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An Auger Electron Spectroscopy Study of Surface-Preparation Contaminants

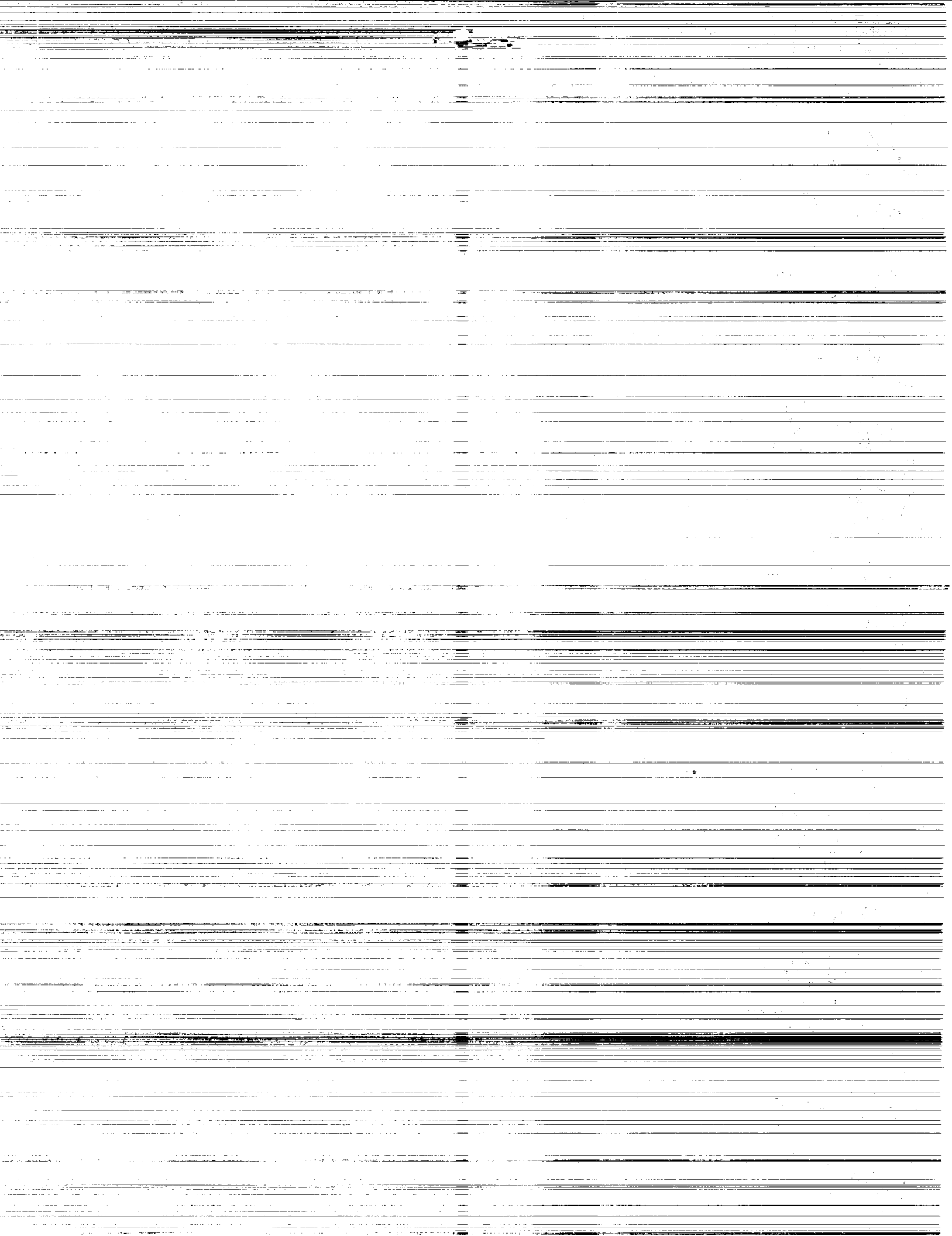
D. Wu,
R. M. Stephens,
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and P. Hopson

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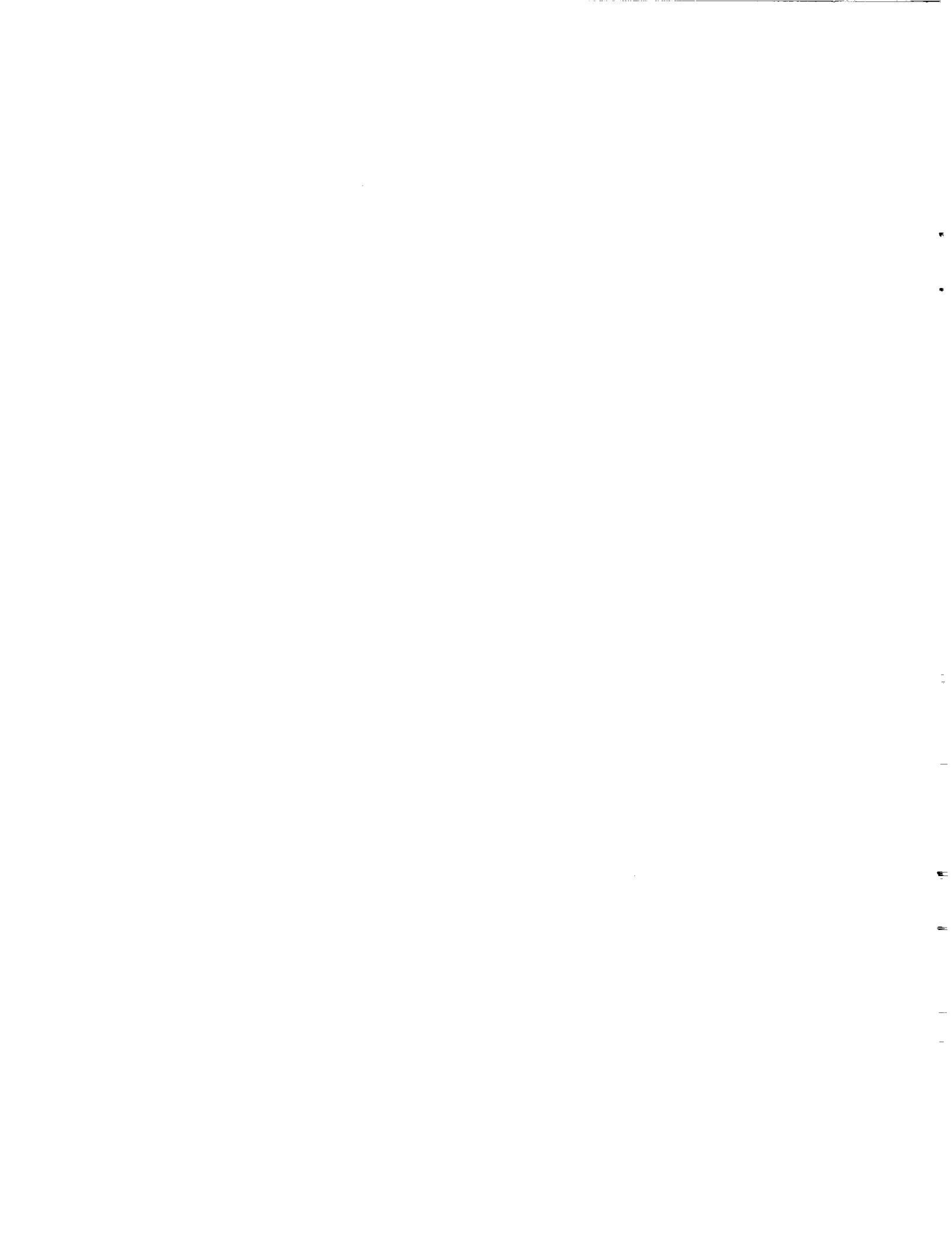
An Auger Electron
Spectroscopy Study
of Surface-Preparation
Contaminants

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Abstract

Many cleaning methods are presently being employed for the surface preparation of materials that are subsequently exposed to ultrahigh vacuum (UHV). Unfortunately, there are virtually no comparative measurements that establish the residual contaminant level of each method. In this report, 11 different cleaning methods, ranging from only detergent cleaning to electrochemical polishing, have been applied to 12 identical samples of 347 stainless steel. Two surface conditions, a standard machined surface and a mechanically polished surface, have been studied. Auger electron spectroscopy (AES) within a UHV environment has been used to detect the types of contaminants and the magnitudes found on the sample surfaces. It has been found that electrochemical polishing gave the least contaminated surface for all metals studied and that mechanically polished surfaces were significantly cleaner than the as-machined surfaces for any given cleaning method. Furthermore, it was also found that the residual contaminants left by methanol, ethanol, isopropyl alcohol, acetone, and Freon finishing rinses were almost the same.

Introduction

One of the most significant problems encountered in preparing surfaces for ultrahigh vacuum (UHV) is that of residual contamination. This contamination level (especially when large surface areas are considered) can substantially affect the ultimate pressure that is obtainable, even following a bakeout. There are many different cleaning methods presently used in research laboratories (refs. 1 to 9), but most researchers depend on their intuition to judge the effectiveness of the method since very few comparison studies have been made.

In this study, comparisons are made of the contamination on the surfaces of 12 identical samples of 347 stainless steel with two different surface conditions, a standard machined surface (considered "rough" and designated by "B") and a mechanically polished surface (considered "smooth" and designated by "A"). These samples were cleaned by 11 different methods that are often used in research laboratories as a standard practice for the preparation of materials that are to be within a UHV environment. Auger electron spectroscopy (AES) was employed to evaluate and compare the residual contamination levels of the different methods. The sensitivity of this method is less than 10^{-2} (1 atomic percent) for every elemental aggregate (except hydrogen). This method is also limited by the detection of Auger electrons other than those from the top sur-

face layer since the inelastic mean-free path for some transitions is greater than 10 \AA .

Experimental Apparatus and Procedure

Sample Preparation

The 347 stainless steel samples were cut to a disk geometry with a 10-mm diameter and a 1-mm thickness and were prepared in the following ways.

Samples A1 to A12. Samples A1 to A12 were machined to a 32- $\mu\text{in.}$ finish and were mechanically polished. The samples were mounted on a metal block by using wax and were polished with successively smaller sized abrasive materials (SiC/H₂O: 600 grit and 5- μm grit; and silica salts: 0.05 μm). After polishing, the samples were rinsed in acetone to remove the wax and were then rinsed in deionized water.

Samples B1 to B12. Samples B1 to B12 were machined to a 32- $\mu\text{in.}$ finish.

Surface Preparation

The sample surfaces were prepared, in general, for UHV applications using UHV techniques. In all cases (where possible), reagent-grade (high-purity) chemicals were used. The preparations extend from the condition of no cleaning (as-machined or polished) to 11 different cleaning procedures that involve the most often used combinations of detergents, solvents, and acids; this was followed by deionized H₂O or solvent rinses. Simultaneous stirring or ultrasonic agitation with the cleaning procedures was also employed. The details of these procedures can be found in the appendix.

Apparatus and Procedure

The experimental apparatus for Auger electron spectroscopy (AES) is shown in figure 1. The samples were introduced into the AES system within 1 hour after cleaning in order to minimize the contamination from the environment and provide some consistent level of atmospheric exposure. The samples were individually mounted on a sample holder where the surface normal is inclined 60° to the analyzer axis. A schematic of this apparatus is shown in figure 1(a). Several specimens were introduced into the system load lock and mounted on the carousel holder at each loading, and then they were sequentially rotated into the analysis position. The samples were exposed to UHV (Pressure $< 5 \times 10^{-10}$ torr) for about 24 hours, and then the analysis was conducted. The AES data were obtained with a cylindrical mirror analyzer (CMA) operated with a coaxial gun at an energy of 3 keV and a beam current of 0.5 μA .

The angle of incidence was 60° to the specimen surface normal. Room temperature AES spectra were taken at three different randomly selected locations on the surface. The average intensity for each element was then normalized by the iron LMM Auger transition intensity at 651 eV.

Results and Discussion

The surface of the samples following the 12 surface treatments (see the appendix) was mainly contaminated with oxygen, carbon, and sulfur. Other surface contaminations included phosphorus, potassium, silicon, calcium, chlorine, and nitrogen. These normalized AES peak intensities are presented in table 1 for the smooth surface and in table 2 for the rough surface. The values in parentheses are the same data except that the relative Auger sensitivity of the elements has been taken into account. Figure 2, which presents electron energy plotted against the derivative AES spectra $dN(E)/dE$, shows a sputter-cleaned 347 stainless steel sample that is presented for reference as a clean, contaminant-free surface.

The spectra for samples B1 and A1 (both no cleaning) are shown in figures 3 and 4, respectively. Figure 3 (sample B1) shows that the machined surface was mainly contaminated by carbon, sulfur, and oxygen, probably from residual machine oil. As is evident from the substantial reduction in AES signal intensities, figure 4 (sample A1) shows that mechanical polishing reduced the surface porosity and inclusions (and therefore the surface area), thus minimizing the traps for oil and other contaminants. The oxygen signal is not decreased by the polishing because normal room-temperature oxidation occurs on both surfaces following the machining and polishing. Furthermore, the oxygen signal is larger than that in figure 3 only because of the heavy carbon overlayer.

Samples A2/B2 (A2 and B2) and A3/B3 (A3 and B3) were all prepared with the same degreasing condition and were finished with deionized water, but A2/B2 were prepared without ultrasonic agitation and A3/B3 were prepared with ultrasonic agitation. Samples A4/B4 and A5/B5 were prepared with the same degreasing conditions and were finished with methanol, but A4/B4 were prepared without ultrasonic agitation and A5/B5 were prepared with ultrasonic agitation. It was found that for a smooth surface (the A samples), the contamination level left on the surface was virtually the same for samples both with and without ultrasonic agitation, but for a rough surface (the B samples), the contamination level of oxygen, carbon, phosphorus, and chlorine was significantly reduced with ultrasonic agitation compared to that without ultrasonic agitation. This may

be because ultrasonic cleaning vibrates off most of the particulates on a rough surface and enhances cleaning of the mechanical grooves. An exception was noticed with methanol finishing (for both smooth and rough surfaces) in that ultrasonic cleaning left a higher carbon concentration compared to that without ultrasonic cleaning. This is somewhat surprising, but we conducted this experiment many times, and always observed the same behavior. Perhaps the added energy of the ultrasonic agitation promoted carbon bonding to the surface.

Samples A3/B3 to A9/B9 were prepared with the same degreasing conditions but with different finishing rinses (methanol, ethanol, isopropyl alcohol, acetone, and Freon¹). A comparison of these different finishing rinses for both smooth and rough surfaces showed that the O/Fe ratio is almost the same for all the specimens, which is as expected. For a rough surface, acetone finishing has the lowest C/Fe and S/Fe ratios, but for a smooth surface, acetone has the highest C/Fe and S/Fe ratios. This result may be explained by considering that acetone is the more efficient solvent for removing oils and contaminants from the rough surface, but on a smooth surface the residue left by the acetone is greater than that for the other solvents. A methanol/ethanol finishing rinse has the lowest C/Fe and S/Fe ratios on smooth surfaces but the highest on rough surfaces, which probably indicates that large-molecule solvents like acetone and isopropyl alcohol, as compared with ethanol and methanol, are more efficient in removing oils and other contaminants from the surface but leave more residual contaminants from the impurities in the solvent itself. The reason for this effect may be the higher physical interaction potential for contaminant molecules.

Samples A10/B10 to A11/B11 were prepared with detergent degreasing plus chemical cleaning. The representative spectra are shown in figures 5 and 6. A comparison between these strong acid cleaning methods shows that the difference between smooth and rough surface samples is small, quite probably because acid etching removed most of the contamination. Comparing the Varian Associates recipe with the Stanford linear accelerator (SLAC) recipe for samples with a rough surface shows that the Varian recipe has a higher C/Fe ratio but the SLAC recipe has a much higher O/Fe ratio, even higher than that for ordinary alcohol finishing methods. It was also observed that the SLAC recipe has higher P/Fe, Si/Fe, and Ca/Fe ratios than the Varian recipe, probably the result of the alkaline soak after

¹ Freon: Registered trademark of E. I. du Pont de Nemours & Co., Inc.

the acid cleaning. The impurity level of the alkaline soak did affect the final contamination of the surface. We also noticed that the chlorine concentration for the SLAC recipe is higher than that for the Varian recipe.

Electrochemical polishing (samples A12/B12) provided the cleanest surface of all methods, partly because of the polishing that reduced the surface area and the chemical removal of the contaminants. The chilled methanol-perchloric bath formed a passivating chlorine-bearing layer on the top of the surface, as can be seen from figure 6. The nickel peak at 848 eV and the chromium peak at 529 eV have disappeared completely. These are valence transitions suggesting that the nickel and chromium have formed a chemical bond with the chlorine and that the resulting chlorine-bearing layer on the surface may have a passivating effect that inhibits adsorption of other species. However, if chlorine is an undesirable specie, this may not be a preferred cleaning method. Figures 7 and 8 show bar graph representations of tables 1 and 2 for the smooth and rough surfaces, respectively. The total intensities of the contaminants give a general indication of the effectiveness of the surface preparation.

Finally, in all the aforementioned procedures, the quality of the solvents and chemicals was found to be important. Reagent-grade chemicals produced the least contaminated surface (for a given recipe), whereas chemicals taken from industrial containers, such as a 500-gal drum, were the most contaminated. As one would expect, the cleanliness of the laboratory beakers and containers used in these recipes was also a major factor.

Concluding Remarks

Comparisons have been made between different cleaning methods often used in the research laboratory as a surface preparation for ultrahigh vacuum systems. The results of this comparative analysis showed that mechanical polishing is a very effective way to reduce the level of surface contamination. The results of the smooth surface without any pre-cleaning were better than the results of the rough surface with chemical cleaning. For a smooth surface it was observed that a methanol/ethanol finishing rinse is better than an isopropyl alcohol/Freon/acetone finishing rinse, but for a rough surface the latter rinse is superior. The probable reason for this is that the surface area for a smooth surface is much smaller, and the residue from a large-molecule solvent like acetone and isopropyl alcohol is competitive with the residual contamination left from the polishing. On the other hand, there is a greater amount of carbon-bearing contaminants being trapped in the inclusions and general surface defects on a rough surface for which the large-molecule solvent is more efficient. Ultrasonic cleaning is also a useful technique to improve the efficiency of a given cleaning method for a rough surface. These results also showed that chemical cleaning can remove most of the carbon and sulfur and is significantly better than just detergent cleaning plus solvent rinse finishing.

From this analysis it appears that electrochemical polishing and mechanical polishing are superior methods for preparing surfaces, but when polishing is not possible, the surfaces should be chemically cleaned.

NASA Langley Research Center
Hampton, VA 23665-5225
January 22, 1990

Appendix

Cleaning Method

A1/B1:

No cleaning.

A2/B2:

1. Cleaned by agitated detergent solution for 5 minutes at a temperature of 190°F.
2. Rinsed by agitated deionized water for 10 minutes at a temperature of 190°F.
3. Rinsed by agitated deionized water for 5 minutes at a temperature of 190°F.
4. Dried by hot air.

A3/B3:

1. Ultrasonically cleaned by detergent solution for 15 minutes at a temperature of 190°F.
2. Ultrasonically rinsed by deionized water for 10 minutes at a temperature of 190°F.
3. Ultrasonically rinsed by deionized water for 5 minutes at a temperature of 190°F.
4. Dried by hot air.

A4/B4:

1. Cleaned by agitated detergent solution for 15 minutes at a temperature of 190°F.
2. Rinsed by agitated deionized water for 10 minutes at a temperature of 190°F.
3. Rinsed by agitated deionized water for 5 minutes at a temperature of 190°F.
4. Rinsed by agitated methanol for 5 minutes.
5. Dried by hot air.

A5/B5:

1. Ultrasonically cleaned by detergent solution for 15 minutes at a temperature of 190°F.
2. Ultrasonically rinsed by deionized water for 10 minutes at a temperature of 190°F.
3. Ultrasonically rinsed by deionized water for 5 minutes at a temperature of 190°F.
4. Ultrasonically rinsed by methanol for 5 minutes.
5. Dried by hot air.

A6/B6:

1. Ultrasonically cleaned by detergent solution for 15 minutes at a temperature of 190°F.
2. Ultrasonically rinsed by deionized water for 10 minutes at a temperature of 190°F.
3. Ultrasonically rinsed by deionized water for 5 minutes at a temperature of 190°F.
4. Ultrasonically rinsed by ethanol for 5 minutes.
5. Dried by hot air.

A7/B7:

1. Ultrasonically cleaned by detergent solution for 15 minutes at a temperature of 190°F.
2. Ultrasonically rinsed by deionized water for 10 minutes at a temperature of 190°F.
3. Ultrasonically rinsed by deionized water for 5 minutes at a temperature of 190°F.
4. Ultrasonically rinsed by isopropyl alcohol for 5 minutes.
5. Dried by hot air.

A8/B8:

1. Ultrasonically cleaned by detergent solution for 15 minutes at a temperature of 190°F.
2. Ultrasonically rinsed by deionized water for 10 minutes at a temperature of 190°F.
3. Ultrasonically rinsed by deionized water for 5 minutes at a temperature of 190°F.
4. Ultrasonically rinsed by Freon for 5 minutes.
5. Dried by hot air.

A9/B9:

1. Ultrasonically cleaned by detergent solution for 15 minutes at a temperature of 190°F.
2. Ultrasonically rinsed by deionized water for 10 minutes at a temperature of 190°F.
3. Ultrasonically rinsed by deionized water for 5 minutes at a temperature of 190°F.
4. Ultrasonically rinsed by acetone for 5 minutes.
5. Dried by hot air.

A10/B10 (recipe from Varian):

1. Degreased by vapor in trichloroethylene for 5 minutes.
2. Rinsed by deionized water for 1 minute.
3. Cleaned by agitated detergent for 5 minutes at a room temperature of 190°F.
4. Rinsed by hot deionized water for 2 minutes.
5. Acid cleaned by 33 percent HNO₃, 33 percent HF, 33 percent H₂O bath (by volume) at room temperature for about 20 sec.
6. Rinsed by hot deionized water for 2 minutes.
7. Dried by hot air.

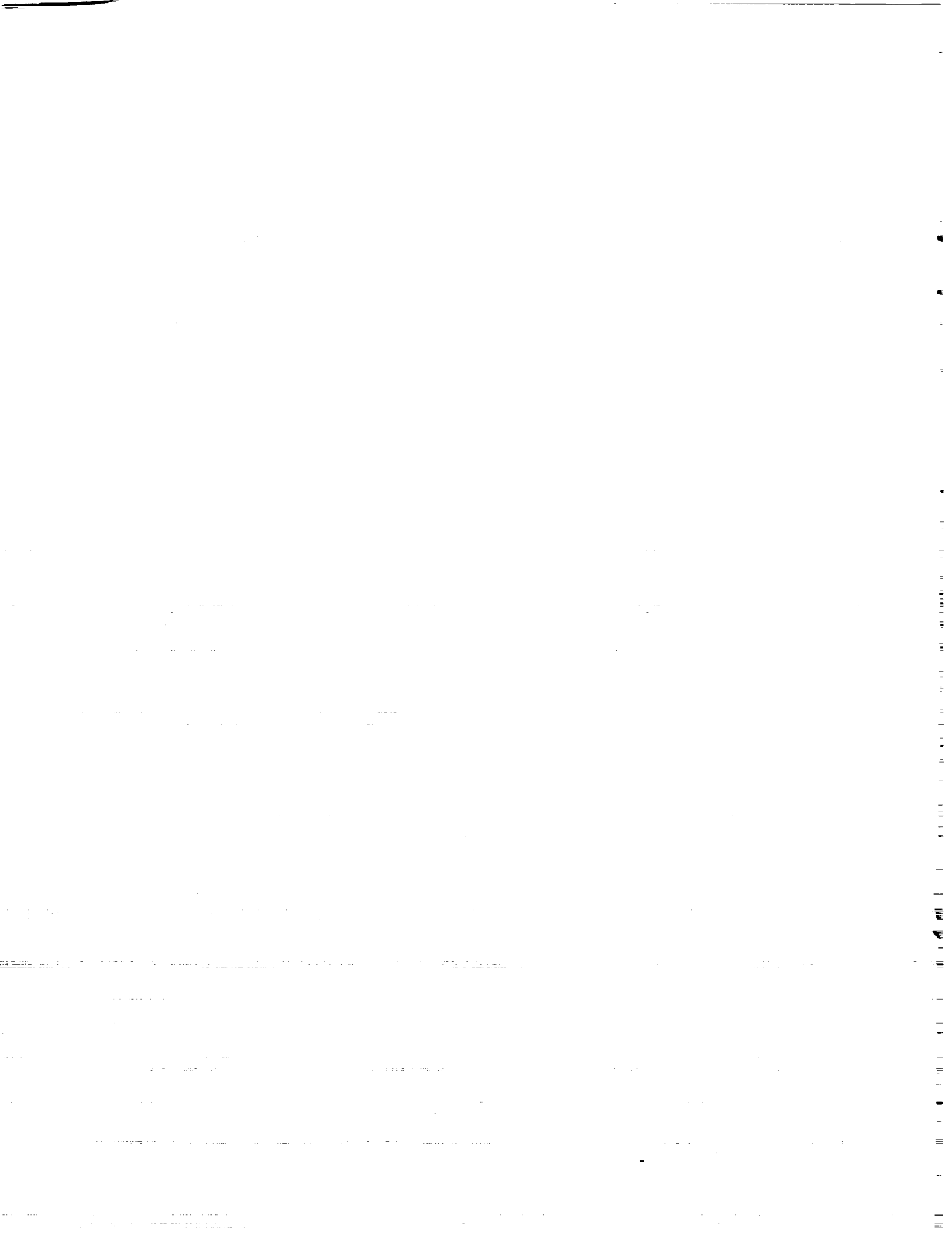
A11/B11: (recipe from SLAC):

1. Degreased by vapor in hot trichloroethane vapor for 5 minutes.
2. Rinsed by cold tap water for 1 minute.
3. Cleaned by alkaline soak for 5 minutes at a temperature of 190°F.
4. Rinsed by cold tap water for 2 minutes.
5. Immersed in a stainless steel pickle consisting of:
 - (a) 1 part nitric acid (42 Baumé)
 - (b) 1 part hydrofluoric acid (48 percent)
 - (c) 1 part water

- (d) Temperature: room
- (e) Time: until part is free from scale and oxide (about 10 sec)
- 6. Rinsed by cold tap water for 2 minutes.
- 7. Cleaned by alkaline soak for 5 minutes at a temperature of 190°F.
- 8. Rinsed by cold tap water for 2 minutes.
- 9. Immersed in 25 to 30 percent nitric acid (by volume) for 2 minutes.
- 10. Rinsed by cold tap water for 2 minutes.
- 11. Rinsed by cold deionized water for 2 minutes.
- 12. Rinsed by hot deionized water for 2 minutes.
- 13. Rinsed by isopropyl alcohol at a temperature of 115°F.

A12/B12 (electrochemically polished):

- 1. Ultrasonically cleaned by detergent for 15 minutes at a temperature of 190°F.
- 2. Ultrasonically rinsed by deionized water for 10 minutes at a temperature of 190°F.
- 3. Ultrasonically rinsed by deionized water for 5 minutes at a temperature of 190°F.
- 4. Electrochemical polishing:
 - (a) 6 percent perchloric acid (70 percent)
 - (b) 94 percent methanol
 - (c) Temperature: -70°C
 - (d) Electrical current: 0.2 A/cm^2
 - (e) Time: about 20 sec
- 5. Rinsed by deionized water.
- 6. Dried by hot air.



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Table 1. Auger Peak-to-Peak Ratios for Smooth Surface

[Sensitivity corrections are given in parentheses]

Sample		O/Fe	C/Fe	S/Fe	P/Fe	Cl/Fe	N/Fe	Si/Fe	Ca/Fe	K/Fe
Number	Description ^a									
A1	No cleaning	3.49 (8.73)	0.75 (0.75)		0.16 (0.42)			0.13 (0.23)	0.14 (0.32)	
A2	D	5.12 (12.80)	.28 (.28)		.23 (.61)	0.12 (0.63)		.12 (.21)		
A3	D(U)	4.31 (10.78)	.29 (.29)		.19 (.50)	.10 (.53)		.11 (.19)		
A4	D + methanol	4.23 (10.58)	.31 (.31)		.25 (.66)	.13 (.68)		.11 (.19)		
A5	D(U) + methanol(U)	4.31 (10.78)	.41 (.41)		.21 (.56)	.08 (.42)		.12 (.21)		
A6	D(U) + ethanol(U)	4.15 (10.38)	.38 (.38)		.19 (.50)	.11 (.58)		.13 (.23)		
A7	D(U) + isopropyl(U)	4.79 (11.98)	.44 (.44)		.22 (.58)	.17 (.89)		.13 (.23)		
A8	D(U) + Freon(U)	4.70 (11.75)	.43 (.43)		.25 (.66)	.14 (.74)		.15 (.26)		
A9	D(U) + acetone(U)	4.49 (11.23)	.47 (.47)	0.13 (0.58)	.22 (.58)	.09 (.47)		.16 (.28)		
A10	Varian CC	5.10 (12.75)	.50 (.50)	.09 (.36)	.14 (.37)	.11 (.58)	0.13 (0.21)		0.09 (.20)	
A11	SLAC CC	5.44 (13.60)	.41 (.41)		.15 (.40)	.11 (.58)			.10 (.23)	
A12	ECP	3.54 (8.85)	.28 (.28)			.34 (1.79)				

^aD: detergent.

U: ultrasonic cleaning.

CC: chemical cleaning.

ECP: electrochemical polishing.

Table 2. Auger Peak-to-Peak Ratios for Rough Surface

[Sensitivity corrections are given in parentheses]

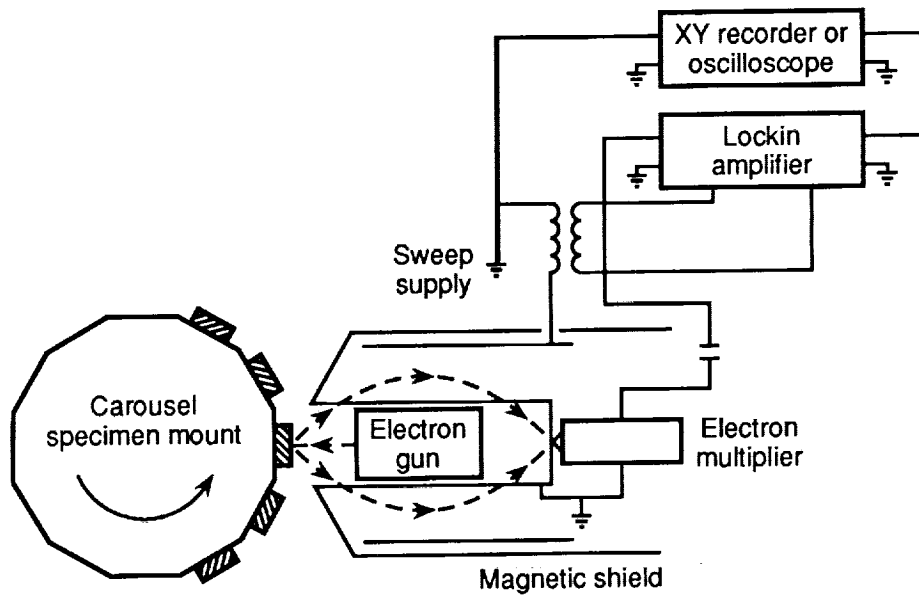
Sample Number	Description ^a	O/Fe	C/Fe	S/Fe	P/Fe	Cl/Fe	N/Fe	Si/Fe	Ca/Fe	K/Fe
		(7.50)	(20.88)	(9.84)	(1.11)				(2.77)	(7.04)
B1	No cleaning	3.00	20.88	2.46	0.42				1.23	1.76
B2	D	3.60	3.91	.43	.12			0.11		.32
B3	D(U)	3.47	2.35	.44	.10			.06		.18
B4	D + methanol	4.04	1.18	.43	.14			.08		.12
B5	D(U) + methanol(U)	3.47	1.41	.47	.08			.06		.12
B6	D(U) + ethanol(U)	3.29	1.32	.42	.11			.07		.12
B7	D(U) + isopropyl(U)	3.47	1.09	.41	.12			.08		.11
B8	D(U) + Freon(U)	3.29	1.24	.51	.10			.06		.09
B9	D(U) + acetone(U)	3.42	.92	.33	.13			.08		.07
B10	Varian CC	3.36	.60	.15		0.05	0.12	.02		
B11	SLAC CC	7.46	.49	.13	.34	.20		.20		
B12	ECP	3.34	.23			.36				

^aD: detergent.

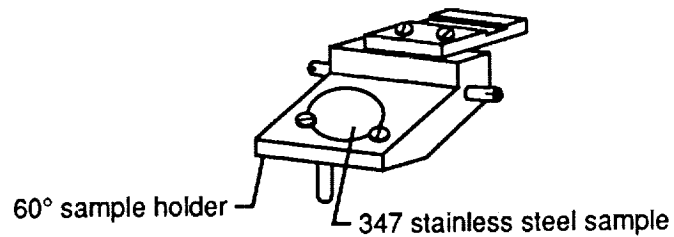
U: ultrasonic cleaning.

CC: chemical cleaning.

ECP: electrochemical polishing.



(a) Schematic of experimental apparatus for conducting AES.



(b) Stainless steel sample mounted on 60° sample holder.

Figure 1. Experimental apparatus for Auger electron spectroscopy (AES).

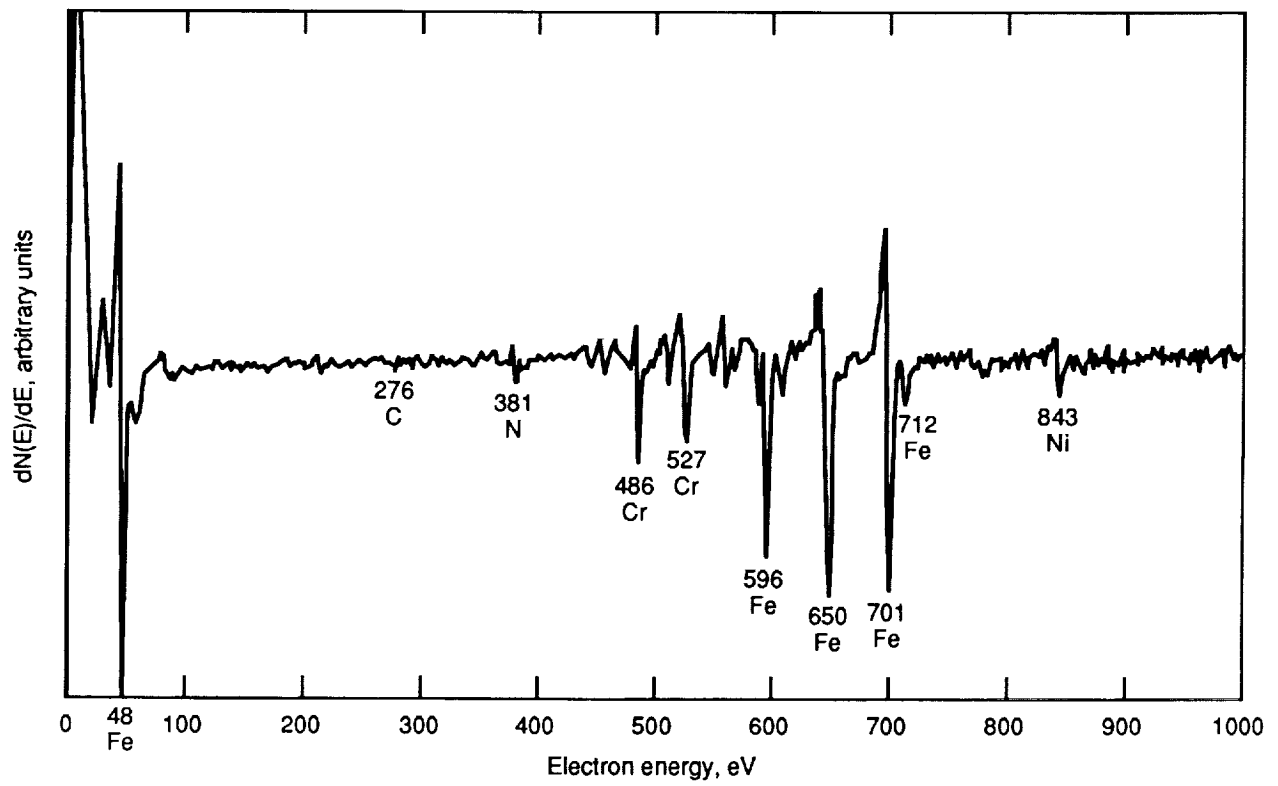


Figure 2. AES survey of sputter-cleaned 347 stainless steel sample.

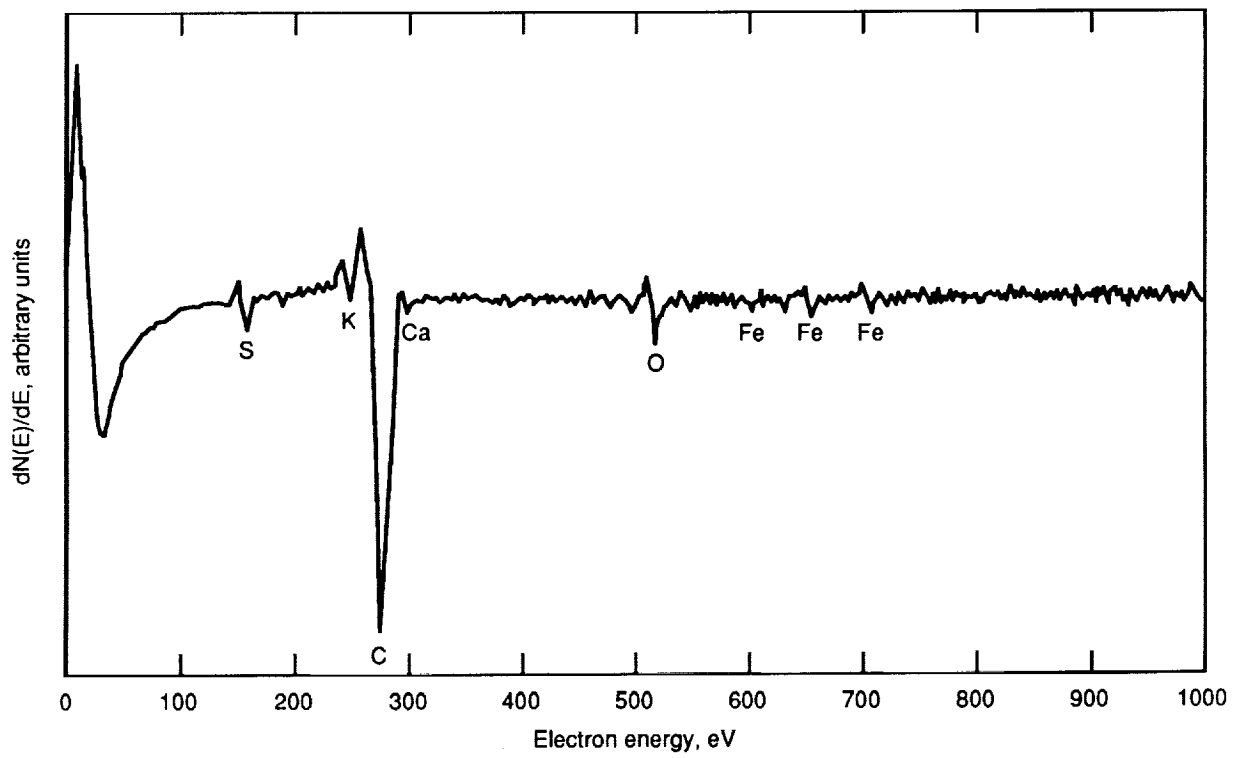


Figure 3. AES survey of machined surface without cleaning. Sample B1.

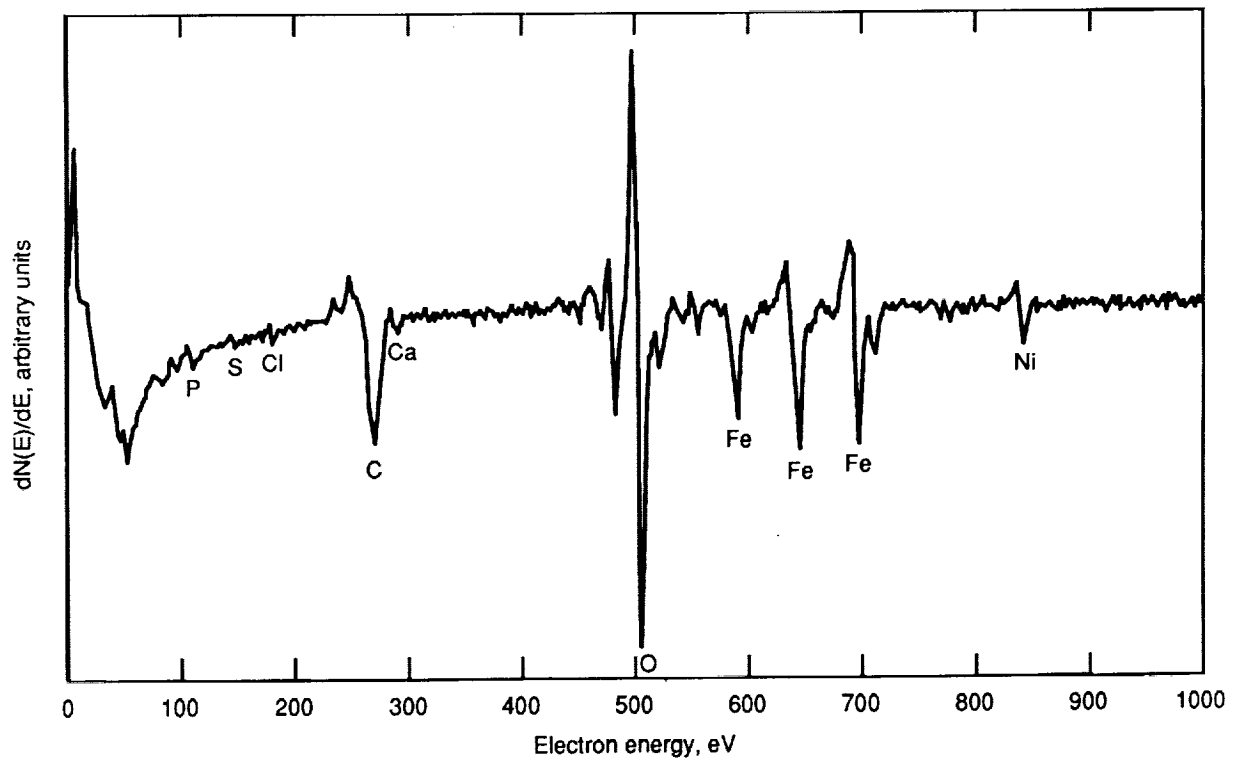


Figure 4. AES survey of polished surface without cleaning. Sample A1.

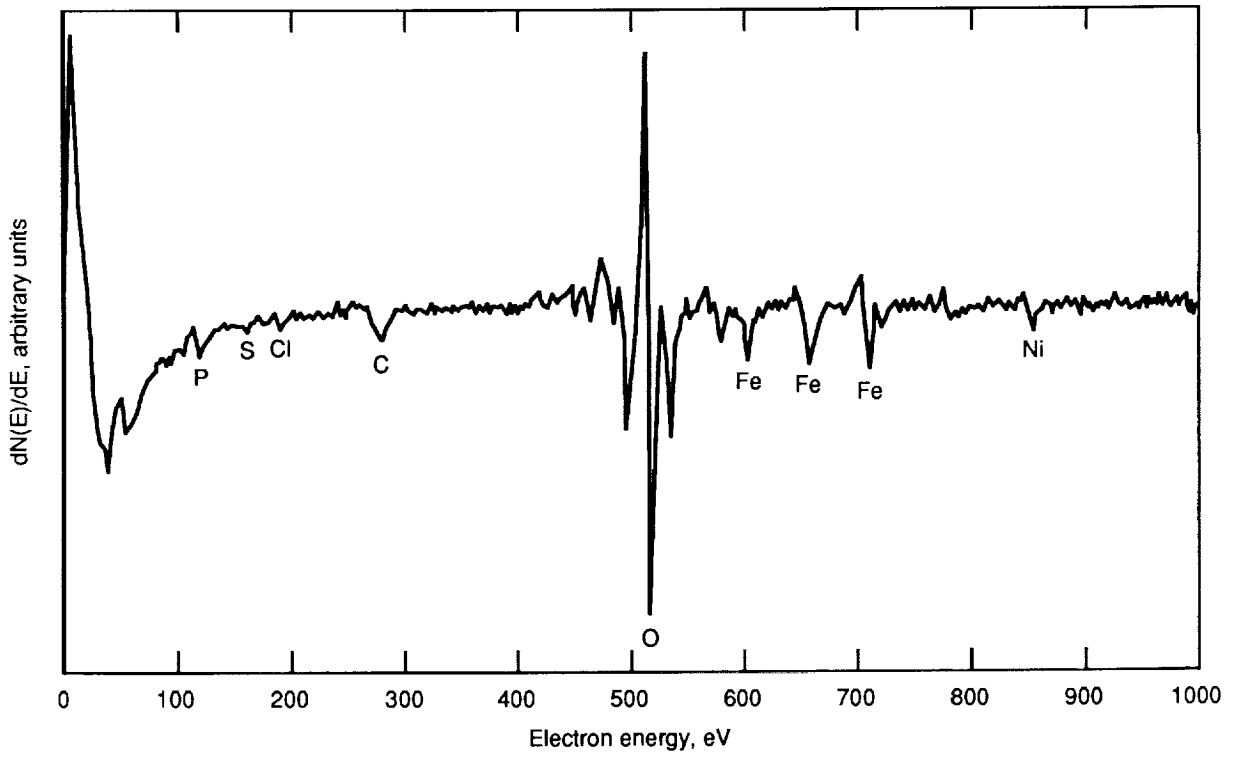


Figure 5. AES survey of sample with chemical cleaning. Samples A10/B10.

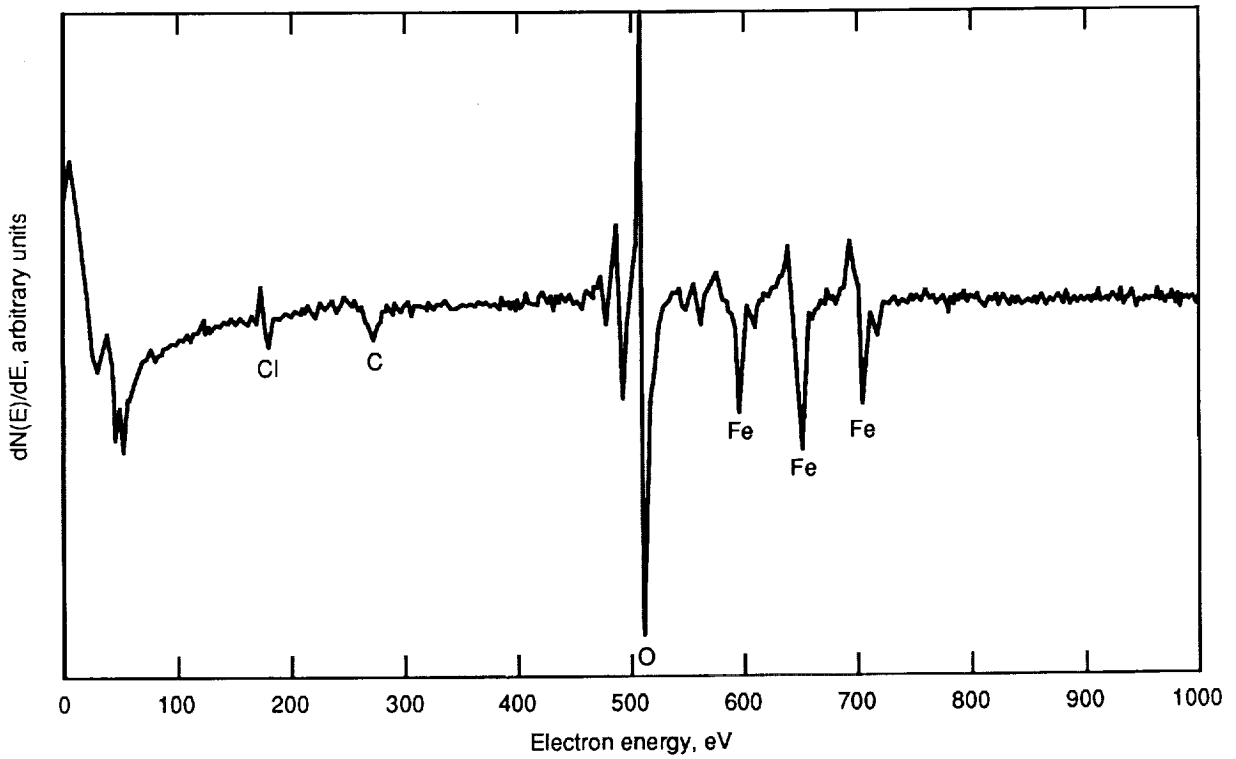


Figure 6. AES survey of sample with electrochemical polishing. Samples A12/B12.

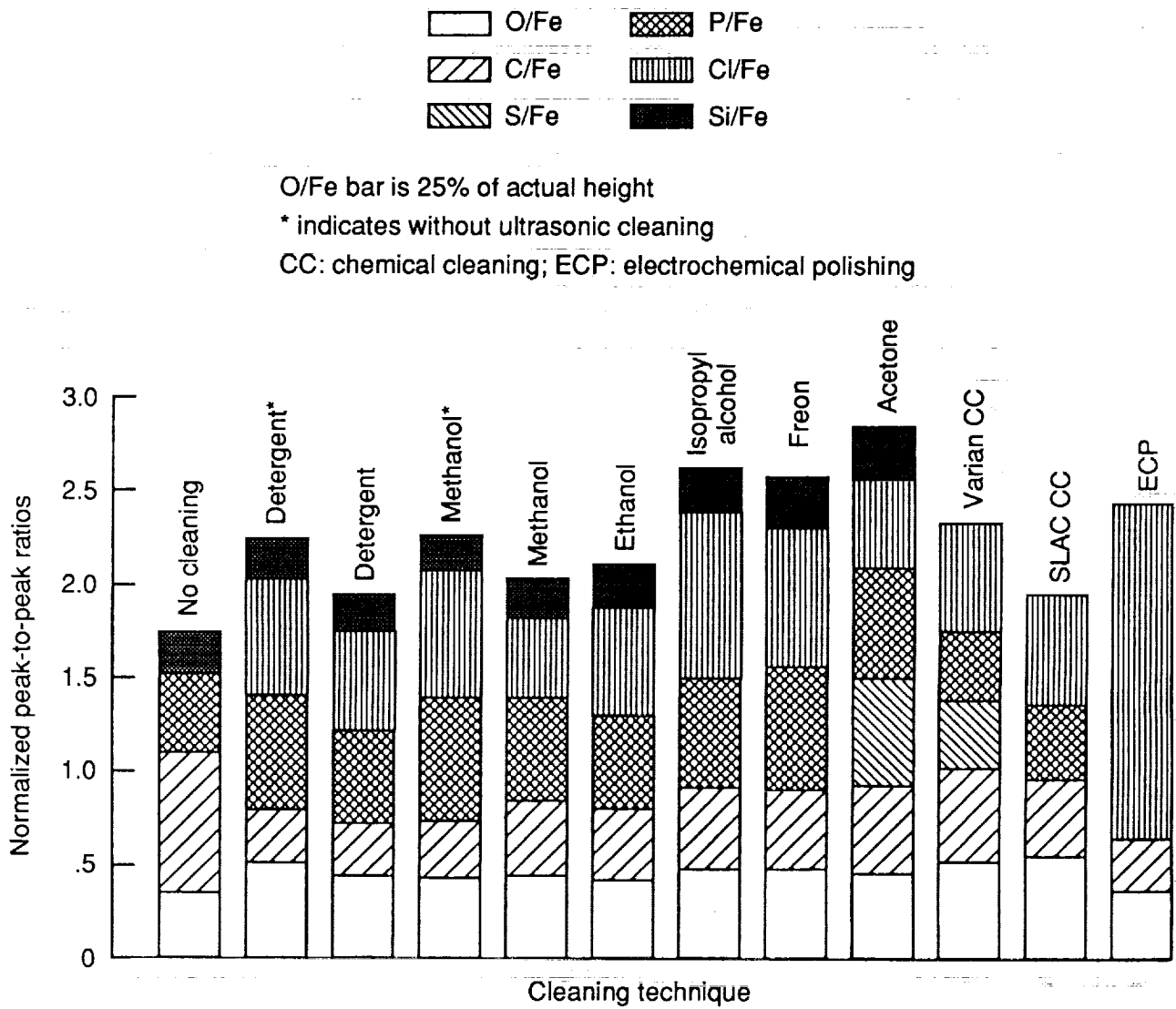


Figure 7. Bar graph of sum of contaminant intensities for smooth (polished) surface. Sensitivity corrections are included. (See table 1 for further clarification.)

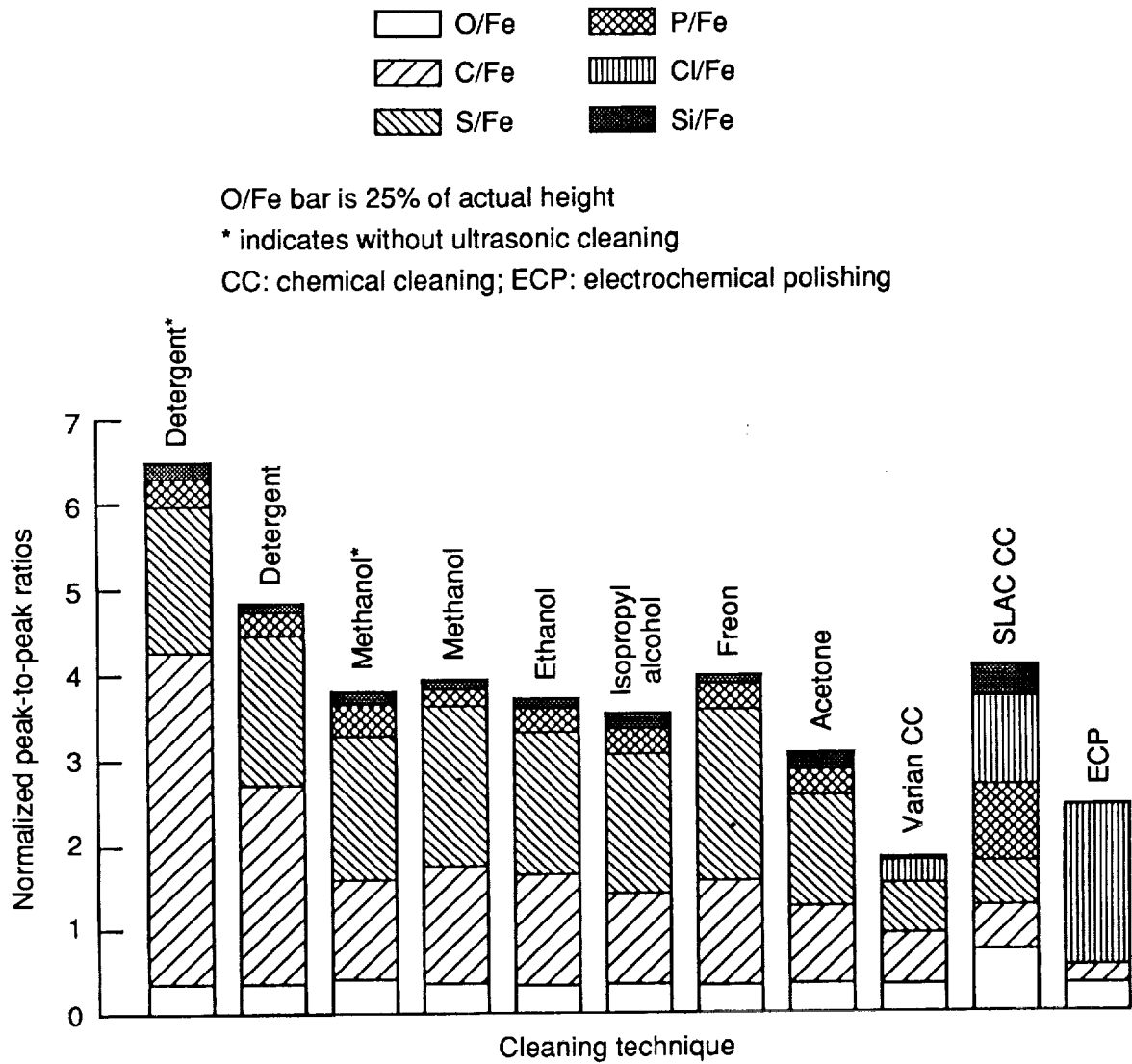


Figure 8. Bar graph of sum of contaminant intensities for rough (as-machined) surface. Sensitivity corrections are included. (See table 2 for further clarification.)

Dear Sir,

I am writing to you regarding the matter of the contract.

The contract was signed on the 15th of the month.

I am sorry that I cannot provide you with more information.

I will be in touch with you again in the near future.

Thank you for your patience and understanding.

Yours faithfully,

John Doe



Report Documentation Page

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16. Abstract Many cleaning methods are presently being employed for the surface preparation of materials that are subsequently exposed to ultrahigh vacuum (UHV). Unfortunately, there are virtually no comparative measurements that establish the residual contaminant level of each method. In this report, 11 different cleaning methods, ranging from only detergent cleaning to electrochemical polishing, have been applied to 12 identical samples of 347 stainless steel. Two surface conditions, a standard machined surface and a mechanically polished surface, have been studied. Auger electron spectroscopy (AES) within a UHV environment has been used to detect the types of contaminants and the magnitudes found on the sample surfaces. It has been found that electrochemical polishing gave the least contaminated surface for all metals studied and that mechanically polished surfaces were significantly cleaner than the as-machined surfaces for any given cleaning method. Furthermore, it was also found that the residual contaminants left by methanol, ethanol, isopropyl alcohol, acetone, and Freon finishing rinses were almost the same.			
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