spectra from the various samples are included here. Only one of the three spectra are shown for each of the different areas on each sample. Key elemental components are labeled.

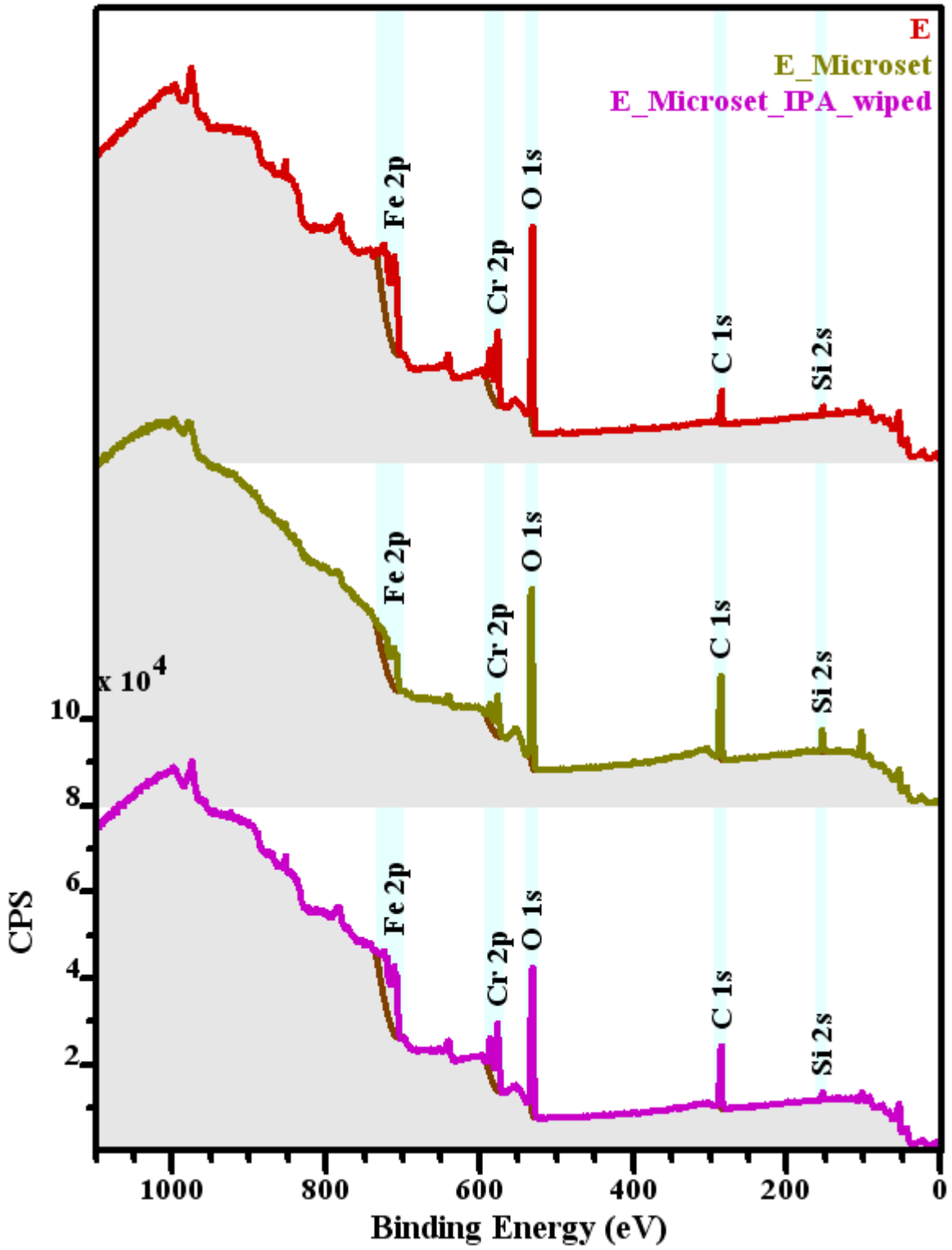


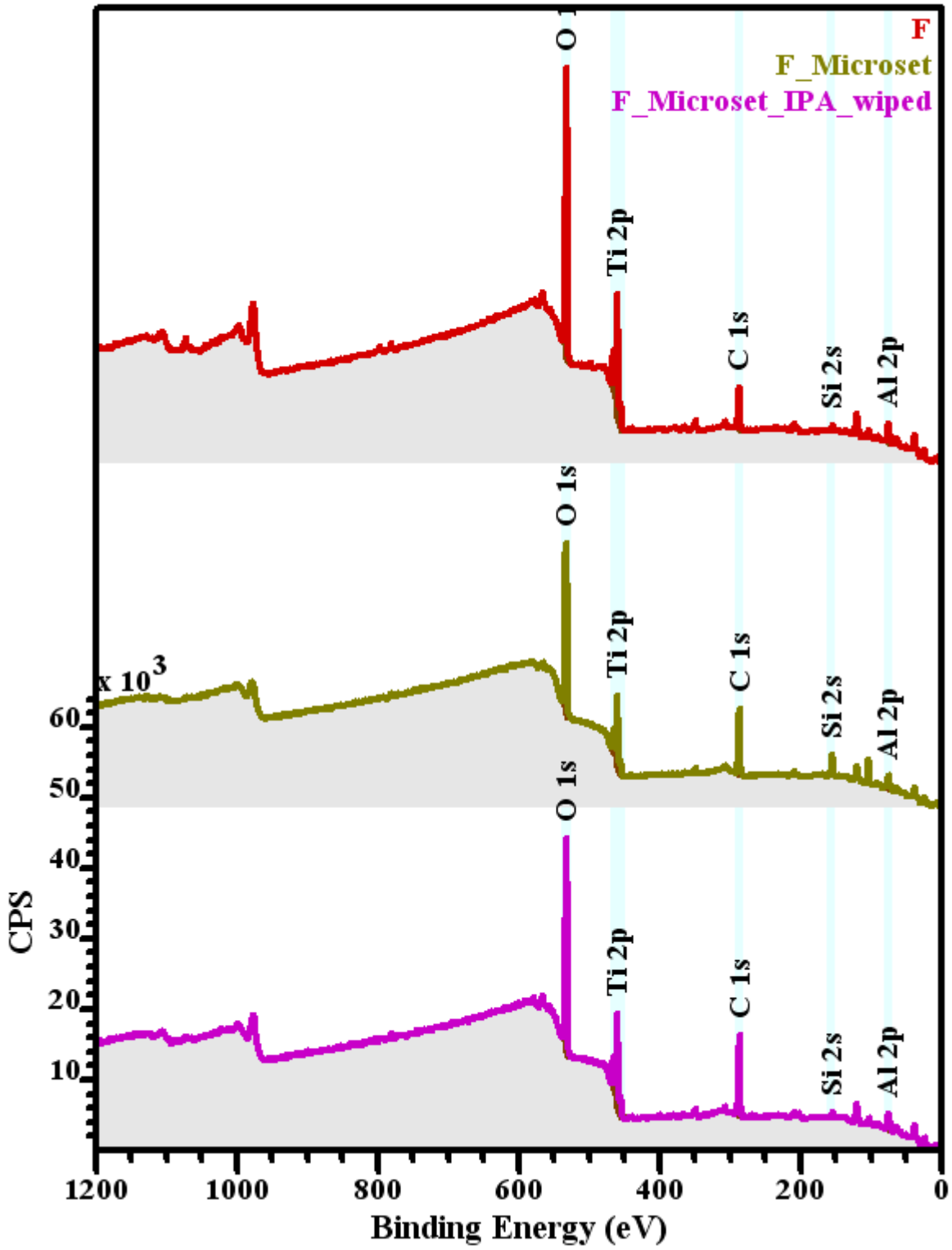

A
A_Coeflex
A_Coeflex_IPA_wiped
Fe 2p
O 1s
Cr 2p
C 1s
Si 2s
Na 1s
Cu 2p
N 1s
Cl 2p
x 10^4
10
8
6
4
2
CPS
1200
1000
800
600
400
200
0
Binding Energy (eV)

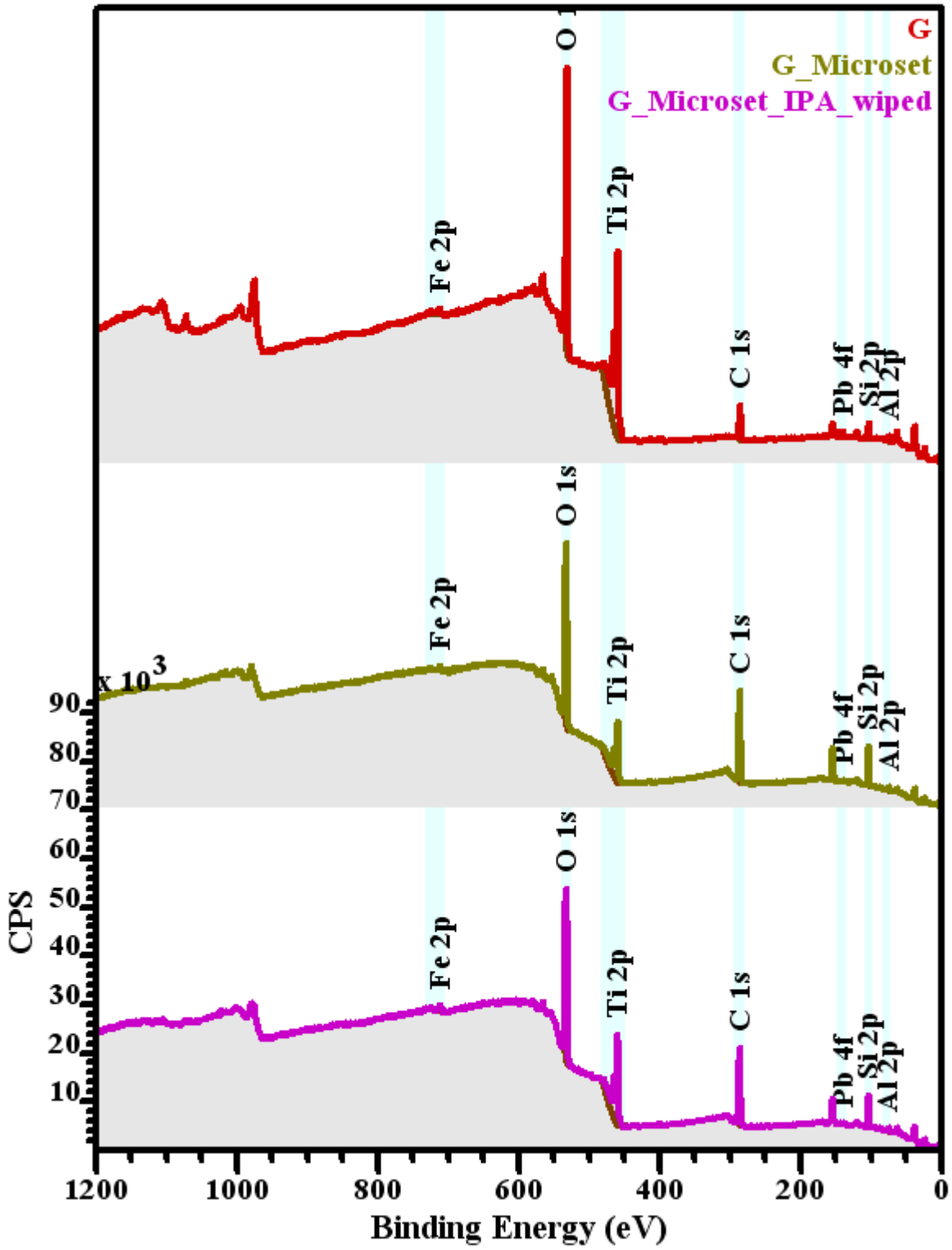

B
B_Microset
B_Microset_IPA_wiped
Fe 2p
Cr 2p
O 1s
C 1s
Si 2s
x 10^3
70
60
50
40
30
20
10
CPS
1000
800
600
400
200
0
Binding Energy (eV)

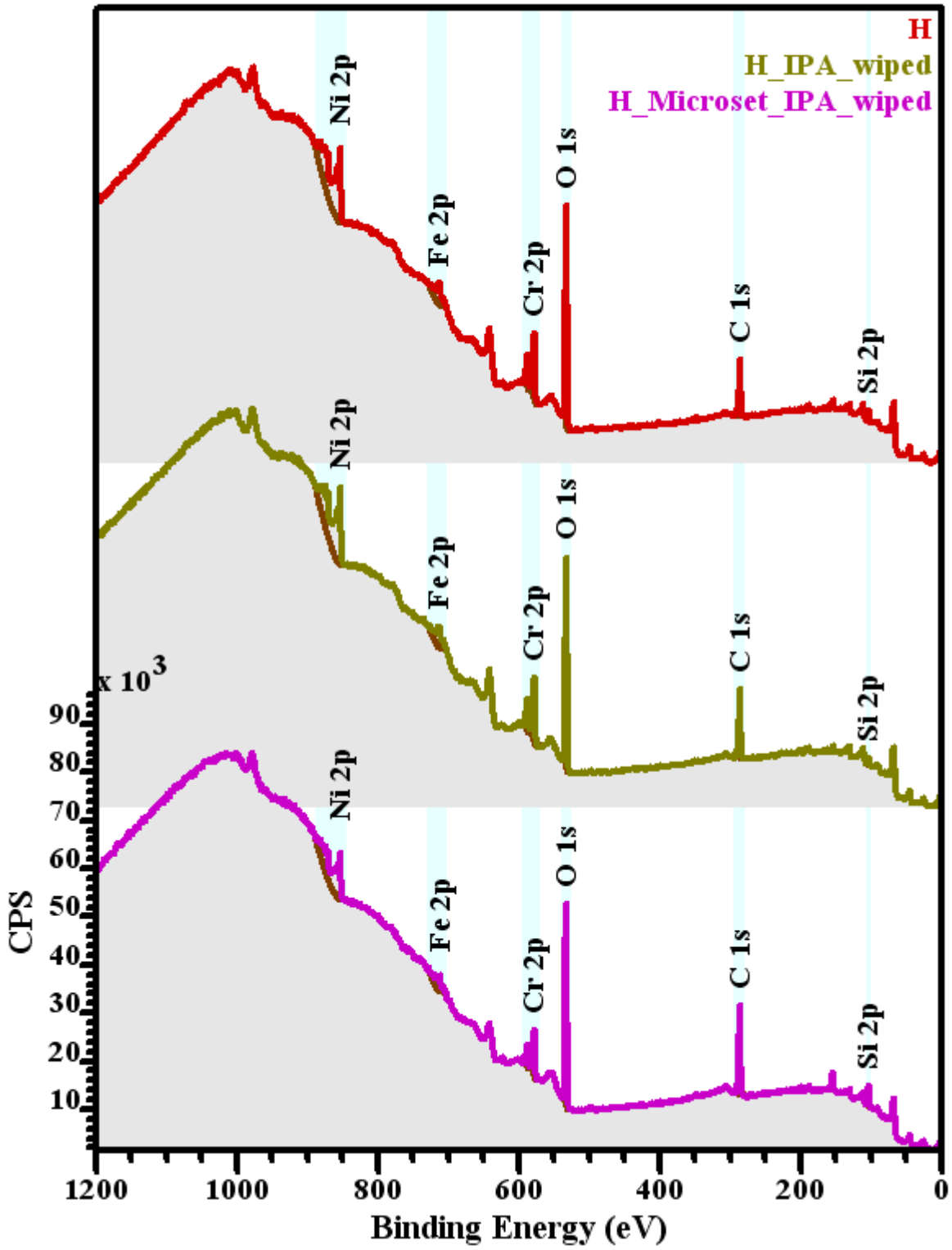

C
C_Microset
C_Microset_IPA_wiped
Fe 2p
Cr 2p
O 1s
C 1s
Si 2s
x 10^3
60
50
40
30
20
10
CPS
1000
800
600
400
200
0
Binding Energy (eV)

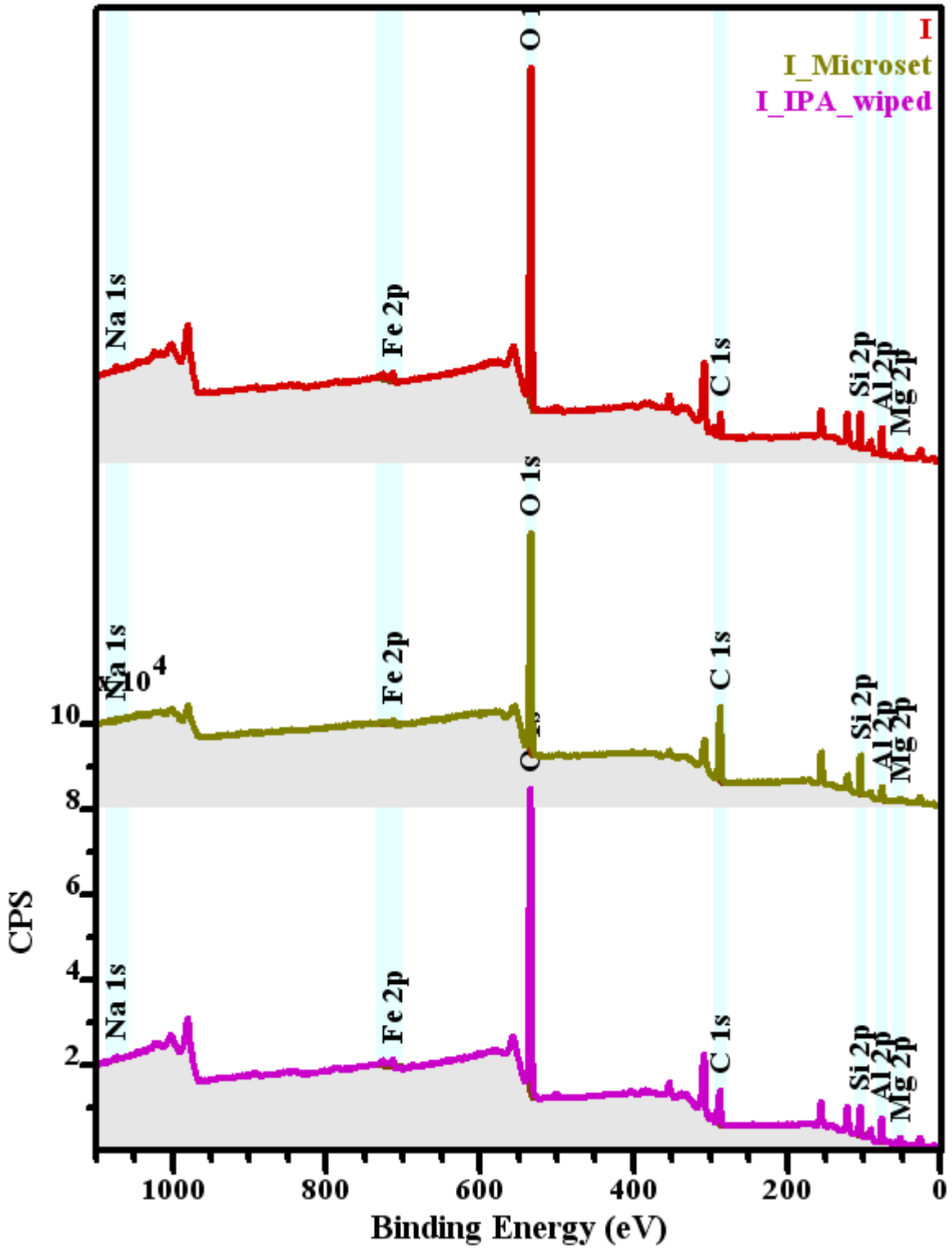

D
D_Microset
D_Microset_IPA_wiped
Fe 2p
Cr 2p
O 1s
C 1s
Si 2s
x 10^4
10
8
6
4
2
CPS
1200
1000
800
600
400
200
0
Binding Energy (eV)

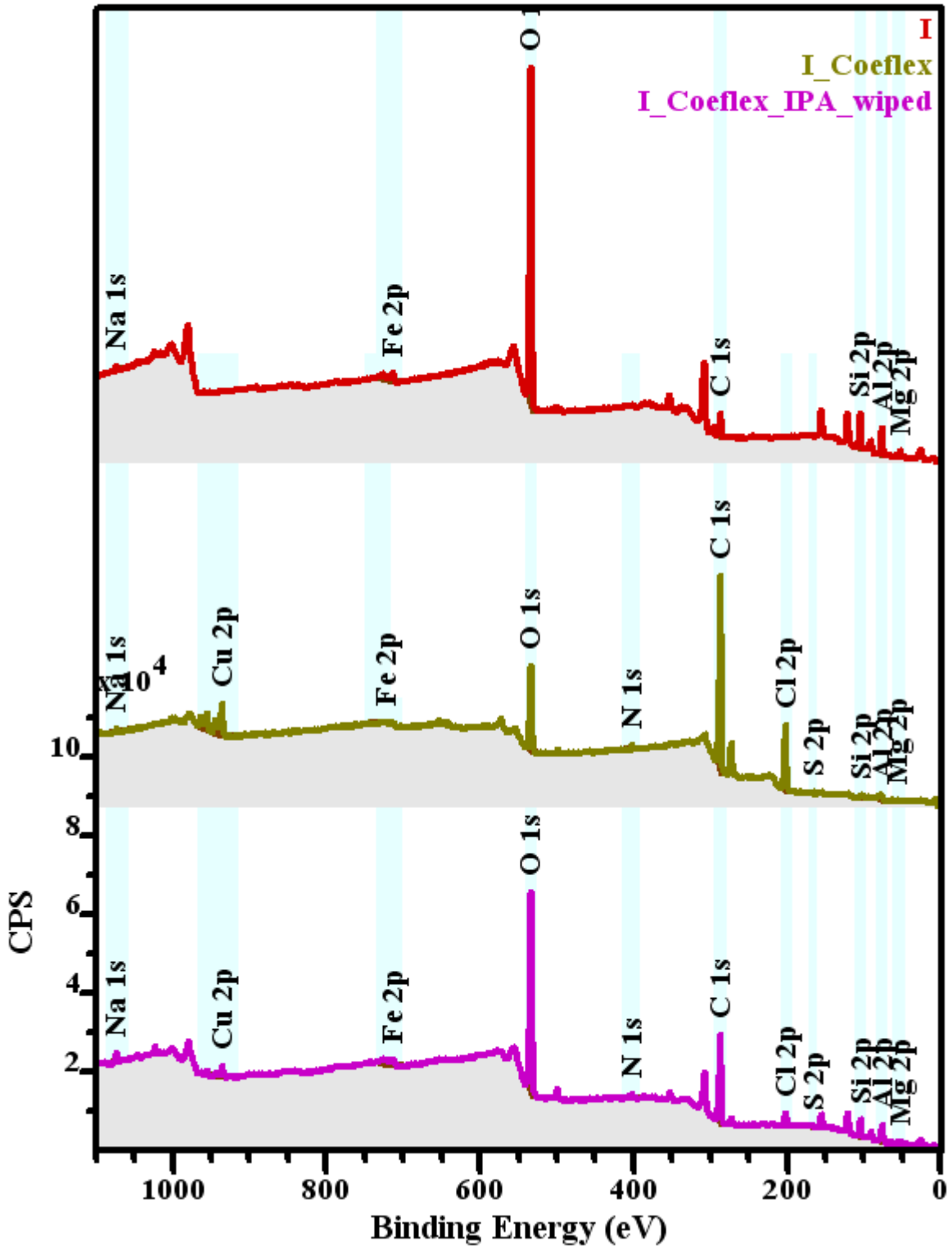

E
E_Microset
E_Microset_IPA_wiped
Fe 2p
Cr 2p
O 1s
C 1s
Si 2s
x 10^4
10
8
6
4
2
CPS
1000
800
600
400
200
0
Binding Energy (eV)


F
F_Microset
F_Microset_IPA_wiped
O 1s
Ti 2p
C 1s
Si 2s
Al 2p
x 10^3
60
50
40
30
20
10
CPS
1200
1000
800
600
400
200
0
Binding Energy (eV)


G
G_Microset
G_Microset_IPA_wiped
O 1s
Fe 2p
Ti 2p
C 1s
Pb 4f
Si 2p
Al 2p
x 10^3
90
80
70
60
50
40
30
20
10
CPS
1200
1000
800
600
400
200
0
Binding Energy (eV)


H
H_IPA_wiped
H_Microset_IPA_wiped
Ni 2p
Fe 2p
Cr 2p
O 1s
C 1s
Si 2p
x 10^3
CPS
90
80
70
60
50
40
30
20
10
1200
1000
800
600
400
200
0
Binding Energy (eV)


I
I_Microset
I_IPA_wiped
Na 1s
Fe 2p
O 1s
C 1s
Si 2p
Al 2p
Mg 2p
CPS
$\times 10^4$
10
8
6
4
2
1000
800
600
400
200
0
Binding Energy (eV)


I
I_Coeflex
I_Coeflex_IPA_wiped
Na 1s
Fe 2p
O 1
C 1s
Si 2p
Al 2p
Mg 2p
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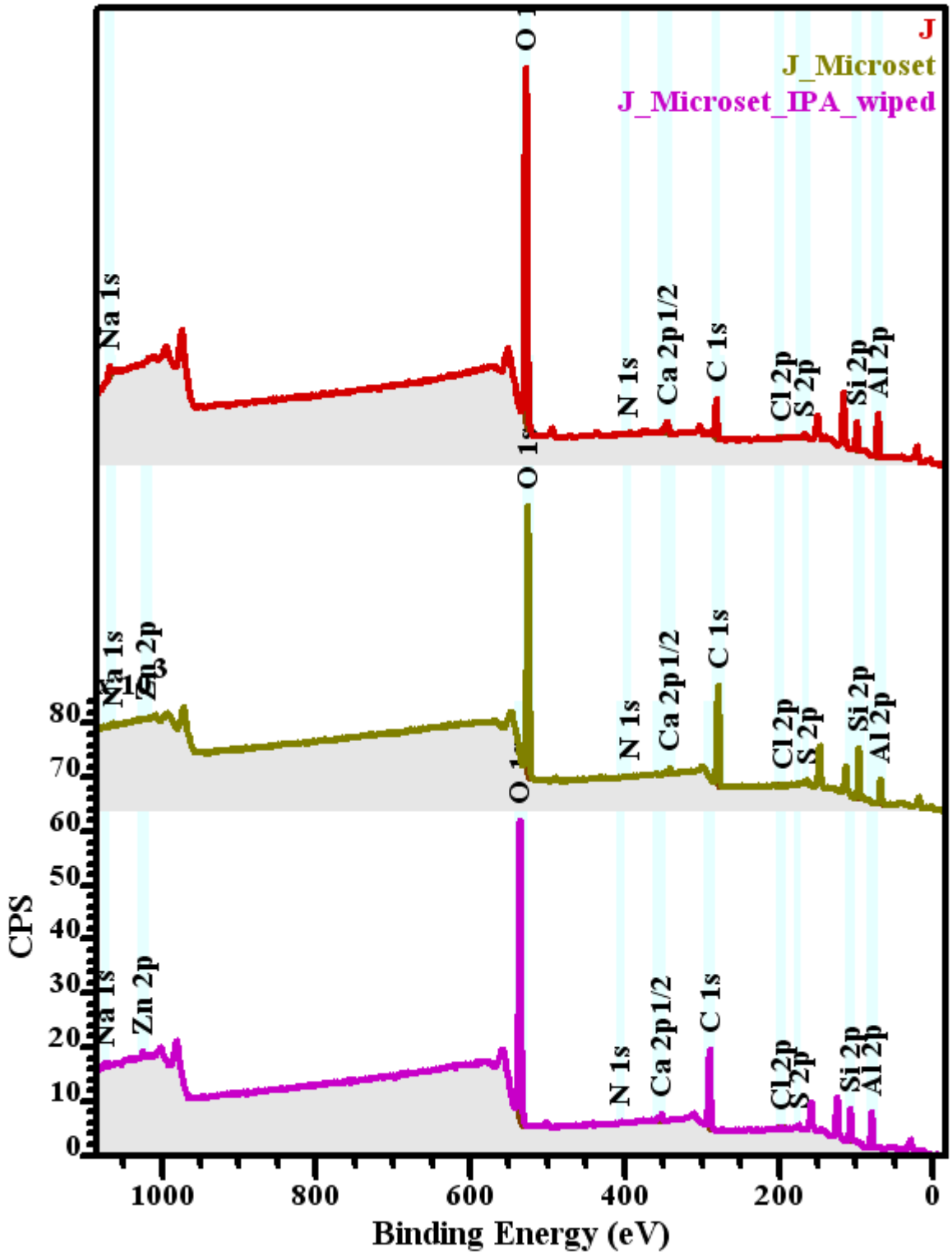
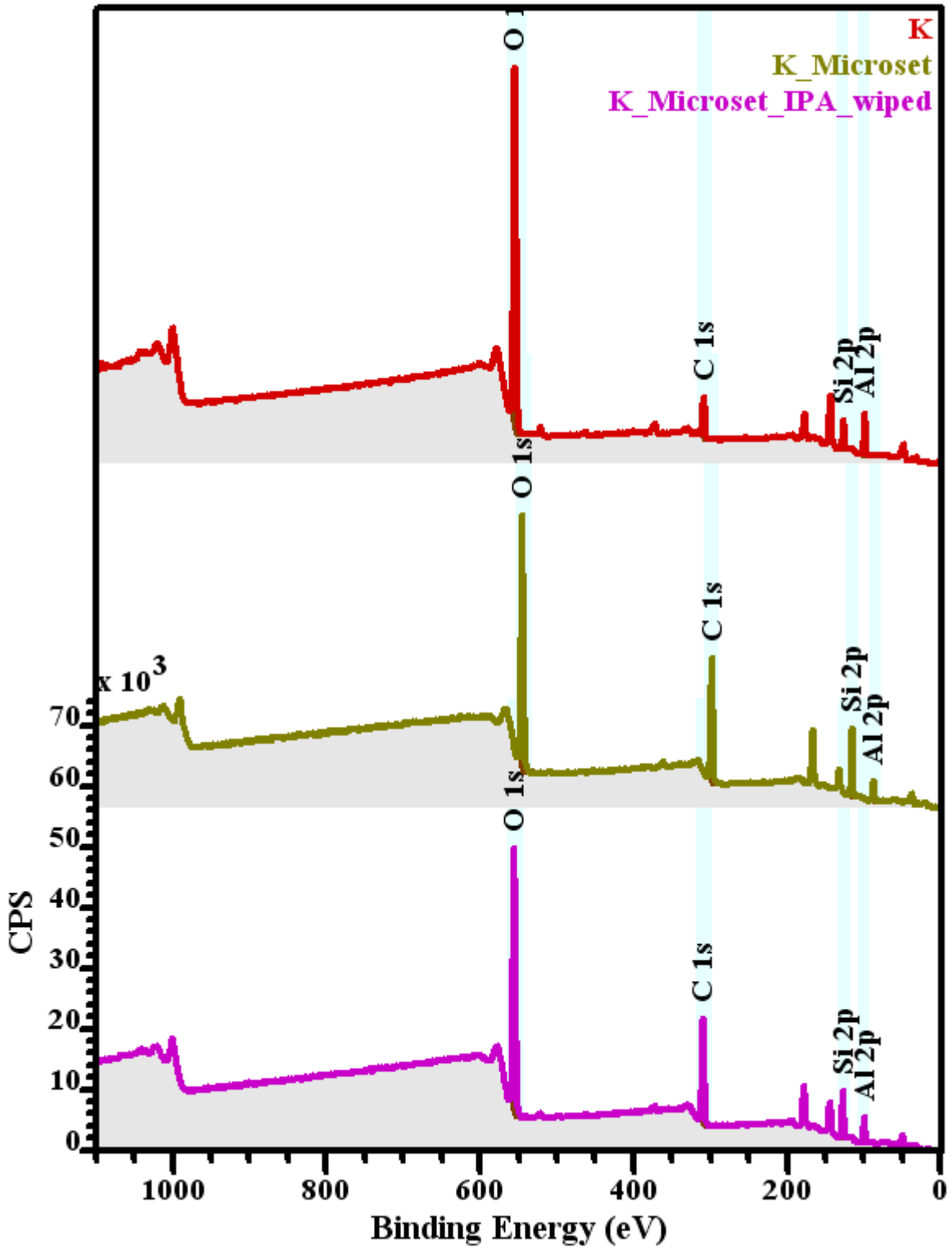
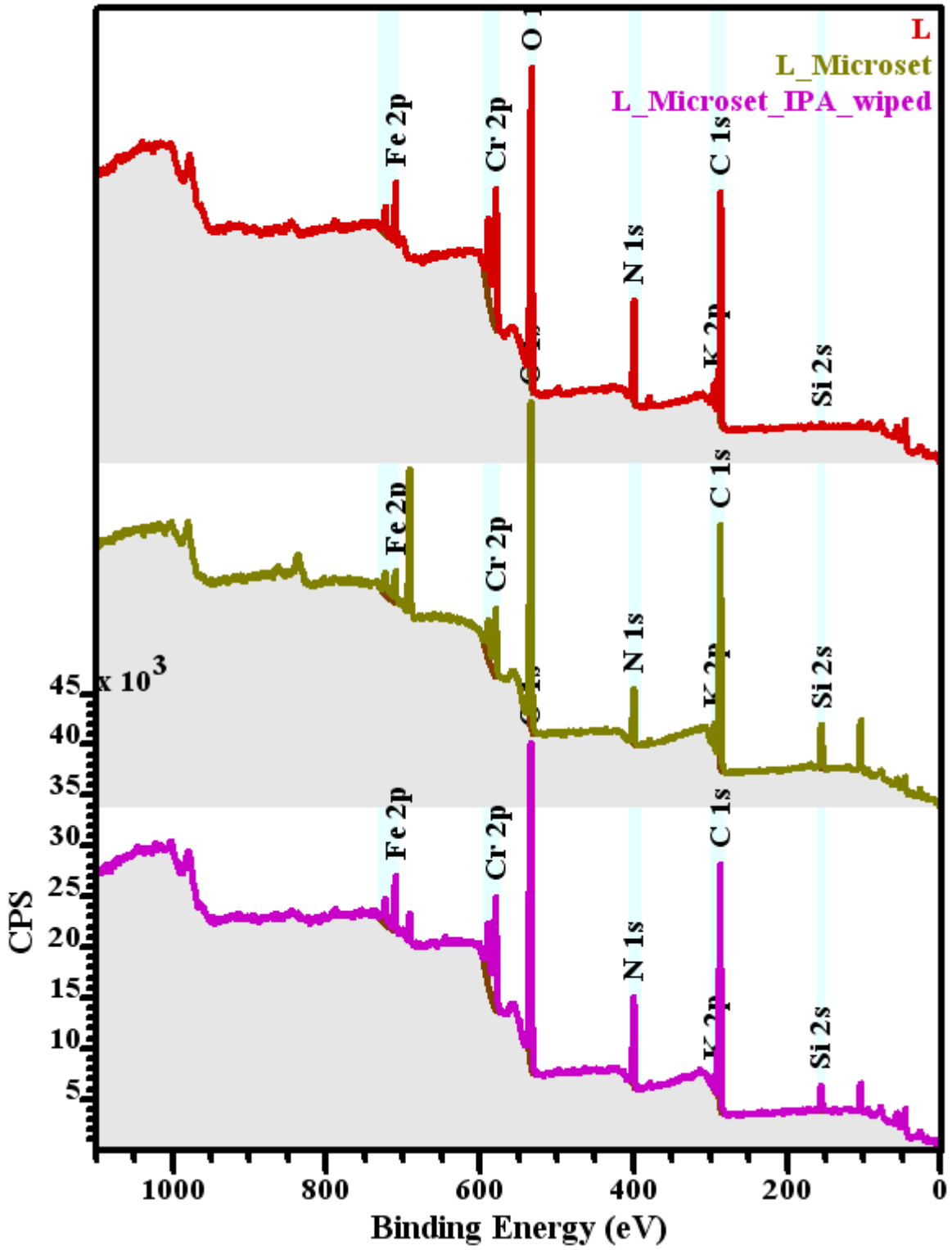
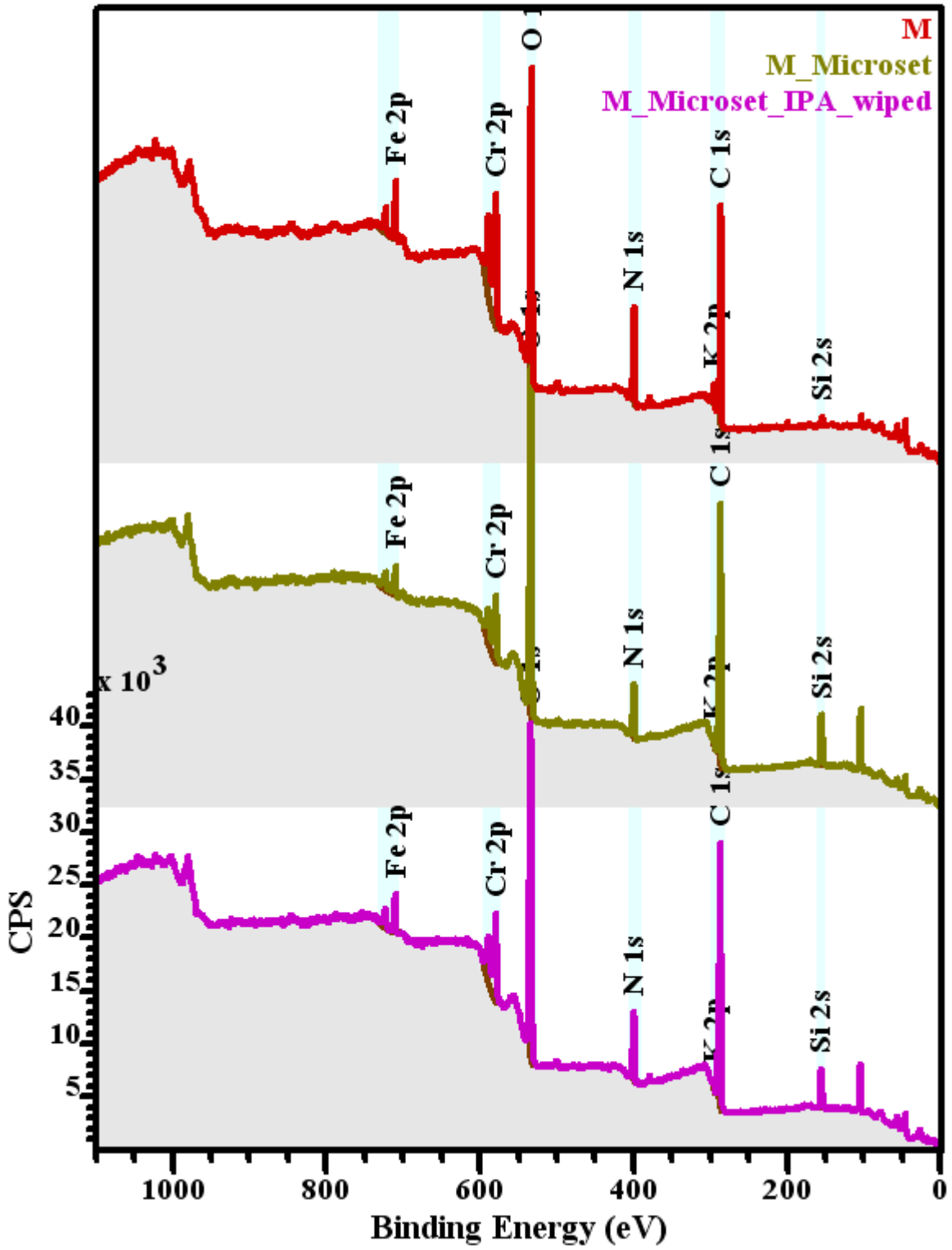
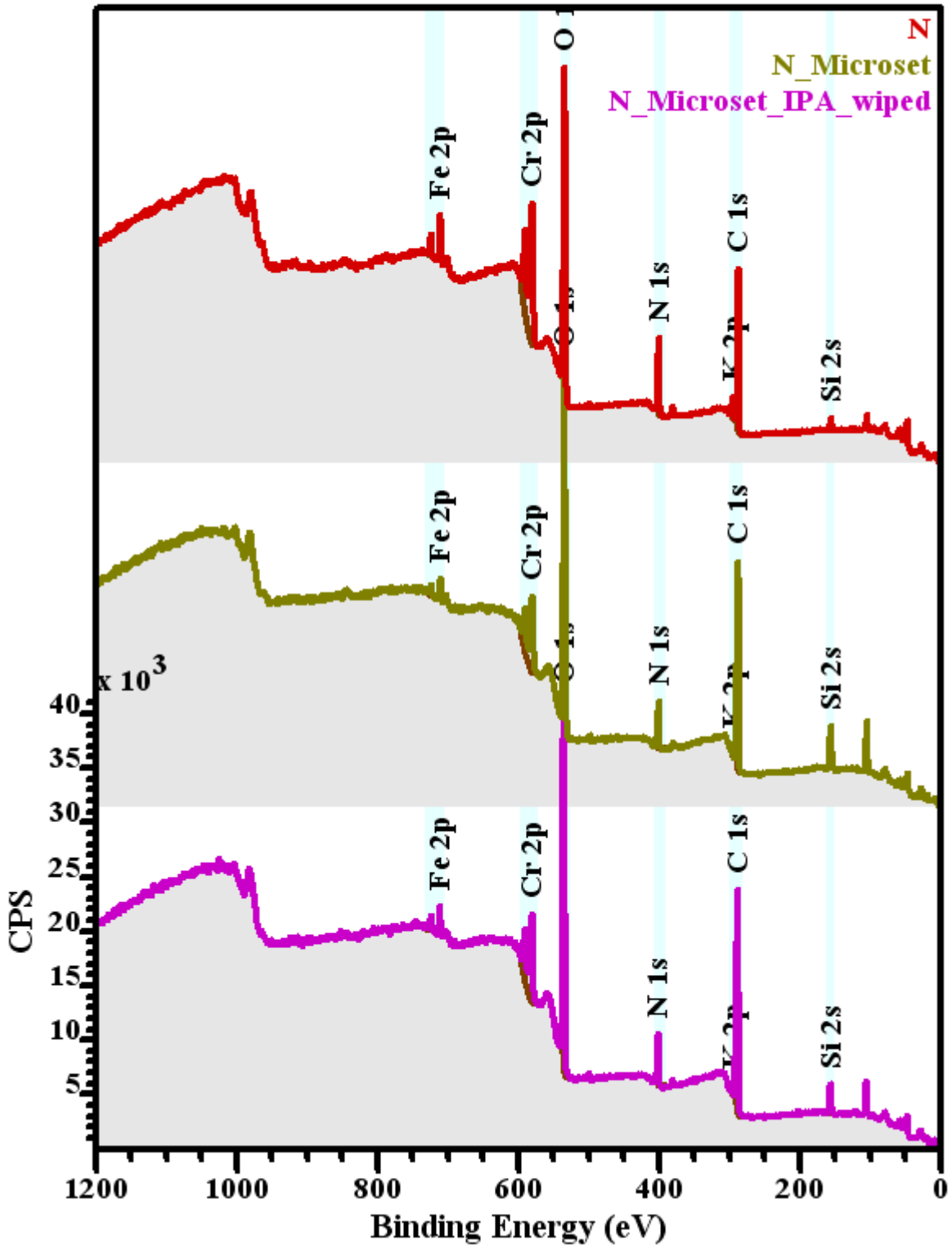
O 1s
N 1s
Cl 2p
S 2p
CPS
Binding Energy (eV)
1000
800
600
400
200
0

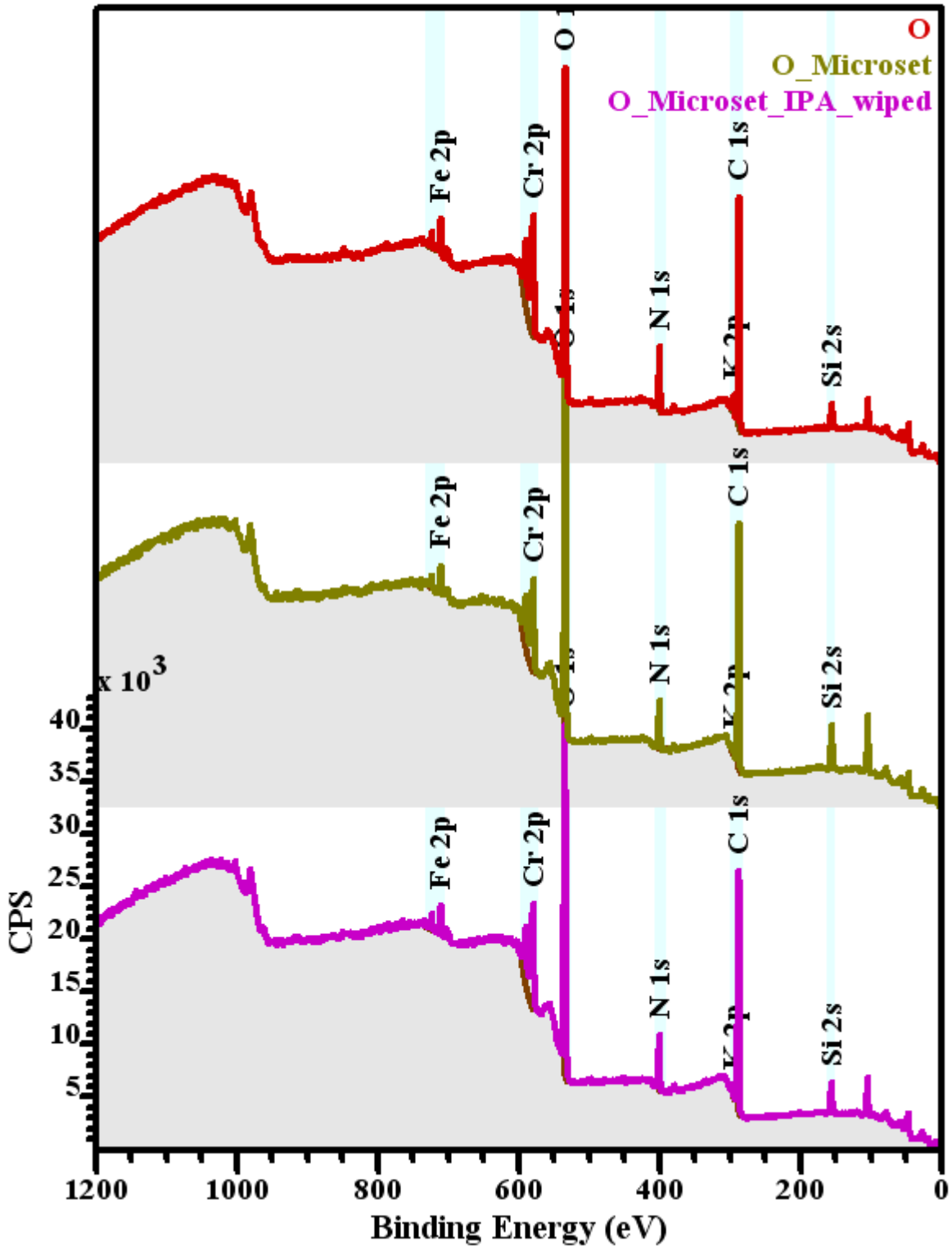

J
J_Microset
J_Microset_IPA_wiped
Na 1s
Zn 2p
O 1s
N 1s
Ca 2p 1/2
C 1s
Cl 2p
S 2p
Si 2p
Al 2p
CPS
0
10
20
30
40
50
60
70
80
1000
800
600
400
200
0
Binding Energy (eV)

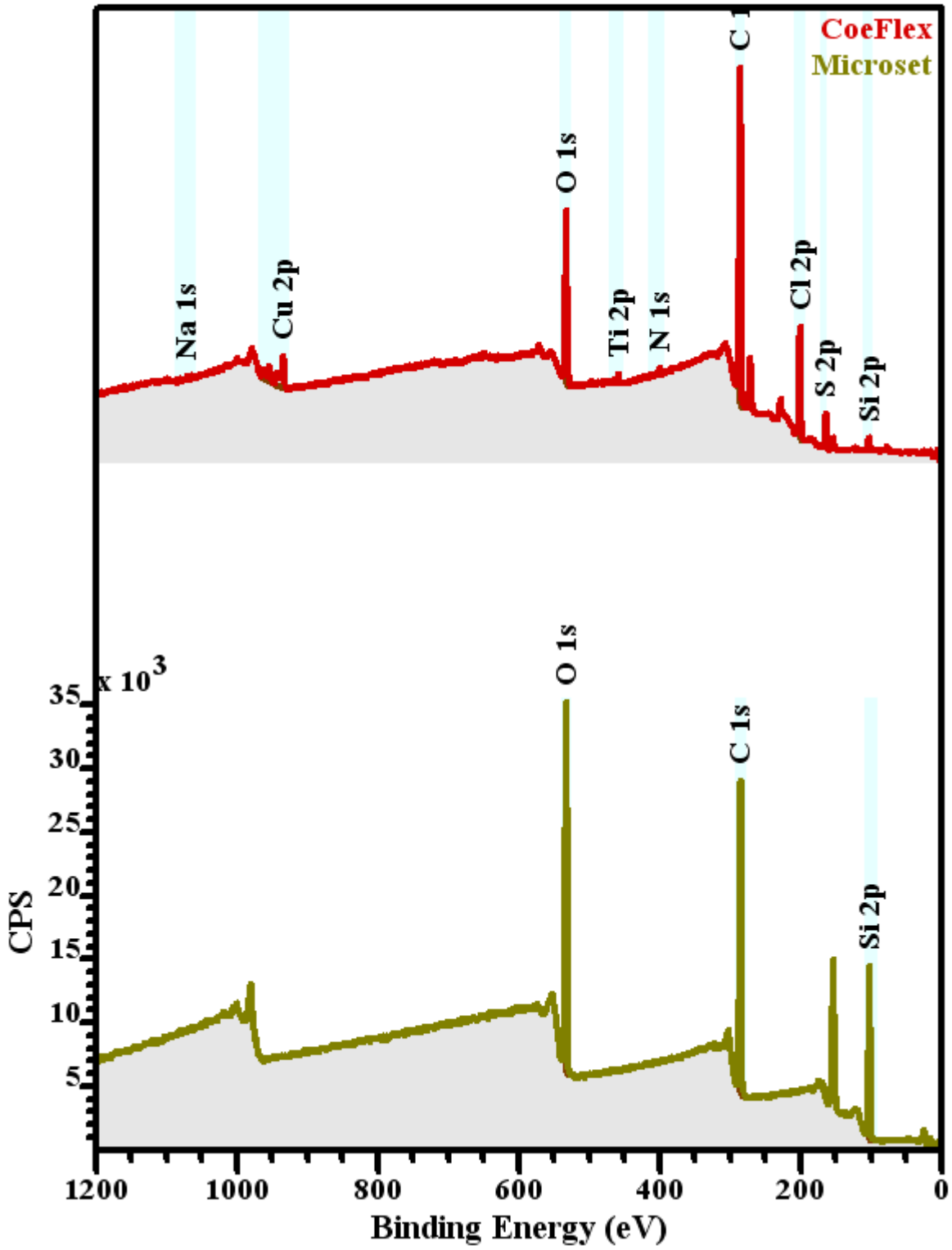

K
K_Microset
K_Microset_IPA_wiped
O 1s
C 1s
Si 2p
Al 2p
x 10^3
CPS
0
10
20
30
40
50
60
70
1000
800
600
400
200
0
Binding Energy (eV)


L
L_Microset
L_Microset_IPA_wiped
Fe 2p
Cr 2p
O 1s
N 1s
C 1s
Si 2s
CPS
x 10^3
45
40
35
30
25
20
15
10
5
1000
800
600
400
200
0
Binding Energy (eV)


M
M_Microset
M_Microset_IPA_wiped
Fe 2p
Cr 2p
O 1s
N 1s
C 1s
K 2p
Si 2s
x 10^3
CPS
40
35
30
25
20
15
10
5
1000
800
600
400
200
0
Binding Energy (eV)


N
N_Microset
N_Microset_IPA_wiped
Fe 2p
Cr 2p
O 1s
N 1s
C 1s
Si 2s
x 10^3
CPS
40
35
30
25
20
15
10
5
1200
1000
800
600
400
200
0
Binding Energy (eV)


O
O_Microset
O_Microset_IPA_wiped
Fe 2p
Cr 2p
O 1s
N 1s
C 1s
K 2p
Si 2s
x 10^3
CPS
Binding Energy (eV)


CoeFlex
Microset
Na 1s
Cu 2p
O 1s
Ti 2p
N 1s
C 1
Cl 2p
S 2p
Si 2p
O 1s
C 1s
Si 2p
x 10^3
CPS
35
30
25
20
15
10
5
1200
1000
800
600
400
200
0
Binding Energy (eV)

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