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PREPARATION OF ATOMICALLY CLEAN SURFACES OF SELECTED ELEMENTS: A REVIEW *

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Surface cleaning procedures for seventy four of the elements having vapor pressures below 1.3×10^{-7} Pa at room temperature have been reviewed and evaluated. The emphasis was on in-situ procedures used to produce a clean surface on a macroscopic bulk sample in an ultra-high vacuum environment. In this review an atomically clean surface was defined to be an annealed surface (except where noted) at ambient temperature with a total surface contamination level of less than a few percent of a monolayer. Wherever possible only cleaning methods documented by element-specific, surface-analytical techniques were reviewed and subsequently incorporated into the table of recommended procedures. For some elements a variety of procedures was deemed acceptable. Any differences in methods for polycrystalline and single-crystalline surfaces of the same element have been detailed. References to the reviewed literature are grouped by element.

Contents

Glossary

AES	Auger electron spectroscopy
bcc	Body centered cubic crystal structure
cub	Cubic crystal structure
cub-dia	Cubic-diamond crystal structure

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fcc	Face centered cubic crystal structure
ELS	Energy loss spectroscopy
FEM	Field electron microscopy
FIM	Field ion microscopy
hcp	Hexagonal close-packed crystal structure
hex	Hexagonal crystal structure
ISS	Ion scattering spectroscopy
L	Langmuir (1 L ≈ 0.13 mPa s)
LEED	Low energy electron diffraction
mon	Monoclinic crystal structure
orth	Orthorhombic crystal structure
rhdr	Rhombohedral crystal structure
RHEED	Reflection high energy electron diffraction
SIMS	Secondary ion mass spectroscopy
SXAPS	Soft X-ray appearance potential spectroscopy
tetr	Tetragonal crystal structure
UHV	Ultra-high vacuum
XPS	X-ray photoelectron spectroscopy (also called ESCA – electron spectroscopy for chemical analysis)

1. Introduction

Preparation of atomically clean, elemental surfaces is required prior to experimental studies of their physical and/or chemical properties. The investigator in search of a cleaning recipe often must track the origin of any cleaning procedure back through several references only to locate a procedure that has not been verified by element-specific surface characterization techniques. Furthermore, that procedure may be neither the only nor the best procedure. The investigator's alternative is the tedious process of developing his own procedure. As surface scientists, we have been in similar situations and have concluded that a paper which reviews the documented cleaning procedures and recommends one "best" procedure for each surface should be quite valuable to the practitioners of surface science. The procedures discussed here are those used after the sample is in an ultra-high vacuum environment (i.e., after mechanical and/or electro-chemical polishing and residue removal). The maintenance of the cleanliness for time scales consistent with the experimental study is a matter of applying the appropriate vacuum technologies and is beyond the scope of this review. Usually, subsequent recleaning can be accomplished by a shorter-term repeat of the specified procedure.

In 1963, Roberts [1] published a paper on the generation of clean surfaces in high vacuum. He described the advantages and disadvantages of six general methods for preparing clean surfaces and gave specific procedures for a few elements. However, techniques for surface analysis were not available at that time to verify the effectiveness of the procedures. In this document, we have reviewed the specific cleaning techniques for more than seventy elements with vapor pressures of less than 1.3×10^{-7} Pa at room temperature. Wherever possible only those procedures verified by element-specific surface-analytical techniques (e.g., AES, XPS, SIMS, ISS) are given. Details are discussed for polycrystalline and various single-crystalline surfaces. Procedures for which no element-specific analyses were reported have been included with the appropriate caveats. From the review and assessment of the various methods, recommended procedures for each element have been combined in a table.

For the purposes of this paper, a clean surface is defined to be an annealed surface (except where noted) at ambient temperature with a total surface contamination level of less than a few percent of a monolayer. Annealing is required to insure that any studies performed will be reproducible and characteristic of a nearly defect-free surface. Estimates of the structural perfection for annealed surfaces of single crystals have been obtained from LEED intensities. A total contamination level of less than a few percent was specified as acceptably clean because (1) the two most widely used techniques for surface analysis (AES and XPS) have detection limits of about one percent of a monolayer for individual elements, and (2) such limited contamination in the first few atomic layers should not markedly affect the results of most experimental studies.

Cleaning procedures considered in this review are (1) heating to a high temperature in UHV or in a partial pressure of a reactive gas, (2) ion sputtering with the sample either at ambient or elevated temperatures, (3) in-situ fracturing or cleaving, and (4) in-situ maching or scraping. Since many methods call for repetition of processing steps until the surface is clean, the need for in-situ element specific analysis of the surface is not eliminated by following the recipes given here.

Specifically excluded from this review are preparation of clean, elemental surfaces by in-situ deposition of films, by laser irradiation, and by field desorption methods. For some elements, in-situ deposition of a film may be the only known method of preparing a large, clean surface (i.e., ~ 1 cm^2); this fact is noted in the table of recommended surface-cleaning procedures. Laser irradiation has limited applications and has not yet been fully established. Field desorption cleaning techniques are not reviewed because the sample size, which is limited to ≤ 5000 Å diameter by the requirements of FEM and FIM, is not large enough to be generally useful.

For the convenience of the reader the elements are arranged in alphabetical order in the text, in the table and in the reference pages. The sources reviewed and quoted are generally in English from easily accessible books and journals. These sources were found by searching for papers published prior to early 1981 using both key-worded computerized systems and journal indices.

2. Review of surface cleaning procedures

Procedures used to generate atomically clean surfaces for surface-related experiments have been reviewed and evaluated. Wherever possible only cleaning methods documented by an element-specific, surface-analytical technique were included in the review. The elements are discussed in alphabetical order with details and references for individual crystallographic planes presented wherever appropriate. Both the crystalline structure type and the melting point T_m are listed parenthetically after the elemental name and abbreviation.

Actinium, Ac (cub, $T_m = 1323$ K)

We found no surface studies on bulk or thin-film actinium.

Aluminum, Al (fcc, $T_m = 933$ K)

Polycrystalline and single crystalline rods with the orientations (100), (110), and (111) are sold commercially with 99.9995% purity; thus, the major cleaning task is the removal of the thin oxide layer which formed on the surface during exposure to air and during electropolishing.

Polycrystalline surface [Al-1 to Al-15]. Polycrystalline surfaces of aluminum have been investigated ever since surface science tools became available. Two different cleaning procedures have been used: (1) heating the sample to 873 K, and (2) repeated cycles of ion bombardment and annealing. For the first procedure, Auger peaks of oxidized aluminum faded away and eventually disappeared after several hours of annealing at 873 K under UHV conditions, such that the ratio of the oxygen peak (510 eV) to the Al peak (68 eV) was less than 0.001 [Al-12]. In the second procedure, the sample was bombarded with noble gas ions at room temperature (5 keV, 78 μA/cm^2) [Al-10], and thereafter annealed at various temperatures (e.g., at 550 and > 723 K). The majority of studies included the ion-bombardment and annealing cycle. The sputtering time necessary to remove the initial oxide layer was approximately 10 h; for recleaning, sputtering times of the order of 1/2 h were cited.

(100) Surface [Al-15 to Al-24, Al-26 to Al-29]. Many studies [Al-16, Al-18, Al-19, Al-21, Al-23] dealt exclusively with the (100) surface, and the other reports cited above treated the (100) surface in conjunction with other low-index planes. Essentially, the same procedures used for cleaning the polycrystalline surface were shown to be successful for cleaning the (100) surface. Some authors used heating to 873 K without sputtering as a cleaning step [Al-26, Al-28]; all others used ion (neon [Al-20], xenon [Al-19]) bombardment and annealing cycles. Most investigators used ion bombardment at room temperature and annealed at 723 K; but some investigators raised the target temperatures during ion bombardment (473 K [Al-29], 673 K [Al-23]) and others preferred an annealing temperature of 805 K [Al-16, Al-19].

(110) Surface [Al-15, Al-17, Al-20, Al-22 to Al-30]. One research report [Al-30] dealt exclusively with the (110) surface; the others treated the (110) surface in conjunction with the other low index planes. The (110) surface was subject to faceting and produced, in general, less satisfactory LEED patterns than either the (100) or the (111) surface [Al-30, Al-27]. The cleaning procedures used were the same as those described above for the (100) surface.

(111) Surface [Al-15, Al-17, Al-20, Al-22, Al-24 to Al-34]. Several groups studied the (111) surface exclusively [Al-25, Al-31 to Al-34]. Other researchers dealt with the (111) surface in conjunction with other low-index planes [Al-15, Al-17 to Al-24, Al-26, Al-28, Al-29]. The UHV cleaning procedures were the same as reported for the (100) and (110) surface.

(421) Surface [Al-35]. A clean surface was obtained by a series of argon-ion bombardment and annealing (823 and then 893 K) cycles.

Americium, Am (hex, T_m = 1267 K)

We found no surface studies on bulk or thin-film americium.

Antimony, Sb (rhdr, T_m = 1267 K)

Although no surface studies on polycrystalline antimony were found, cleaning procedures for three single crystal planes consisted of cleaving and/or sputter–anneal treatments.

(0001) Surface [Sb-1, Sb-2]. Antimony has been cleaved to yield (0001) surfaces in air and dry nitrogen. These surfaces were immediately transferred into UHV systems and produced a well-defined LEED pattern [Sb-1, Sb-2]. The small oxygen contamination was removed by argon-ion sputtering (200 - 300 eV, few $\mu A/cm^2$, 1 min). This surface plane is relatively inert to residual gases since no oxygen or carbon contamination was observed over the 6–10 h period required for XPS measurements [Sb-1]. Anneals for a few hours at ~520 K have been shown to improve surface structure as indicated by improved LEED patterns.

$(01\bar{1}2)$ and $(11\bar{2}0)$ surfaces [Sb-2]. In LEED-only studies the $(01\bar{1}2)$ and $(11\bar{2}0)$ surfaces were given the same sputter–anneal treatment used for the (0001) plane to improve the LEED pattern; however, inclined facets in the $(01\bar{1}2)$ plane caused maverick LEED beams whereas this surface had a well-defined LEED pattern before any treatment. The $(11\bar{2}0)$ surface yielded a LEED pattern, which indicated a reconstructed surface, only after the treatment.

Arsenic, As (rhdr, T_m = 1090 K)

Arsenic is not a UHV-compatible material because of its high vapor pressure (10^{-2} Pa at 483 K). However, this high vapor pressure proved useful for preparation of an atomically clean (0001) crystal plane for LEED, AES, and XPS investigations. Impurities of concern were carbon and oxygen. An air-cleaved arsenic crystal was heated to 493 K in vacuum to evaporate the oxide and all surface impurities. The sample was surrounded by cold surfaces to condense the arsenic vapor and prevent contamination of the UHV system. The amount of carbon and oxygen on cleaned arsenic surfaces did not increase with time of exposure in the UHV system [As-1, As-2].

Barium, Ba (bcc, $T_m = 998$ K)

We found no surface studies on bulk barium; however, evaporated films have been investigated.

Beryllium, Be (hcp, $T_m = 1551$ K)

Removal of silicon is the most difficult part of the cleaning procedure; the difficulty increases with increasing concentration of silicon in the bulk [Be-1]. Cleaning was usually accomplished by repeated cycles of argon-ion sputtering and annealing.

Polycrystalline surface [Be-1, Be-2]. For beryllium containing less than 1 ppm silicon, several cycles of argon-ion sputtering and annealing at less than 900 K were sufficient to produce a clean surface; however, long-term annealing at 1240 K resulted in resegregation of silicon on this surface [Be-1]. Beryllium with 60 and 600 ppm silicon was also cleaned by the above sputter–anneal procedure, but lower temperatures were sufficient to bring silicon to the surface. A temperature of 1070 K was high enough to create a silicon-contaminated surface for a bulk impurity level of 600 ppm silicon. Evidence exists for the segregation of the silicon in a layer on top of the beryllium [Be-2].

(0001) Surface [Be-3, Be-4]. Cycles of argon-ion bombardment (0.6 or 3 keV, few μA/cm^2, 1–2 h, 300 or 1070 K) and annealing (1020–1070 K, ~ 1 h) have produced clean surfaces.

Bismuth, Bi (rhdr, $T_m = 544$ K)

A clean (0001) crystal face was prepared for LEED, AES and ELS studies by removing carbon, oxygen and chlorine with argon-ion bombardment (150–175 eV, 5 μA/cm, 6 h) and a UHV anneal (510 K) [Bi-1]. Sharp LEED patterns of the (0112) and (1120) planes were produced by a similar argon-ion bombardment (300 eV, 1–2 μA/cm^2) and UHV annealing (523 K) procedure; however, surface purity was not verified by an element-specific analytical technique [Bi-2].

Boron, B (α rhdr; β rhdr > 1470 K, $T_m = 2350$–2500 K)

Boron is commercially available in both polycrystalline and single-crystalline forms. Although the main crystal structure is rhombohedral, other crystal structures have been reported to exist. The exact temperatures for the various phase transformations are not well known. For the few surface studies reviewed, carbon and nitrogen impurities were the most difficult to remove.

Polycrystalline surfce [B-1]. A polycrystalline sample produced by fusing 1 μm particles in a solar furnace showed, after various alternate cycles of heat treatment and ion bombardment, an Auger spectrum free of chlorine, sulfur and phosphorus but containing small oxygen and carbon peaks.

(001) Surface and surfaces parallel to the C axis [B-2]. Crystals were heated indirectly by radiation from a hot tungsten filament mounted behind the sample or by a quartz-iodine lamp. During the heat treatment (923–1023 K, 5 min) the vacuum was $< 1 \times 10^{-5}$ Pa. Traces of carbon, oxygen and nitrogen were still visible in the Auger spectrum even after a heat treatment at 1123 K. No LEED pattern could be seen.

(111) Surface [B-3]. Heat treatment at high temperature was used to obtain a clean ordered surface. After heating to 1473 K, large amounts of oxygen and carbon still remained at the surface, and the LEED pattern showed a completely disordered surface structure. Oxygen completely disappeared after heating for some minutes at 1573 K. To remove carbon and to obtain a good LEED pattern, temperatures higher than 1673 K were found to be necessary. Heating to 1823 K did not seem to improve appreciably the surface condition; the standard treatment was to heat at 1723 K for 1 min. Nitrogen could be removed by argon-ion bombardment but reappeared after the high temperature anneal necessary to obtain a regular surface structure. The minimum ratio between nitrogen and boron Auger peaks was about 1:50.

Cadmium, Cd (hcp, $T_m = 594$ K)

No bake-out of the vacuum system containing cadmium is possible due to the high vapor pressure (10^{-9} Pa at 293 K) and the low melting point (594 K) of cadmium. In common with other soft metals, cadmium was easily cleaned.

Polycrystalline surface [Cd-1 to Cd-4]. Specimens cut from 99.999% cadmium ingots were mechanically scraped with an oxide-free tungsten carbide blade in a preparation chamber at $\sim 10^{-6}$ Pa and transferred to the measuring chamber without breaking the vacuum. Photoelectron spectra of scraped specimens showed no trace of the oxygen 1s line and only slight traces of the carbon 1s line [Cd-1]. Sputter cleaning with argon ions at 3 keV has also produced surfaces free of contaminants [Cd-2]. Scraping or micromilling with a rotating diamond is preferred whenever it is important to keep the vacuum system free of cadmium contamination [Cd-3, Cd-4].

(0001) Surface [Cd-5]. After ion bombardment (700 eV, 3 μA/cm^2, 295 K, 24 h) the Auger spectrum showed no traces of sulfur, nitrogen or oxygen. A small carbon peak, if present, could have been concealed by the metal peak at

285 eV. The clean surface LEED pattern was well ordered and had a high background intensity, as expected from metal with a low melting temperature.

Calcium, Ca (fcc, $T_m = 1112$ K)

We found no surface studies on bulk calcium.

Carbon, C

Bulk carbon exists in four forms: amorphous, glassy, graphitic and diamond. Consequently, the review is divided into four parts.

Amorphous carbon
Amorphous carbon surfaces have been produced by evaporating high purity carbon under UHV conditions or by ion bombardment of graphitic carbon surfaces.

Glassy carbon [C-1]
A contamination-free surface of glassy carbon has been produced by fracturing a disk-shaped ingot under dry nitrogen in a glove bag and inserting it directly into UHV spectrometer without exposure to the atmosphere.

Graphitic carbon (hex, $T_m = \sim 3820$ K)
Graphite surfaces are very sensitive to ion bombardment. A dose of 3×10^{15} argon ions/cm^2 (300 eV) led to a completely amorphous surface [C-2]. A temperature of 1773 K was necessary to anneal the radiation damage.

(0001) Surface [C-2 to C-10]. Many Auger electron spectroscopy and LEED experiments have shown that the basal plane (0001) is an inert surface. At least one group [C-10] has shown by LEED that there is no chemical adsorption of H_2O, CO, oxygen, iodine or bromine on the (0001) surface or graphite in UHV at 300 K. An air-cleaved (0001) surface inserted quickly into a vacuum system and annealed (723 K, 5 h) produced a clean surface as judged by Auger spectroscopy [C-4]. However, neither Auger spectroscopy, nor LEED, nor the growth pattern of inert gases condensed on the surface was established as the most sensitive test of surface cleanliness. Use of the gold decoration technique under UHV conditions [C-9] demonstrated that surfaces, undistinguishable by these conventional techniques of surface analysis provided different reaction site densities. Furthermore, the reaction site density depended on the number of charged particles (from ion gauges, ion pumps, etc.) that impacted onto the surface. The gold decoration technique does confirm the observation made earlier by AES: an air-cleaned (0001) surface is slightly contaminated, and the contamination can be removed by annealing (723 K, 5 h). This low annealing temperature reflects the weakness of the adsorbate–substrate interaction. In

contrast, a UHV-cleaved surface exposed for 24 h to a UHV vacuum system having sources of free charges must be annealed at 1273 K; unfortunately, the resulting surface was still not perfectly clean. One must conclude that a clean (0001) graphite surface can be maintained only in a charge-free UHV system. If the sample is cleaved under a nitrogen atmosphere and placed within seconds under vacuum, contamination of the cleaved surface is avoided almost completely.

(10$\bar{1}$0) and (11$\bar{2}$0) surfaces [C-11]. The (10$\bar{1}$0) and (11$\bar{2}$0) surfaces fractured in air and analyzed within minutes by X-ray photoelectron spectroscopy showed, in contrast to the (0001) planes, considerable oxygen contamination.

Diamond (cub-dia) [C-12 to C-15]

The (100), (110) and (111) surfaces of diamond have been studied. Producing a crystalline surface for this allotropic form of carbon was more difficult than cleaning it. All authors agreed that ion sputtering was useless, because the damage generated could not be annealed out without graphitizing the surface. Therefore, all authors used an annealing procedure for cleaning. For high purity diamond, annealing in UHV ($< 10^{-8}$ Pa) between 1173 and 1573 K produced clean surfaces.

Cerium, Ce (fcc, $T_m = 1072$ K)

Polycrystalline samples of 99.9%-pure cerium have been cleaned by cycles of argon-ion sputtering (1 keV, 10 μA/cm^2) and low temperature annealing (temperature not specified). The main residual contaminants on the surface were determined by soft X-ray appearance spectroscopy. After four cleaning cycles, all the impurities which were observed between excitation potentials of 50 and 1500 V, except oxygen and zirconium, had been reduced below the sensitivity of the instrument. Additional cycles did not further reduce the presence of these two impurities [Ce-1]. A more recent study also made use of argon-ion sputtering (900 eV, 11 μA) to produce clean surfaces on cerium (99.9% pure) as verified by XPS [Ce-2].

Chromium, Cr (bcc, i$T_m = 2130$ K)

The major impurities found to segregate onto chromium surfaces (~ 99.99% pure) during in-situ cleaning processes were sulfur, carbon, nitrogen, oxygen and chlorine. Oxygen was somewhat difficult to detect by AES because the oxygen (KLL) peak at ~ 512 eV is located between two major chromium peaks with almost equal sensitivity factors, i.e., the LMM peak at ~ 484 eV and the LMV peak at ~ 529 eV. However, the Cr(MMM) transitions at ~ 37 and ~ 41 eV were very sensitive to minute traces of contamination [Cr-1]. In general, cleaning of chromium was accomplished by a combination of argon-ion bombardment and annealing as detailed below.

Polycrystalline surface. The only studies we located were on evaporated films.

(100) Surface [Cr-1 to Cr-4]. Iterations of argon-ion sputtering (~ 500 eV. 3–5 μA, 1000 K [Cr-1] or 300 K [Cr-2]) followed by annealing in UHV (900 K [Cr-1] to 1170 K [Cr-2]) led in one case to a clean, unreconstructed surface with a (1 × 1) LEED pattern [Cr-1] and in another case to a clean. reconstructed surface exhibiting a c(2 × 2) LEED pattern [Cr-2]. A c(2 × 2) reconstructed surface was also observed when the following cleaning routine was used [Cr-3]: Outgas sample (670 K, several h), sputter with argon ions (1.5 keV), heat in UHV (≤ 1170 K), cool, and repeat the sputtering until the near surface region is depleted of sulfur. To deplete nitrogen in the near surface region, heat in UHV (~ 970 K, several days) and then perform a final anneal (670 K, 15 min). An argon-ion bombardment (450 eV, 4 μA/cm^2) and annealing cycle performed for an unspecified number of times, produced a (1 × 1) LEED pattern that appears to have been stabilized by sulfur [Cr-4]. Temperatures between 870 and 1070 K drove carbon and sulfur from the bulk to the surface.

(110) Surface [Cr-1, Cr-5, Cr-6]. The (110) surface was cleaned by argon-ion bombardment (600 eV, 3–5 μA/cm^2, 1000 K) and annealing (900 K) while removing the argon gas used for cleaning [Cr-1]. Some authors used variations on the basic sputter (500 eV, 25 μA, 300 K, 30 min [Cr-5]; 2000 to 500 eV [Cr-6] and annealing (670 K, 15 min [Cr-5]; 870 K [Cr-6]) cycle. Clean (110) surfaces with (1 × 1) LEED patterns were produced in all cases.

(111) Surface [Cr-7]. Clean, unreconstructed (111) surfaces have been produced by removing the initial oxide coating using argon-ion bombardment (starting at 8 keV, 60 μ/cm^2 and gradually lowering the voltage and beam current to 2 keV, 5 μA/cm^2). This was followed by alternately heating the sample in UHV (1170 K) and argon-ion sputtering (2 keV, 5 μA/cm^2) until heating (770 K, 15 min) did not result in further impurity segregation to the surface.

Cobalt, Co (hcp; fcc > 693 K, T_m = 1768 K)

Cobalt is difficult to clean because it undergoes a hcp-to-fcc phase transition at 693 K. This means that cleaning must be done below this temperature for single-crystalline specimens. However, several authors reported obtaining sharp LEED patterns after brief anneals above the transition temperature [Co-1 to Co-5]. The most common impurities found on the surfaces of cobalt samples (99.999% pure) were sulfur, oxygen, chlorine, carbon and nitrogen. Carbon was the most persistent and difficult to remove. Sputtering with low-energy argon ions was reported to be effective in removing oxygen and chlorine but not carbon [Co-1, Co-4]. Similar sputtering with neon ions effectively removed carbon and oxygen [Co-1, Co-2, Co-4]. The same result was achieved with a 1 keV krypton-ion beam [Co-3].

Polycrystalline surface [Co-1, Co-2, Co-6]. Polycrystalline specimens could be cleaned by heating in oxygen (900 K, 1.3×10^{-4} Pa), bombarding with neon ions (530 eV, 1 μA/cm^2, 600 K) and then annealing (1000 K, 15 h) [Co-1]. LEED studies indicated that 90% of a surface cleaned in this way had recrystallized into ~ 1 mm crystals with surfaces oriented parallel to the (0001) plane, while the remainder had $(10\bar{1}2)$ surface orientations [Co-1, Co-2]. Sputtering with argon ions at 3 keV also produced a clean surface [Co-6].

(0001) Surface [Co-1 to Co-3, Co-7 to Co-9]. Sputtering with neon ions (530 eV, 1 μA/cm^2) at elevated temperatures (600 K) followed by cyclical annealing between 650 and 800 K produced clean (0001) surfaces that yielded sharp LEED patterns [Co-1, Co-2]. Krypton bombardment (1 keV, 300 μA/cm^2, 300 K, 15 h) with intermittent annealing at 900 K in UHV also proved to be effective in producing a clean, well-ordered surface [Co-3]. Clean (0001) surfaces have also been obtained by many iterations of inert-gas-ion bombardment (500 eV, ~ 200 μA/cm^2) and annealing (620 K) for a total of 15 h of ion bombardment and 25 h of annealing; this was followed by 20 h of annealing in UHV at 620 K to improve the ordering of the clean surface [Co-7, Co-8]. One author [Co-9] used a multistep procedure that included (a) removal of oxygen with argon-ion bombardment or heating in hydrogen, and (b) removal of carbon by heating in ~ 3×10^{-6} Pa oxygen at 590 K. At pressures greater than 2×10^{-3} Pa, oxygen was found to remain on the surface [Co-9].

$(10\bar{1}0)$ Surface [Co-3]. Sputtering with krypton (1 keV, 300 μA/cm^2, ~ 15 h, 300 K) coupled with intermittent anneals at 900 K was an effective method for cleaning this crystal face.

$(10\bar{1}2)$ Surface [Co-4]. A cycle of neon-ion bombardment (300 eV, 600 K) followed by annealing at 700 K was continued over a period of several weeks to eventually produce a carbon-free surface.

(100) Surface [Co-10, Co-11]. One set of authors reported on the cleaning of a cobalt sample in the fcc form at room temperature; therefore heating past the transition temperature was not a concern. Furthermore, the crystal did not return to the hcp form if it was cooled quickly; but it was quite difficult to remove carbon from the surface. The reaction of surface carbon with up to ~ 10^{-4} Pa oxygen at temperatures up to 1020 K was so slow that carbon actually enriched on the surface. It was found that argon-ion bombardment (150–180 eV) and annealing at 420 K would produce a clean, well-ordered surface.

Copper, Cu (fcc, $T_m = 1356$ K)

The impurities most commonly observed on polycrystalline [Cu-1] and single-crystalline [Cu-2, Cu-3] copper surfaces were carbon, oxygen and sulfur. Chlorine and nitrogen were also detected on the (111) crystal plane [Cu-3]. Most investigators used repeated cycles of low energy ($\leqslant$ 600 eV) argon-ion bombardment and annealing in UHV (600–1000 K) to clean single crystal copper surfaces.

Polycrystalline surface [Cu-1]. Low-temperature hydrogen-ion bombardment was used to prepare a clean polycrystalline surface wthout a high temperature annealing step. Omission of the annealing step prevented segregation of the bulk impurities to the surface. The chemical reactivity of the hydrogen ions enhanced the cleaning process, and the low mass of the ions contributed to minimized surface damage from ion bombardment. AES revealed that all carbon and sulfur had been removed after only 1 h of ion bombardment (800 eV, 10μA/cm^2). A trace of oxygen remained on the surface even after 16 h of ion bombardment.

(100) Surface [Cu-2, Cu-4 to Cu-9]. Clean (100) crystal planes were prepared by several cycles of low energy argon-ion bombardment (500–550 eV, 1 μA/cm^2) and annealing in UHV at 700 to 850 K [Cu-4 to Cu-6]. Clean (100), (110) and (111) crystal planes were prepared by cycles of 3 keV argon-ion bombardment and 1000 K UHV anneals [Cu-7]. Other investigators also reported preparing clean (100) crystal planes with argon-ion bombardment and UHV annealing [Cu-8, Cu-9]. Another cleaning procedure for the (100) surface consisted of heating the crystal in oxygen (800 K, 1.3 × 10^2 Pa) to remove carbon and sulfur followed by heating in atomic hydrogen to remove the surface oxide [Cu-2].

(110) Surfaces [Cu-10 to Cu-13]. Cycles of argon-ion bombardment (600 eV, 5 μA/cm^2) and UHV annealing at 723 K were used to prepare clean (110) surfaces [Cu-10]. Other investigators also reported using cycles of argon-ion bombardment and annealing (650–1023 K) but failed to give any other details of their procedures [Cu-11 to Cu-13].

(111) Surface [Cu-3, Cu-4, Cu-7, Cu-9]. Sulfur, chlorine, carbon, nitrogen and oxygen impurities were detected with AES on a (111) crystal surface [Cu-3]. These impurities could be removed by argon-ion bombardment (100–300 eV, 100 μA/cm^2, 20 min) and annealing (573–723 K) in UHV. However, the carbon and sulfur contamination on the surface increased by diffusion from the bulk when the sample was heated between 773 and 1023 K in UHV. The amounts of carbon and sulfur on the surface decreased when the sample was heated above 1023 K.

(311) Surface [Cu-14]. A (311) plane was cleaned with cycles of xenon-ion bombardment (300 eV, 1 μA/cm^2, 2 h) and annealing (650 K, UHV, 1 h). A total of 16 h of ion bombardment was required to remove all traces of carbon and oxygen from the surface.

Dysprosium, Dy (hex, T_m = 1685 K)

We found no surface studies on bulk dysprosium. Dysprosium has been evaporated to form films.

Erbium, Er (hcp, T_m = 1802 K)

We found no surface studies on bulk erbium. Erbium has been evaporated to form films.

Europium, Eu (bcc, $T_m = 1095$ K)

We found no surface studies or bulk europium. Europium has been evaporated to form films.

Gadolinium, Gd (hcp, $T_m = 1586$ K)

Polycrystalline samples of gadolinium were found by AES to contain "small amounts" of carbon and oxygen after cleaning by repeated argon-ion bombardment and low temperature annealing (conditions not specified) [Gd-1].

Gallium, Ga (orth, $T_m = 303$ K)

Gallium is a liquid at slightly above room temperature; however, it is a UHV-compatible material with a vapor pressure of 1.3×10^{-7} PA at 840 K. The only surface study on gallium found in the literature was one using AES with imaging capability to study the surface of a liquid gallium drop held in a pyrolytic carbon cup [Ga-1]. When the gallium was heated to 600 K its surface became almost free of carbon and oxygen. Surface precipitates (mainly carbon, gallium oxide and occassionally sulfur) were first observed when the gallium was cooled to 390 K. These precipitates slid down the drop and accumulated near the edge of the carbon cup. The thin oxide and carbon overlayer on the liquid gallium could be cleaned by ion bombardment (3 keV, 20 $\mu A/cm^2$). Unexpectedly, impurity precipitates originating outside the ion impact area migrated into the impact region. Thus, an area larger than the actual ion impact area was cleaned by ion bombardment. The impurities collected near the carbon cup were redistributed by heating to 600 K, then cooling to 300 K. The entire liquid gallium surface was cleaned by a "few" sequences of ion bombardment followed by temperature cycling.

Germanium, Ge (cub-dia, $T_m = 1210$ K)

Few surface studies on germanium were found that included both an element-specific technique for surface analysis and a cleaning procedure. Single crystal surfaces have been prepared using one of three possible procedures: ion bombardment and annealing, cleaving in UHV, or growth and evaporation of a sulfide layer.

Polycrystalline surface. No surface studies were found on polycrystalline germanium.

(100) Surface [Ge-1]. Neon-ion bombardment and a short anneal at 1073 K produced clean (100) surfaces.

(110) Surface [Ge-2, Ge-3]. A clean (110) crystal face was prepared by evaporating a previously grown sulfide film in the UHV system at 623 K. However, there was no documentation for the purity of this surface with an element-specific technique. This method allowed preparation of a clean surface at a lower temperature than was possible by argon-ion etching and annealing [Ge-3].

(111) Surface [Ge-4, Ge-5]. A clean (111) crystal face has been prepared by cleaving in vacuum.

Gold, Au (fcc, $T_m = 1337$ K)

High purity gold (99.999%) is commercially available in polycrystalline and single-crystalline forms. The major contaminants important in preparing clean surfaces are carbon, sulfur and calcium.

Polycrystalline surface [Au-1 to Au-5]. An effective cleaning cycle for calcium-free material consisted of a series of heatings at 1000 K: 6 h in vacuum of less than 6×10^{-8} Pa, then 24 h in 6×10^{-3} Pa oxygen, and finally 8 h in 6×10^{-3} Pa hydrogen [Au-2]. If the metal contained calcium, sputter cleaning was essential to obtain a clean surface. One recommendation was that the sample be subjected to alternate cycles of heating, exposure to oxygen and ion bombardment in various combinations over a long period of time [Au-3]. The purpose of this treatment was to purify the gold sample by continuusly segregating calcium at the surface where it could be removed by ion bombardment. The temperature of the target during exposure to oxygen at 5×10^{-5} Pa was 873 K. Cycles of heating under ultra-high vacuum ($T > 1073$ K), ion bombardment and annealing at 873 K have also been effective [Au-4].

(100) Surface [Au-6 to Au-10]. All investigators produced clean surfaces by repeated cycles of argon- or xenon-ion sputtering [Au-5, Au-6] and annealing (500–1000 K). Annealing temperatures close to the melting point produced irreversible high-temperature surface structures [Au-6]. Heating in oxygen has also been included in the cleaning process [Au-10].

(110) Surface [Au-11 to Au-15]. Clean surfaces were produced by repeated cycles of ion bombardment and annealing at temperatures from 523 K [Au-11] to 800 K [Au-12]. A two-stage process has been used to minimize the annealing temperature and calcium segregation [Au-15]. In the first stage, the crystal was bombarded by argon ions (0.5–3 eV) at a variety of angles and annealed at temperatures up to 1073 K. After the contamination was reduced, low-energy neon ions (~ 300 eV) were used in the second stage to bombard the sample at oblique angles of incidence; this was followed by annealing at less than 573 K, which proved to be sufficient to produce a sharp (1 × 2) LEED pattern.

(111) Surface and its vicinals [Au-15 to Au-18]. Carbon was successfully removed by exposing the surface to 10^{-5} Pa oxygen at 973 K for 10 h; all other impurities were eliminated by argon-ion sputtering at 773 K [Au-17] or by

successive cycles of argon-ion bombardment at room temperature and annealing at 973 K [Au-16].

Hafnium, Hf (hcp, $T_m = 2500$ K)

The major contaminant found in hafnium foil samples was zirconium (~ 3%) that was not effectively removed in the refining process [Hf-1]. Hafnium is found in zirconium-bearing minerals; and, because of their nearly identical chemistries, these two elements are extremely difficult to separate [Hf-2]. Zirconium tended to segregate on the surface at 1070 K, and became the major surface species in ≤ 30 s. Other bulk impurites that migrated to the surface during the heating operation were sulfur, carbon, chlorine and oxygen [Hf-3]. Sulfur segregation increased at 1370 K; the sulfur content of the surface region decreased but did not disappear at 1570 K [Hf-4]. Most of the contaminants listed above could be removed by argon-ion bombardment [Hf-1, Hf-3, Hf-4]. This suggests that the most effective cleaning method would consist of a prolonged period of sputtering at 1070–1370 K [Hf-4]. To estimate this period assume that ~ 1 monolayer of zirconium migrates to the surface in 30 s at 1070–1370 K and that sputtering completely removes this layer as it forms; then it would take about 20 days of continuous sputtering at temperature to remove one-half of the zirconium impurity from a 1 mm thick sample (the other half of the impurities are assumed to segregate near the opposite surface).

Holmium, Ho (hcp, $T_m = 1747$ K)

Polycrystalline samples of high purity holmium have been cleaned by argon-ion sputteing and annealed under an X-ray beam. Soft X-ray appearance potential measurements showed a weak oxygen peak that probably resulted from the reaction of the metal with residual gases [Ho-1].

Indium, In (tetr, $T_m = 430$ K)

Only polycrystalline indium has been subjected to surface studies. Argon-ion bombardment (0.5–5 keV) quickly produced a clean surface, either in the molten or in the solid state. This does not imply that the material is clean to any depth or will stay clean. On solidification from the molten state, sulfur, carbon and oxygen reappeared on the surface. Since the melting point of indium is low, it is reasonable to assume that these impurities will diffuse in time at room temperature from the bulk to the surface. It is therefore recommended that a clean surface on solid indium be prepared by many repeated cycles of sputtering at a temperature above the melting point and solidification [In-1 to In-5]. In-situ scraping has also been used to create clean surfaces [In-6, In-7].

Iridium, Ir (fcc, $T_m = 2683$ K)

Cleaning of iridium was routinely accomplished by heating in oxygen. The impurities most often encountered on bulk iridium surfaces during the cleaning process were carbon, oxygen, nitrogen, sulfur and phosphorus. Studies on clean single crystals were complicated by reconstruction in the (100) and (110) planes.

Polycrystalline surface [Ir-1]. A polycrystalline foil was cleaned by repeated heating (1500 K) in oxygen (1.3×10^{-4} Pa, several h) and then flashing in UHV (1700 K, ~ 20 min) until no impurities could be detected by AES.

(100) Surface

A. (5 × 1) reconstructed surface [Ir-2 to Ir-11]. A (5 × 1) LEED pattern corresponding to a distorted hexagonal overlayer on the normal (100) surface reuslted when the (100) surface was subjected to alternating heating cycles in oxygen (1400–1500 K, 7×10^{-6} to 1.3×10^{-5} Pa, 5–10 min) and in UHV (1500 K, ~ 1 min) until no impurities were detectable by AES [Ir-2 to Ir-8]. In most cases, the initial contamination on the surface was removed by argon-ion bombardment prior to heating in oxygen [Ir-2 to Ir-7]. Variations on this basic procedure included lower temperature (1200–1300 K) oxygen cycles [Ir-9] and higher temperature anneals (2100 K, > 1 h) [Ir-10]. One author also repeatedly obtained a clean (5 × 1) surface by: (a) heating in UHV (~ 1670 K), (b) argon-ion bombardment (800 eV, ~ 100 μA/cm²), and (c) annealing (~ 670 K) in UHV [Ir-11].

B. (1 × 1) unreconstructed surface [Ir-2 to Ir-6]. It has been found that one can generate (100) surfaces that reflect the underlying bulk structure by chemical means. This involved adding 10–20 L of oxygen at ~ 7×10^{-6} Pa to the previously prepared (5 × 1) surface at 475 K [Ir-2] to 670–870 K [Ir-3 to Ir-6]. It was important that the sample be heated to ~ 660 K at some point during [Ir-3 to Ir-6] or after [Ir-2] the addition of oxygen. The foregoing operation converted the clean (5 × 1) surface to an oxide. At this point, the oxygen was removed by the addition of hydrogen (400–700 K, ~ 10^{-5} Pa [Ir-2]; ~ 30 L, ≤ 400 K [Ir-3]) or CO (~ 300 L, ≤ 400 K) [Ir-3 to Ir-6]. Gradual heating to less than 800 K removed adsorbed gases and yielded a clean, unreconstructed (1 × 1) surface [Ir-3 to Ir-6]. At temperatures higher than 800 K, the (1 × 1) structure converted irreversibly to the (5 × 1) structure [Ir-2].

(110) Surface [Ir-12 to Ir-20]. The (110) surface is also subject to reconstruction as evidenced by the (2 × 1) LEED pattern observed for this crystal face. The general procedure for cleaning this surface was as follows: argon-ion bombard to remove initial contaminants, then heat in oxygen (~ 800 K, 7×10^{-6} to 1.3×10^{-5} Pa, several min) and anneal (~ 1600 K) in UHV; repeat the heating and annealing cycle until the surface is atomically clean. Variations on this procedure were to hold the sample at 1470 K for several days to aid in calcium removal [Ir-19] or to anneal at lower temperatures (970 K) [Ir-20].

(111) Surface [Ir-9, Ir-16, Ir-21 to Ir-30]. No reconstruction has been observed on this face of iridium. The most commonly used cleaning method was to remove initial contaminants by argon-ion bombardment, then to heat in oxygen (800–1100 K, 7×10^{-6} to 1.3×10^{-4} Pa) and to anneal in UHV (1200–1600 K) [Ir-9, Ir-16, Ir-21 to Ir-27]. One variation on this procedure was to conduct the initial cleaning by first heating the sample in oxygen (1100 K, $\sim 10^{-6}$ Pa), and then bombarding it with xenon ions (600 eV, ~ 2 μA/cm^2, 10–30 min) before proceeding with the heating cycle described above [Ir-28, Ir-29]. It was possible to obtain a clean surface by cycles of argon-ion bombardment (500 eV, ~ 200 μA/cm^2, 1 h) with intermittent flashing to 1770 K and annealing [Ir-30].

(755) Surface [Ir-25, Ir-26]. This surface corresponds to a stepped surface consisting of 6-atom wide (111) terraces separated by 1-atom high (100) steps; this configuration is stable at temperatures up to 1470 K. It has been prepared by heating in oxygen (770–870 K, 7×10^{-5} Pa, several min) followed by flashing in UHV to 1470 K [Ir-25, Ir-26].

Iron, Fe (bcc, fcc > 1183 K; bcc > 1663 K, $T_m = 1808$ K)

Iron is difficult to clean for two reasons: (1) the bcc to fcc phase transition at ~ 1183 K necessitates that all cleaning be done below this temperature; (2) most commercially available samples have high concentrations of non-metallic impurities, particularly carbon and sulfur. The nominal purity of single crystal samples ranged from 99.92% to 99.995%, while most polycrystalline samples were nominally 99.999% pure. The use of some form of pretreatment in hydrogen to remove those impurities that form volatile hydrides increased as a function of the openness of the iron crystal face under consideration (i.e. increased use going from (100) to (111) to (110) to poly; all investigators pretreated poly with hydrogen). The major contaminants found on single crystals from a variety of sources were carbon, sulfur, oxygen, nitrogen, phosphorus and chlorine (ordered in decreasing amounts). In general, the cleaning of iron surfaces was accomplished by removing gross contamination and oxides by gentle sputtering with argon ions, then depleting carbon, sulfur and phosphorus in the bulk by repeated cycles of argon-ion sputtering and annealing in UHV. Recleaning between exposures was accomplished by argon-ion sputtering and a flash anneal.

Polycrystalline surface [Fe-1 to Fe-3]. Polycrystalline iron samples have been cleaned by argon-ion sputer–anneal cycles [Fe-1, Fe-2] and by the combination of sputter–anneal cycles with oxidation–reduction cycles [Fe-3]. Few details were listed for these processes but they can be inferred from the discussions below.

(100) Surface [Fe-4 to Fe-16]. The most frequently used method for cleaning this crystal face consisted of cycles of argon-ion sputtering (~ 500 eV, ~ 20 μA/cm^2, 15–30 min) and annealing in UHV (700–800 K, 1–60 min) [Fe-4 to

Fe-15]. Sputtering was carried out at both elevated temperatures (600–700 K) [Fe-4 to Fe-7] and at ambient temperature [Fe-8 to Fe-15]. This basic cycle has been combined with additional chemical cleaning steps by heating the sample to ~770 K and exposing it to oxygen (0.5–10 L) followed by oxygen "titration" with acetylene [Fe-13, Fe-14] or by heating in either oxygen or water vapor (770–870 K, ~10^{-4} Pa) [Fe-15]. Another method included cycles of argon-ion sputtering at 1070 K and annealing at 670 K for 12 h periods for one week and, finally, flashing to 970 K [Fe-16]. Sulfur has been oberved to segregate to the Fe(100) face between 500 K [Fe-6, Fe-12, Fe-13] and 700 K [Fe-4].

(110) Surface [Fe-4, Fe-11, Fe-17 to Fe-26]. The preferred method for cleaning the (110) surface consisted of the sputter–anneal cycle outlined for the (100) plane [Fe-4, Fe-17 to Fe-21], although higher ion energies (1–4 keV) were sometimes used for the (110) plane [Fe-11, Fe-22, Fe-23]. In some cases sputtering was carried out at elevated temperatures (920 K, ~1 h) [Fe-4, Fe-20, Fe-22]. Variations of the preferred method were to combine the basic treatment outlined above with a hydrogen treatment (650 K, 10^5 Pa) [Fe-24] or to include an oxygen treatment (970 K, 7×10^{-6} Pa, several h) [Fe-25] or to combine the sputter–anneal cycle with oxidation–reduction treatments in oxygen (1070 K, ~10^{-3} Pa, long periods) and hydrogen (1070 K, ~10^{-2} Pa, long periods) [Fe-26]. Oxygen and sulfur were observed to concentrate on the surface at 820 K and above 870 K, respectively [Fe-17, Fe-19, Fe-20].

(111) Surface [Fe-11, Fe-20, Fe-27 to Fe-30]. The (111) surface has been cleaned by argon-ion sputtering (250–500 eV, 1–10 μA/cm^2) and annealing (900–1120 K) cycles [Fe-11, Fe-20, Fe-27 to Fe-30]. The sputtering was done at 700 K [Fe-20] or at ambient temperature [Fe-11, Fe-27, Fe-30]. Sulfur migration to the surfaces began at 670 K and produced a monolayer of sulfur at 970 K [Fe-27, Fe-29]. Oxygen, carbon and nitrogen were observed to dissolve into the bulk metal at 720 K [Fe-27].

Lanthanum, La (hcp, T_m = 1194 K)

Lanthanum foil (99.9% pure) was sputtered in static argon (900 eV, 11 μA, ~6×10^{-3} Pa), but the surface was not freed of oxygen [La-1].

Lead, Pb (fcc, T_m = 601 K)

Lead is commercially available in polycrystalline and single-crystalline forms with nominal purity of 99.999%. The major non-metallic impurities are carbon, oxygen, nitrogen and hydrogen. All investigators succeeded in reducing the surface concentrations of oxygen and carbon to about one percent of a monolayer.

Polycrystalline surface [Pb-1 to Pb-3]. Polycrystalline lead foils were cleaned by prolonged argon-ion bombardment (700 eV, 5–10 μA, ~2.7×10^{-3} Pa, several h) until the oxygen 1s XPS signal fell below the detection level (~0.1%

of lead 4f intensity) and the carbon 1s signal had ceased to diminish. Bombardment of the surface with microwave-discharged oxygen ($\sim 10^{-3}$ Pa, 2450 MHz, 80 W) followed by further ion sputtering reduced this residual carbon to a negligible level ($\sim 0.1\%$ of lead 4f intensity) [Pb-1]. Polycrystalline surfaces have also been cleaned successfully by in-situ scraping with a tungsten carbide blade [Pb-2] or micromilling with a rotating diamond edge [Pb-3].

Single crystal surface [Pb-4]. Single crystal surfaces (planes not specified) were cleaned by repeated argon-ion bombardments (900 eV, 10 μA, 15 min). All impurity XPS lines were eliminated by this procedure.

(100) and (110) surfaces [Pb-5]. These surfaces have been cleaned in situ by argon-ion bombardment and annealing (430 K). The surface showed no traces of impurity at the detection limits for oxygen and carbon. The O(1s):Pb(4f) and C(1s):Pb(4f) XPS intensity ratios were $\leqslant 0.001$.

Lithium, Li (bcc, $T_m = 454$ K)

Bulk samples of lithium (99.97% pure) have been cleaned by abrading in UHV with a file [Li-1] and by argon-ion bombardment (1–3 keV, ~ 5 $\mu A/cm^2$, 300 K, ~ 2.7 h) [Li-2, Li-3]. Sodium segregation to the lithium surface at room temperature was found to be a major source of contamination [Li-2]. To our knowledge, no work has been reported on single crystals of lithium.

Lutetium, Lu (hcp, $T_m = 1936$ K)

Attempts to clean the (100) surface by ion bombardment and annealing were unsuccessful because oxygen and carbon segregated at the surface upon cooling [Lu-1]. Clean surfaces have been prepared by evaporation procedures.

Magnesium, Mg (hcp, $T_m = 1090$ K)

We found two investigations [Mg-1, Mg-2] that reported cleaning procedures for solid metal surfaces. In both studies a polycrystalline magnesium surface was cleaned by argon-ion bombardment (3 keV) [Mg-1] and annealing in vacuum.

Manganese, Mn (cub, $T_m = 1517$ K)

We were unable to locate any data on cleaning bulk manganese. Clean, thin films of this metal have been produced by various deposition techniques.

Molybdenum, Mo (bcc, $T_m = 2890$ K)

Sulfur and carbon are the contaminants that must be purged from molybdenum to achieve a clean surface. Heating to approximately 800 K for a short time led to sulfur segregation on the (100), (110) and (112) surfaces; it also led to carbon segregation on the (110) and (112) surfaces, but not appreciably on the (100) surface [Mo-1]. Although AES can be used to uniquely monitor the presence of carbon, detection of small amounts of sulfur is difficult because of the overlap of the S(KLL) and the Mo(MNN) Auger peaks at approximately 150 eV. However, the sum of the amplitudes of the sulfur and molybdenum peaks (S + Mo) at 150 eV relative to the purely molybdenum peak near 163 eV has been shown to be practically constant for incident electron energies over 400 eV when sulfur contamination was quite low [Mo-2]. For a fixed incident energy, the ratio of Auger peak amplitudes was monitored as a function of cleaning steps or cycles; the minimum ratio was taken as corresponding to a clean surface [Mo-3]. Using the ratio of the S + Mo peak at ~ 150 eV to the purely molybdenum peak at ~ 188 eV, published AES spectra of "clean" surfaces have a ratio of less than 0.10 [Mo-3, Mo-4, Mo-5]. Quantifying the level of sulfur contamination requires careful spectrum modeling and stripping and is not normally undertaken as part of a cleaning process. The best established cleaning procedure is reaction with oxygen to remove carbon (as CO gas) and possibly sulfur, followed by flashing to high temperature in UHV to desorb the molybdenum oxides formed.

Polycrystalline surface [Mo-6 to Mo-8]. When only sulfur segregated on the surface, either argon-ion bombardment at elevated temperatures (~ 1200 K), heating at 1100–1500 K in oxygen, or flashing at ~ 2100 K in UHV has been used to clean the surface. When carbon was present, heating at 1500 K in 1.3×10^{-4} Pa oxygen followed by flashing to 1900–2100 K in UHV yielded a surface free of carbon, sulfur and oxygen.

(100) Surface [Mo-3, Mo-4, Mo-9 to Mo-14]. Carbon, oxygen and sulfur appeared on the surface after heating above 1300 K. All three contaminants were removed by repeated application of the oxygen–anneal treatment detailed for the polycrystalline surface. A lack of flatness was found for surfaces that had been subjected to oxygen–anneal treatments with flashes limited to 1900 K, however, prolonged annealing at 2200–2300 K led to a flattening of the surface [Mo-12]. Carbon and oxygen have also been removed by multiple cycles of argon-ion bombardment (at 45° from surface normal) and annealing at 1700 K; these cycles were preceded by an initial oxygen–anneal treatment (heating at 1700 K in ~ 7×10^{-4} Pa oxygen followed by annealing at 1700 K in UHV) [Mo-10].

(110) Surface [Mo-4, Mo-5, Mo-15, Mo-16]. This surface plane has usually been cleaned by repeated heating at 1200–1800 K in ~ 10^{-4} Pa oxygen and subsequent flashing at 1900–2200 K in UHV. In one case, the oxygen treatment was performed at 1800 K, and the oxide was reportedly removed by heating at 1800 K in 10^{-2} Pa hydrogen followed by flashing to 2400 K [Mo-16]. The usefulness of this hydrogen reduction step appears negligible because other investigators needed only the high temperature flash after the

oxygen treatment to obtain a clean surface.

(111) Surface [Mo-4, Mo-17]. The oxygen–anneal treatment detailed for the polycrystalline surface has been used successfully to clean this plane. However, prolonged heating at temperatures above 1700 K in ~ 7×10^{-6} Pa oxygen may lead to irreversible faceting to (112) planes [Mo-17].

(112) Surface [Mo-18]. Heating to 1300 K resulted in sulfur segregation on the surface but the sulfur disappeared at temperatures above 1500 K. Carbon was removed by the oxygen–anneal treatment detailed for the polycrystalline surface.

Neodymium, Nd (hex; bcc > 1135 K, T_m = 1294 K)

A neodymium crystal (orientation not specified) was "cleaned" by heating in UHV at 1000 K, but SXAPS measurements disclosed that carbon and oxygen contamination remained [Nd-1]. It is doubtful that this treatment alone would produce atomically clean surfaces. A useful procedure should be cycles of argon-ion bombardment and annealing in UHV.

Neptunium, Np (orth; tetr > 551 K; cub > 773 K, T_m = 913 K)

We found no surface studies on bulk or thin-film neptunium.

Nickel, Ni (fcc, T_m = 1726 K)

Nickel was the first element to be subjected to surface analysis [Ni-1] and has been one of the most thoroughly studied elements. For a compilation of work done prior to the advent of techniques to verify the state of surface cleanliness, the reader is referred to a useful bibliography [Ni-2]. Surfaces prepared from nickel samples of 99.99 to 99.9995% nominal purity have been found to contain carbon, sulfur and oxygen contaminants. These can usually be eliminated by a number of sputter–anneal cycles. However, many workers found carbon to be very persistant and resorted to high-temperature treatments in oxygen for cleaning. The latter treatment often left an oxygen residue on the surface which could be removed in most cases by a high-temperature tretment in hydrogen and/or additional sputter–anneal cycles. The utilization of these chemical treatments varied with the crystal face.

Polycrystalline surface [Ni-3 to Ni-9]. Argon-ion bombardment (no specific conditions listed) with periodic annealing to 1000 to 1300 K either during bombardment [Ni-3] or in UHV [Ni-4, Ni-5] has been found to produce clean nickel surfaces. This basic treatment has also been combined with oxygen exposures (900 K, ~ 10^{-5} Pa, ~ 1000 s) and hydrogen reduction (900 K, ~ 10^{-5} Pa, ~ 1000 s) to remove residual carbon [Ni-6, Ni-7]. Removal of sulfur has been effected by sputtering hot nickel with hydrogen ions (~ 2 keV, 10–30 $\mu A/cm^2$, 920 K) [Ni-8]. In-situ sanding was also used to produce a clean surface [Ni-9].

(100) Surface [Ni-5, Ni-10 to Ni-48]. Many authors found cycles of argon-ion sputtering (100–500 eV, 0.3–10 $\mu A/cm^2$) and annealing (625–1170 K) sufficient to obtain clean nickel surfaces [Ni-10 to Ni-25]. The number of cycles reported varied from 15 [Ni-22] to 100 [Ni-11]. Although most researchers sputtered with the sample at room temperature, some reported satisfactory results with the sample at ~620 K [Ni-14, Ni-24]. One author reported the generation of a clean nickel surface by cycles of sputtering with neon ions at elevated temperatures (670–970 K) and annealing (670 K) in UHV [Ni-25]. Surfaces cleaned in the manner described above were frequently found to obtain ~0.05 monolayers of carbon which resisted all attempts at removal. In those cases, a chemical treatment in oxygen was necessary [Ni-5, Ni-26 to Ni-40]. Most groups carried out this operation at pressures from 7×10^{-7} to 7×10^{-4} Pa and at elevated temperatures (550–1200 K) [Ni-5, Ni-26, Ni-28 to Ni-40]; however, exposure to oxygen at temperatures above 300 K has been reported to result in solution of oxygen into the bulk [Ni-27]. One interesting variation on this procedure consisted of simultaneously bombarding the sample with krypton and oxygen ions (no specific conditions listed) [Ni-41]. The use of oxygen to aid in carbon removal generally left an oxygen residue on the cleaned surface. This was removed by high-temperature treatments in hydrogen (950–1400 K, 10^{-5} to 10^{-2} Pa) [Ni-42 to Ni-48].

(110) Surface [Ni-3, Ni-11, Ni-19, Ni-26, Ni-39, Ni-42, Ni-49 to Ni-79]. The (110) surface was usually cleaned by iterations of argon-ion sputtering (250–500 eV, 1–40 $\mu A/cm^2$) and annealing (470–1170 K) [Ni-3, Ni-11, Ni-19, Ni-49 to Ni-70] for up to 100 cycles [Ni-11]. However, some groups did use higher ion-beam energies (2.8–3 keV) [Ni-57 to Ni-59]. Sulfur was observed to segregate to the surface between 870 and 1120 K [Ni-51, Ni-53]. Generally, repetitions of the sputter–anneal cycle outlined above depleted the sulfur from the near-surface region. However, removal of the carbon contamination remaining after many cycles was effected by heat treatments in oxygen gas. This step was done at either elevated sample temperatures (820–1070 K) [Ni-71, Ni-26] or at ambient temperature with subsequent heating to 670–870 K [Ni-72 to Ni-74]. Occasionally, this treatment left residual oxygen on the surface which was removed by reduction in hydrogen (650–1300 K, ~10^{-4} Pa) [Ni-42, Ni-45, Ni-46, Ni-75 to Ni-78]. Carbon and oxygen were also removed by electron-stimulated desorption (1500 eV) at 1070 K and 1270 K, respectively [Ni-79].

(111) Surface [Ni-5, Ni-11, Ni-19, Ni-26, Ni-27, Ni-32, Ni-45, Ni-46, Ni-80 to Ni-96]. Cycles of argon-ion sputtering (400–600 eV, 1–10 $\mu A/cm^2$) and annealing (473–1200 K) were reported to produce clean (111) surfaces in some cases [Ni-11, Ni-19, Ni-80 to Ni-85]. Carbon remaining on the surface could be removed by oxygen treatments (800–1100 K, 10^{-4} Pa) [Ni-5, Ni-26, Ni-32, Ni-86 to Ni-90]. In some studies the decarburization process consisted of a lengthy treatment in oxygen (1170 K, 10^{-4} Pa) followed by repolishing of the crystal [Ni-86 to Ni-88]. Oxygen adsorption at ~300 K followed by flash heating to 850–1200 K was also effective in carbon removal [Ni-27, Ni-42, Ni-91, Ni-92].

Removal of residual oxygen was effected by treatment in hydrogen (820–1300 K, $\sim 10^{-3}$ Pa) [Ni-42, Ni-45, Ni-46, Ni-77, Ni-93 to Ni-95]. Cleaning by heating alone has been reported ($\sim$ 100 cycles, 1450 K in UHV for 45 s) [Ni-96].

Niobium, Nb (bcc, $T_m = 2741$ K)

Removal of carbon was the most difficult aspect of cleaning niobium surfaces. Many investigators found that mere heating for prolonged times above 2300 K was sufficient to clean the surface [Nb-1 to Nb-4]. An oxygen-anneal treatment was found effective by one group [Nb-5, Nb-6]. Argon-ion sputtering followed by anneals has also produced clean surfaces [Nb-7, Nb-8].

Polycrystalline surface [Nb-1, Nb-2, Nb-5]. Prolonged heating above 2300 K in UHV has been demonstrated to produce clean surfaces [Nb-1, Nb-2]. Decarburization at 2000 K in 1.3×10^{-4} Pa oxygen followed by heating in UHV at 2300 K also purified the surface [Nb-5].

(100) Surface [Nb-1, Nb-3, Nb-4, Nb-7, Nb-8]. Heating for several hours at 2300 K in UHV was shown to be quite effective in cleaning this plane [Nb-1, Nb-3, Nb-4]. An oxygen–anneal treatment was attempted but found unsatisfactory [Nb-3]. Ion bombardment with anneals above 2300 K has also yielded clean surfaces [Nb-7]. Numerous cycles of argon-ion sputtering (0.3–3 keV, $\leq 10\ \mu A/cm^2$) and anneals at 1000 K purged the sulfur and carbon from the sample but left a small, unspecified amount of oxygen on the surface of a crystal having approximately 0.1 at% oxygen in the bulk [Nb-8]. However, this low-temperature procedure may prove effective for higher purity niobium.

(110), (111) and (750) surfaces [Nb-6 to Nb-8]. Both heating at 2300 K with a flash to 2700 K [Nb-7] and oxygen–anneal treatments (similar to those used to clean the polycrystalline surfaces) [Nb-6] have been found effective in cleaning the (110) planes. The only documented procedure for the (111) surface is to heat it at 2300 K in UHV followed by flashing to 2700 K [Nb-7]. A low-temperature bombardment–anneal treatment may prove quite effective for cleaning the (750) surface [Nb-8].

Osmium, Os (hcp, $T_m = 3318$ K)

In spite of its prominent position in the periodic table, very few surface studies have been done on osmium. Atomically clean surfaces of polycrystalline osmium have been prepared by repeated cycles of flashing to 1670 K in UHV and bombardment with 2 keV argon ions [Os-1].

Palladium, Pd (fcc, $T_m = 1825$ K)

The main contaminants are sulfur and carbon which segregate on the surface during mild heating in UHV. Sulfur concentrations on the surface could be estimated directly from AES; but AES detection of less than a 0.25 monolayer of carbon is difficult due to overlap of the C(KLL) peak at approximately 270 eV and the large Pd(MNN) peak at 279 eV [Pd-1]. Lower levels of carbon contamination were detected indirectly by CO and CO_2 desorption from a palladium surface following oxygen adsorption at room temperature [Pd-2]. Only O_2 was desorbed from a clean surface after oxygen adsorption [Pd-2, Pd-3]. The three most effective cleaning techniques were (1) reaction with oxygen to remove carbon as CO and CO_2 followed by flashing in UHV to desorb the oxygen as O_2, (2) alternate noble-ion sputtering and annealing, and (3) combinations of (1) and (2).

Polycrystalline surface [Pd-4 to Pd-8]. Heating a palladium ribbon to 1500 K for a few minutes in UHV was reported to be sufficient to obtain a clean surface [Pd-6]. However, most investigators used more complicated cleaning procedures. The most effective oxygen–anneal treatment was to heat the material at 1000 K in 1.3×10^{-4} Pa oxygen and follow with flashing at 1200 K in UHV [Pd-4]. A similar oxygen–anneal treatment with each of the two temperatures reduced by 200–300 K resulted in a sample that exhibited sulfur segregation after subsequent heating to temperatures above 600 K in UHV [Pd-7]. Repeated oxygen exposure and argon-ion bombardment at approximately 1200 K were found to provide a clean surface [Pd-5]. Alternate cycles of argon-ion bombardment (10 keV, 1 μA/cm^2) and annealing at 900 K were repeated until a clean surface was obtained [Pd-8].

(100) Surface [Pd-1, Pd-9, Pd-10]. Sulfur and carbon were removed completely using argon-ion sputtering (500 eV, $\sim 5\,\mu$A/cm^2, 900 K) for about 300 h per mm of sample thickness followed by annealing at 1100–1300 K. The rate of impurity removal appeared to be limited by the rate of segregation of sulfur and carbon on the surface [Pd-1]. In one case, elimination of the last traces of carbon required additional heating in oxygen (1000 K, $\sim 7 \times 10^{-5}$ Pa) followed by brief argon sputtering to remove the oxygen.

(110) Surface [Pd-3, Pd-11]. Initial cleaning has been accomplished by alternating cycles of inert-gas ion sputtering and annealing [Pd-3, Pd-11]. Remaining traces of carbon were removed by treatment in $\sim 3 \times 10^{-5}$ Pa oxygen at 800 K followed by flashing at 1300 K to desorb oxygen [Pd-3].

(111) Surface [Pd-2, Pd-9, Pd-11 to Pd-13]. Most investigators found that repeated argon-ion sputtering and annealing were sufficient to remove sulfur; but complete carbon removal required an additional oxygen–anneal treatment (similar to that given for the polycrystalline surfaces).

(210) Surface [Pd-9, Pd-11, Pd-14]. Cleaning procedures were essentially the same as for (111) surfaces.

(311) Surface [Pd-11]. Argon sputtering and annealing left some residual carbon.

Phosphorus, P (cub, $T_m = 317$ K)

We found no surface studies on bulk or thin-film phosphorus.

latinum, Pt (fcc, $T_m = 2045$ K)

The major impurities detected on high-purity platinum single crystal surfaces were carbon, calcium and phosphorus. Other impurities commonly observed on platinum were sulfur, silicon, chlorine and oxygen. Carbon presented a special problem because it dissolved into the bulk above 1423 K and precipitated onto the surface below 1348 K [Pt-1]. Carbon has been removed by three different procedures: (1) heating in a partial pressure of oxygen [Pt-2]; (2) extended argon-ion bombardment [Pt-3]; (3) bombardment at elevated temperatures (950–1150 K) [Pt-4]. Calcium was found to segregate on the surface during heating [Pt-5, Pt-6]. Calcium and the other impurities could be easily removed by ion bombardment or by heating in UHV [Pt-5, Pt-7].

Polycrystalline surface [Pt-8]. A high purity platinum foil was cleaned by heating to 1400 K in UHV, argon-ion sputtering, heating in 1.3×10^{-4} Pa oxygen for 10 min at 1300 K, and finally annealing in UHV (1300 K, 10–20 min). Cleanliness was verified by XPS.

(100) Surface [Pt-2, Pt-7, Pt-9 to Pt-16]. Several investigators [Pt-2, Pt-9 to Pt-11] prepared a clean (100) crystal face by one or more cycles of heating in an oxygen partial pressure (1000–1300 K, 10^{-3}–10^{-5} Pa) and annealing in UHV (973–1700 K). Others [Pt-7, Pt-12 to Pt-16] added an argon-ion bombardment step (300–500 eV) to the treatment cycle. One of the more lengthy cleaning procedures consisted of argon-ion bombardment (300 eV, 2 h) followed by anneals in oxygen (1173 K, 7×10^{-6} Pa, 15 h) and in UHV (1373 K, 7×10^{-8} Pa, 30 h) [Pt-12]. Two surface structures have been observed by LEED on the cleaned (100) crystal faces after use of similar cleaning procedures: (5 × 1) [Pt-2, Pt-9, Pt-12 to Pt-14] and (5 × 20) [Pt-7, Pt-10, Pt-15, Pt-16]. The reasons for this apparent anomaly are not clear at this time.

(110) Surface [Pt-5, Pt-17, Pt-18]. A clean (110) surface was prepared by one or more cycles of heating in oxygen (973–1300 K, 10^{-4}–10^{-5} Pa) and argon-ion bombardment. The ion bombardment was required to remove calcium [Pt-5, Pt-18]. Even after annealing the surface in oxygen ($> 10^{-4}$ Pa) no oxygen was detected by AES on the reconstructed surface, but the surface structure was reported to be a function of the cleaning procedure used [Pt-18]. Argon-ion bombardment (500 eV) resulted in an unstable (1 × 1) structure that changed to a (1 × 2) structure when heated to 773 K. Heating above 1073 K in 10^{-4} Pa of oxyge [illegible] ulted in a (1 × 3) surface structure [Pt-18].

(111) Surface [illegible] to Pt-4, Pt-6, Pt-7, Pt-9, Pt-14, Pt-15, Pt-17]. Calcium was observed to segregate on the surface of the (111) crystal face above 1400 K [Pt-6]; carbon precipitated on the surface when the sample was cooled below 1348 K [Pt-1]. Several cleaning procedures have been used to generate clean (111) crystal faces: (1) a sequence of one or more argon-ion bombardments (~ 500 eV), heatings in oxygen (673–1273 K, 7×10^{-4} Pa) and annealing in UHV at 1373 K [Pt-7, Pt-14]; (2) argon-ion bombardment and heating in oxygen (673 K, 3×10^{-5} Pa) [Pt-15]; (3) oxygen treatment and annealing

without argon-ion bombardment [Pt-2, Pt-6, Pt-9]; (4) cycles of prolonged argon-ion bombardment (600 eV) and heating to 1400 K in UHV [Pt-3]; (5) argon-ion bombardment for several hours at 950–1150 K followed by a final flash in UHV to 1300 K [Pt-4].

(112), (113), (133), (122) and (012) surfaces [Pt-19]. These surfaces were cleaned by argon-ion bombardment, heating in an oxygen partial pressure and annealing in UHV.

Plutonium, Pu (mon; bc mon >388 K, fc orth >458 K, fcc >583 K; bc tetr >725 K; bcc >753 K; $T_m = 913$ K)

The cleaning of plutonium surfaces is complicated by the radiation hazard resulting from alpha particle emission and by the low-temperature α-monoclinic to β-body-centered-monoclinic phase transition, which is accompanied by a large change in density. Interstitial impurities (sulfur, chlorine, phosphorus, oxygen and carbon) segregated to the surface during cleaning with carbon and oxygen being the most difficult to remove. An oxygen-free surface could not be obtained by in-situ scraping with a titanium carbide blade [Pu-1]. AES and XPS measurements on a plutonium sample showed that removal of oxygen, carbon, chlorine and sulfur was effected by the use of argon-ion sputtering (1 keV, ~ 130 μA/cm^2); however, a large amount of chlorine segregated to the surface upon heating (773 K, 30 min) and plutonium carbide was formed from residual gases. After 25 sputter–heating cycles, the surface was estimated to have 10 at% oxygen as oxides and 5 at% carbon as carbides [Pu-2]. Because of the great affinity of plutonium for oxygen and carbon it is doubtful that a surface free of these two contaminants can be produced using the purest metal currently available. The following recommended cleaning procedure has been formulated based on our experience with thorium and uranium (see refs. [Th 2 to Th-6] and [U-7]). Remove the gross contamination with argon-ion bombardment (0.5–4 keV, $1-5 \times 10^{-5}$ A/cm^2, 300 K), follow with bombardment of the sample beginning at temperature near 676 K and continue with gradual cooling to room temperature; this procedure may have to be repeated many times before a clean surface is produced. Then anneal at a temperature below 676 K for several minutes in a vacuum of less than 10^{-8} Pa. This procedure is applicable to polycrystalline specimens only, and the sample must be mounted to accommodate changes in volume caused by the phase changes. For single crystalline α-plutonium, the heating temperature cannot exceed 388 K.

Polonium, Po (mon, $T_m = 527$ K)

We found no surface studies on bulk or thin-film polonium.

Praseodymium, Pr (hex, $T_m = 1204$ K)

We found no surface studies on bulk praseodymium. Praseodymium has been evaporated to form films.

Promethium, Pm (hex, $T_m = 1350$ K)

We found no surface studies on bulk or thin-film promethium.

Protactinium, Pa (tetr, $T_{tr} = < 1873$ K)

We found no surface studies on bulk or thin-film protactinium.

Radium, Ra (bcc, $T_m = 973$ K)

We found no surface studies on bulk or thin-film radium.

Rhenium, Re (hcp, $T_m = 3453$ K)

The majority of the authors surveyed found that rhenium is effectively cleaned by heating in oxygen ($2000 \leq T \leq 2500$ K, $\sim 10^{-4}$ Pa) and then annealing in UHV. Polycrystalline samples subjected to this treatment produced crystallites that were oriented parallel to the (0001) plane [Re-1 to Re-5]. The polycrystalline and single-crystalline samples obtained from various suppliers ranged from 99.95 to 99.995% pure. The contaminants most frequently encoutered on rhenium surfaces were sulfur, chlorine, carbon and nitrogen.

Polycrystalline surface [Re-1 to Re-9]. Heating in oxygen (2000–2500 K, $\sim 10^{-4}$ Pa, 2–24 h) and then flash annealing in UHV (~ 2200 K) was the most cited method for producing clean rhenium surfaces [Re-1 to Re-6]. An alternate method that was also successful consisted of the following sequence: (a) conduct preliminary decontamination by argon-ion bombardment (2 keV); (b) heat in oxygen (1470 K, 2.7×10^{-6} Pa); (c) flash anneal in UHV (1970 K); (d) repeat (b) and (c) until clean [Re-7]. Another variation consisted of argon-ion sputtering (2 keV) and annealing (1970 K) cycles repeated until a contaminant-free surface was obtained [Re-8, Re-9].

(0001) Surface [Re-1, Re-10 to Re-14] Unreconstructed (0001) surfaces were obtained by heating in oxygen (2200–2770 K, 10^{-5}–10^{-4} Pa, ~ 2 min) followed by several flash anneals in UHV (2200–2770 K, ~ 20 s) [Re-1, Re-10, Re-11]. Argon-ion bombardment has also been utilized to produce clean (0001) surfaces by repeated cycles of sputtering (390 eV, ~ 10 μA/cm^2, 300 K) [Re-12] and annealing in UHV (1300 or 1770 K, 30 s) [Re-13] until all impurities were removed. In one case [Re-13] this procedure was followed by a long anneal (1500 K, several h) without segregation of any impurity to the surface. One author reported obtaining a clean surface after heating the sample to 1770 K in UHV several times [Re-14].

Rhodium, Rh (fcc, $T_m = 2239$ K)

Bulk rhodium samples of 99.9 to 99.999% purity have been reported to contain the following impurities: carbon, sulfur, boron, oxygen, silicon and magnesium. Of these, boron was the most troublesome as it migrated to the surface at ~1300 K [Rh-1 to Rh-3]. Pretreatment of the (110) surface by annealing the sample in flowing hydrogen at 1270 K for 90 h was reported to alleviate this problem [Rh-4]. However, most workers reported methods for boron depletion that involved either argon-ion sputtering [Rh-1, Rh-2, Rh-5, Rh-6] or heat treatments in oxygen [Rh-1, Rh-5, Rh-6]. Heating in hydrogen (~600 K, ~10^{-5} Pa) was found to be a good way to remove traces of adsorbed oxygen in the final cleaning stage [Rh-1, Rh-5].

(100) Surface [Rh-1, Rh-5]. In the initial cleaning stage, various surface contaminants were removed by argon-ion sputtering (500–2000 eV, ~5 μA, 300 K, ~10 min), and carbon was removed by either annealing in UHV or in oxygen (10^{-4} Pa) at 1000–1270 K. This was followed by repetition of the following steps: (a) argon-ion sputtering (as above); (b) heating in oxygen (~1270 K, ~10^{-5}–10^{-4} Pa); (c) annealing in UHV at ~900 K for a few minutes; (d) heating in hydrogen (~600 K, ~10^{-5} Pa). This treatment sequence eventually depeted boron in the samples.

(110) Surface [Rh-2, Rh-4]. This surface has been successfully cleaned by a combination of argon-ion sputtering (~300 eV, 10 μA/cm^2, 300–970 K), heating in oxygen (670–1300 K, 7×10^{-6} to 10^{-4} Pa), and annealing in UHV (1270–1300 K).

(111) Surface [Rh-3, Rh-5, Rh-7]. Argon-ion bombardment (1–2 keV, 1–10 μA, 300–1000 K, ~10 min) followed by "high-temperature" treatment in oxygen (~10^{-5} Pa) and flash annealing to 1250–1300 K have been reported to produce clean (111) surfaces.

(775) and (331) surfaces [Rh-6]. Carbon was removed by heating to 1270 K in UHV. Sulfur has been removed by argon-ion etching (2 keV, 300 K). Boron was eliminated by repeated cycles of argon-ion bombardment (500 eV, 300 K) and annealing at 1070 K; it was removed from the (331) surface after a few cycles, but many more cycles were required to cleanse the (775) surface.

Ruthenium, Ru (hcp, $T_m = 2583$ K)

The impurities most often found in ruthenium single crystals were carbon, sulfur and oxygen. The absence of carbon on clean ruthenium surfaces was difficult to confirm by AES because its MNN Auger peak at 274 eV interferes with the carbon KLL Auger peak at ~272 eV. One method for establishing the state of cleanliness has been to obtain a minimum in the ratio of $R = (\mathrm{Ru}_{274} + \mathrm{C}_{272})/\mathrm{Ru}_{235}$, because the ruthenium Auger peak at 235 eV is free of interfering effects from carbon [Ru-1, Ru-2]. The lowest R value found in a survey of published Auger spectra was 1.57 for the (110) surface [Ru-1];

values between 1.85 and 2.05 were more typical [Ru-2 to Ru-4]. The absence of CO in the thermal desorption spectra from ruthenium exposed to several langmuirs of oxygen has also been used to insure the absence of carbon [Ru-5]. A series of small, temperature-dependent peaks was found between 300 and 500 eV in the Auger spectrum of almost all the crystal faces of ruthenium [Ru-1 to Ru-4, Ru-6, Ru-7]. These peaks could not be removed with up to 200 h of argon-ion sputtering [Ru-6]; they have been interpreted to be diffraction peaks characteristic of clean surfaces [Ru-7], although some ambiguity remains [Ru-4]. The low index planes of ruthenium could be cleaned by a combination of heating in gas (usually oxygen at low pressures) and a brief anneal at a higher temperature.

(001) Surface [Ru-3 to Ru-16]. Most authors [Ru-3 to Ru-14] cleaned the (001) plane by repeatedly heating in oxygen (1300–1500 K, 7×10^{-6}–1.3×10^{-5} Pa, 10–15 s) and annealing (1500–1600 K, UHV) to remove adsorbed oxygen. An alternate method [Ru-15, Ru-16] was to heat the sample in hydrogen (1450–1500 K, $\sim 7 \times 10^{-5}$ Pa). This procedure was reported to remove adsorbed oxygen completely [Ru-15]. Some authors also carried out a preliminary cleaning step by submitting their samples to argon-ion bombardment [Ru-4, Ru-12, Ru-14].

(110) Surface [Ru-1, Ru-17, and Ru-18]. Repeated cycles of heating in oxygen (~ 1500 K, $\sim 10^{-5}$ to $\sim 10^{-3}$ Pa) and annealing (~ 1400–1550 K) have been found to be an effective method for cleaning the (110) plane.

$(10\bar{1}0)$ Surface [Ru-2, Ru-19]. Two methods for cleaning $(10\bar{1}0)$ surfaces have been published. In the first, the sample was heated to 1470 K, submitted to argon sputtering, and annealed below 1270 K. This process yielded an Auger spectrum with $R \simeq 1.85$ at 470 K and sharp LEED patterns [Ru-2]. The second method consisted of a preliminary outgassing at 770 K and cycles of argon-ion bombardment and annealing at 1270–1470 K [Ru-19].

Samarium, Sm (rhdr, $T_m = 1350$ K)

Polycrystalline samples of samarium have been cleaned by repeated argon-ion bombardment and annealing (conditions not specified) [Sm-1]. AES analysis of the sample disclosed "very small" amounts of carbon and oxygen after cleaning.

Scandium, Sc (fcc, $T_m = 1814$ K)

Preparation of an annealed surface that remains clean at room temperature has not been reported. Repeated cycles of argon-ion bombardment and annealing at temperatures above 1100 K produced a surface that remained clean only as long as the temperature was held above 1100 K. Oxygen, carbon, sulfur and chlorine reappeared on the surface when the sample was cooled to ambient temperature. This phenomenon was found to hold for polycrystalline

[Sc-1], (100) [Sc-2, Sc-3] and (001) [Sc-3] surfaces. In one AES study, the sulfur Auger peak increased to a maximum at 850 K, but carbon and scandium peaks decreased slightly to a minimum near 850 K with only small changes in the oxygen and chlorine peaks [Sc-2]. Chlorine disappeared above 900 K, sulfur was not observed above 950 K, and oxygen and carbon vanished at 1100 K.

Selenium, Se (hex, $T_m = 490$ K)

We found no surface studies on bulk or thin-film selenium.

Silicon, Si (cub-dia, $T_m = 1683$ K)

Carbon and oxygen are the major impurites on high purity, single-crystal silicon surfaces. Carbon is the most dificult to remove because of its low sputter yield and its high thermal stability. We found three methods for preparing a clean silicon surface [Si-1]: (1) heating above 1473 K, (2) argon-ion bombardment and annealing (~ 1000 K), and (3) in-situ cleaving of a (111) surface.

Polycrystalline surface [Si-2]. Polycrystalline surfaces have been cleaned by brief argon-ion sputtering (5 keV, 2.5 μA/cm^2, 10 s) without creating any surface texture. However, changes in the surface characteristics were observed after 45 s of sputtering.

(100) and vicinal surfaces [Si-3 to Si-8]. The (100) crystal face was cleaned by repeated cycles of argon-ion bombardment (500 eV) and annealing (1173 K, UHV) [Si-3, Si-4] or by simply heating the crystal in UHV to 1473–1523 K for 15–150 s [Si-5 to Si-7]. Although the (100) vicinal surfaces are quite similar to the (100) arrangement, somewhat difference specifics have been used in cleaning the vicinal surface. Repeated cycles of argon-ion bombardment (350 eV, 20 min) and heating (1375 K, 2 min, followed by 1223 K, 30 min) produced a clean AES spectrum and an optimized RHEED pattern for a (100) vicinal surface [Si-8].

(110) Surface [Si-9]. Clean (110) crystal faces have been prepared by ion bombardment or by heating in vacuum. No details were given.

(111) Surface [Si-1, Si-5, Si-6, Si-10 to Si-13]. A clean reconstructed (111) crystal face has been prepared by cleaving in UHV; the resultant (2 × 1) LEED structure changed to a (1 × 1) structure on heating to 700 K and to a (7 × 7) structure on heating to 1000 K [Si-1]. The clean (111)-(7 × 7) reconstructed surface was also achieved by argon-ion bombardment (2 keV, 4 μA/cm^2) and annealing (1073 K, 30 min) [Si-10]. In most of the surface studies on the (111) crystal face, rapid or flash heating to 1473–1523 K was used to prepare a clean surface. Conflicting reports exist on carbon removal: heating to only 1183 K in UHV did not remove carbon but produced a carbide structure [Si-12]; however, heating in vacuum to 1200 K and slowly cooling to room temperature resulted in a Si(111)-(7 × 7) surface that had less than 5% of a monolayer of carbon [Si-11].

Silver, Ag (fcc, $T_m = 1235$ K)

Polycrystalline and single-crystalline samples of silver (99.99 to 99.9999% pure), have been found to contain mainly sulfur, carbon, oxygen and chlorine impurities. The most commonly used method for establishing surface cleanliness has been AES. However, the carbon KVV Auger transition at ~ 272 eV is superimposed on the silver MNN transitions at ~ 260 and 266 eV. In order to establish the absence of carbon, most investigators relied on the ratio

$$R = (C_{272} + Ag_{260+266})/Ag_{303},$$

since the silver MNN Auger transition at ~ 303 eV is well separated from any carbon Auger features. R values for clean silver have been recorded in the range of 0.42–0.55, depending on the modulation voltage used to obtain the spectrum. There is also an overlap of the N(KLL) peaks at ~ 358 and 360 eV with the Ag(MNN) peaks at ~ 351 and 356 eV. The cleaning of silver samples in UHV was usually accomplished by a combination of argon-ion sputtering and annealing.

Polycrystalline surface [Ag-1 to Ag-3]. Silver foils have been cleaned by argon-ion bombardment [Ag-1] at 2 keV [Ag-2] or by prolonged heating to 1000 K in UHV [Ag-3]. Free evaporation of silver took place in the latter case, and the resulting surface consisted mainly of crystallites oriented in such a way as to expose their (111) surfaces.

(100) Surface [Ag-4]. The initial cleaning of (100) surfaces has been accomplished by repeating a cycle of argon-ion bombardment (200–500 eV) and annealing in UHV (670 K) until sharp LEED patterns and R values of 0.40–0.45 were obtained. Subsequent cleaning was carried out by heating to 620 K in UHV. Occasionally, another cycle of sputtering and annealing was required. Embedded argon was observed to desorb between 300–450 K.

(110) Surface [Ag-3 to Ag-13]. The (110) surface has been cleaned by cycles of argon-ion sputtering and annealing in UHV. Most of the authors reviewed preferred to use a gentle ion bombardment (300–600 eV, 1–3 μA/cm^2, 300 K) [Ag-3 to Ag-7]; however, others executed this operation at higher ion energies (1 keV [Ag-10]; 5 keV [Ag-11]). The annealing temperatures ranged from 670 to 720 K [Ag-4 to Ag-6, Ag-8], although some groups did anneal their samples at a higher temperature (800 K [Ag-9, Ag-10], 1000 K [Ag-7]). Oxygen dissolved into the bulk upon heating a (110) sample in this gas [Ag-13]. Tellurium segregation from the bulk induced a c(2 × 2) surface structure after heating one sample above 570 K for the first time [Ag-4].

(111) Surface [Ag-4, Ag-14 to Ag-20]. Cycles of inert-gas ion sputtering followed by UHV annealing were commonly used to clean this crystal face. Most authors used argon ions [Ag-4, Ag-14 to Ag-17]; however one did use xenon ions [Ag-18]. Sputtering was carried out with ion energies ranging from 200–500 eV [Ag-4, Ag-14, Ag-18] to 2 keV [Ag-15] and ion currents ranging from 1–3 μA/cm^2 [Ag-14, Ag-18] to 40 μA [Ag-15]. Sample temperature

during bombardment was 300 K [Ag-4, Ag-14], 420 K [Ag-17], or 750 K [Ag-15]. Annealing was performed in UHV at 570 to 900 K. Heating samples in oxygen and then annealing in UHV also produced clean (111) surfaces [Ag-19, Ag-20]. Tellurium segregation to the surface created a $(\sqrt{3} \times \sqrt{3})$-30° structure after the crystal was heated above 570 K for the first time; however, the tellurium was easily removed by argon-ion bombardment [Ag-4].

(331) Surface [Ag-21]. Clean (331) surfaces have been prepared by many repetitions of argon-ion sputtering (300 eV, 1 μA/cm^2, 300 K) and annealing (750 K, UHV). The (311) surface was unreconstructed and was stable to < 900 K, the temperature at which evaporation became significant.

Sodium, Na (bcc, $T_m = 371$ K)

We found no surface studies on bulk sodium. However, pure sodium films have been routinely prepared by evaporation techniques.

Strontium, Sr (fcc; hcp > 488 K; bcc > 878 K, $T_m = 1042$ K)

Since a thick oxide layer formed on polycrystalline strontium after a few seconds of air exposure, the target was mechanically cleaned in a vessel filled with tetrachlorethylene, then inserted into the vacuum system under a nitrogen atmosphere. The surface was cleaned in situ by argon-ion bombardment (total ion dosage: ~ 1 A s/cm^2 at 3 keV) [Sr-1]. Alternatively, a clean surface was generated and maintained by continuous in-situ scraping [Sr-2].

Tantalum, Ta (bcc, $T_m = 3269$ K)

The most frequently found impurities in outgassed tantalum samples were sulfur, carbon and oxygen. Heating tantalum single crystals in vacuum to within 10% of their melting point is reported to produce clean surfaces. However, many groups also used a high temperature treatment in oxygen.

Polycrystalline surface [Ta-1, Ta-2]. Carbon and sulfur were removed from polycrystalline tantalum by heating in oxygen (> 2000 K, 10^{-5} Pa) [Ta-1]. Sulfur, titanium and scandium contaminants were removed by argon-ion bombardment, and carbon was eliminated by heating to > 1400 K in vacuum [Ta-2].

(100) Surface [Ta-3 to Ta-5]. The (100) crystal plane was cleaned by heating alternately to 2800–3000 K and 1800–2000 K for 1 min each [Ta-3, Ta-4]. Another method consisted of heating the crystal to 2300 K, first in vacuum and then in 1.3×10^{-4} Pa oxygen, and finally flashing in UHV (> 2300 K) to remove the last of the oxygen [Ta-5], probably by desorption of oxides.

(110) Surface [Ta-6, Ta-7]. This crystal face has also been cleaned successfully by heating in UHV to ~ 2670 K. It was held at this temperature until the vacuum system reached its base pressure [Ta-6, Ta-7].

Tellurium, Te (hex, $T_m = 723$ K)

Reaction of tellurium with atmospheric oxygen and water vapor is an activated process requiring temperatures in excess of 333 K [Te-1]. Thus it is possible to prepare tellurium surfaces in air using polishing or cleaving techniques, then transfer the crystal quickly to a UHV vacuum system with minimal contamination of the surface by oxygen [Te-1, Te-2]. The main surface impurity was carbon which has been removed by either argon-ion sputtering and annealing or sublimation of the surface layers of the tellurium. (The vapor pressure of tellurium is about 1.3×10^{-4} Pa at 473 K.) Care must be taken when subliming tellurium because some thermal etching has been observed after heating at 443 K for 15 min, and marked thermal etching occurred after heating at 563 K for 30 min [Te-3]. The only polycrystalline surfaces studied were thin films evaporated on substrates.

(0001) Surface [Te-2 to Te-4]. By limiting air exposure to 1/4 h after electropolishing and rinsing, these surfaces were found to be clean to the 0.1 monolayer level for oxygen and to a 0.05 monolayer level for carbon using electron-excited X-ray analysis [Te-2]. A similarly prepared surface was cleaned by heating at 423 K in UHV for short periods without thermally etching the surface [Te-3]. Another electrolytically polished surface did not yield a LEED pattern after heating to 473 K for 1 h, but successive argon-ion sputtering (150 eV, 1 μA/cm², 15 min) and annealing (473 K, 30 min) gave surfaces with well-defined LEED patterns; however, no element-specific analysis was performed to verify the cleanliness [Te-4].

(10$\bar{1}$0) Surface [Te-2 to Te-6]. This surface has been prepared by cleaving in air or in UHV. Cleaving in UHV produced surfaces that gave good LEED patterns [Te-4]. Air-cleaved surfaces were found to have ~0.25 monolayer of crbon and ≤ 0.05 monolayer of oxygen after 15 min of air exposure [Te-2]. The carbon was removed by either heating in UHV (at 423 K [Te-3] or 548 K [Te-5]) for a short time or by repeated argon-ion sputtering (150–250 eV, 1 μA/cm², 1–15 h) and annealing at 473 K from a few minutes to 1 h [Te-4, Te-6]. In one study, the electron energy loss spectrum continued to change after AES indicated a carbon- and oxygen-free surface. The cleaning process was repeated until the energy loss spectrum remained constant and AES continued to indicate a clean surface [Te-6].

(1$\bar{2}$10) Surface [Te-7]. The chemically polished surface was annealed at temperatures for which sublimation was small yet sufficient to remove some impurities. Ion bombardment (550 eV, 2 μA/cm², 1 h) cleaned the surface, and annealing (523 K, 1 h) recrystallized the surface.

Terbium, Tb (hex, $T_m = 1629$ K)

Polycrystalline samples of terbium were found by AES to contain "small amounts" of carbon and oxygen after repeated cycles of argon-ion bombardment and annealing (conditions not specified) [Tb-1].

Thallium, Tl (hcp; bcc > 503 K, $T_m = 577$ K)

Samples were cut from 99.999% pure polycrystalline ingots, and clean surfaces were produced by mechanical scraping at a pressure of less than 10^{-}[illegible] Pa [Tl-1]. Immediately after scraping, the carbon and oxygen 1s photoemission lines could not be detected; but the thallium surface acquired detectable 1s photoemission signals from both carbon and oxygen during a 24 h period in the analyzing chamber maintained at $\sim 10^{-7}$ Pa by an ion pump.

Thorium, Th (fcc; bcc > 1673 K, $T_m = 2023$ K)

The most common impurities found in high purity thorium are sulfur, chlorine, phosphorus, oxygen and carbon. The first three are relatively easy to remove while oxygen and carbon require extensive treatment. Clean polycrystalline and single-crystal surfaces have been produced by argon-ion sputtering followed by annealing at temperatures above 675 K. Annealing temperatures should be kept below 1673 K where a phase transformation from α-Th fcc to β-Th bcc occurs. Once a thorium surface is clean, it is difficult to maintain in this condition due to the rapid chemisorption of CO from residual gases in vacuum systems.

Polycrystalline surface [Th-1, Th-2]. The segregation of impurities (sulfur, carbon and phosphorus) to the surface of bulk polycrystalline thorium has been studied in detail [Th-1]; impurities, present in the bulk in the part per million range, equilibrate rapidly and reproducibly with the surface at elevated temperatures. The surface was found to saturate with sulfur at 1173–1373 K; this sulfur returned to the bulk at 1373–1443 K. Carbon returned to the bulk at ~ 753 K and phosphorus at ~ 1060 K. Extended argon-ion bombardment (0.5–5 keV, 5 μA, 5.2×10^{-3} Pa, 6 h) and a combination of high temperature (400–775 K) and ion bombardment was used to remove carbon and oxygen from thorium [Th-2]. Preparation of clean, annealed thorium surfaces required ion bombardment at temperatures above 673 K.

(100) and (111) surfaces [Th-3 to Th-6]. Cleansing these crystal surfaces of carbon, oxygen and other interstitial contaminants has been accomplished by the following procedure: (a) removal of gross surface contamination by argon-ion bombardment (0.5–4 keV, 10–50 μA/cm², 300 K); (b) bombardment of the sample, beginning at a temperature near 1000 K and continuing during gradual cooling to room temperature (repeat until clean surface is produced); (c) anneal at a temperature near 1000 K for several minutes in a vacuum of less than 10^{-8} Pa. This procedure is also applicable to polycrystalline samples.

Thulium, Tm (hcp, $T_m = 1818$ K)

We found no surface studies on bulk thulium. Thulium has been evaporated to form films.

Tin, Sn (cub; tetr >286 K; rhdr >434 K; $T_m = 505$ K)

Tin is a soft metal requiring special precautions in polishing and mounting. Under certain conditions, tin undergoes a transformation between the cubic and tetragonal phases. This transformation is accompanied by a change in volume and can cause creation of local "warts" on the surface or disintegration of the solid to a coarse powder. Initiation of this transformation in the bulk is difficult to achieve; however, effects in surface studies may be significant.

Polycrystalline surface [Sn-1 to Sn-4] Clean tin surfaces have been produced in situ by micromilling with a rotating diamond edge [Sn-1] or by scraping with a tungsten carbide blade in a preparation chamber at $\sim 10^{-7}$ Pa prior to transfer into the UHV chamber [Sn-2]. Alternately, clean surfaces were prepared from zone-refined, Marz-grade [99.999 + % pure] tin by in-situ argon-ion sputtering (1–2 keV, 20 μA/cm^2). After sputtering, no structure was observed in the tin Auger spectra indicative of the presence of impurities (carbon, oxygen, etc.) up to transition energies of 2000 eV [Sn-3, Sn-4].

(100) Surface [Sn-5]. A (100) single crystal (99.999% purity, β phase) was cleaned by ion sputtering and in-situ annealing. Exposure of the sample to a vacuum of $\sim 3 \times 10^{-8}$ Pa for 10 h resulted in a surface having no detectable Auger signal from impurities and no change from the initial LEED pattern.

Titanium, Ti (hcp; bcc >1155 K, $T_m = 1933$ K)

Titanium is one of the most reactive transition metals. Its ability to decompose simple gases and to form stable compounds with the products has led to the widespread use of freshly deposited titanium films as getters for vacuum pumping. This high reactivity means that titanium is very difficult to clean and to maintain in a clean state; all the studies cited below show traces of residual carbon and oxygen on titanium surfaces. The cleaning of titanium is further complicated by the hcp → bcc phase transition that occurs at ~ 1155 K; thus, single crystal samples must be cleaned at lower temperatures. Sulfur was found to be the most persistant surface contaminant as it tended to segregate on hot surfaces. In many instances traces of sulfur remained on the surface despite efforts to remove it [Ti-1 to Ti-4]. The most effective purification procedures involved sulfur depletion by argon-ion sputtering with the sample at elevated temperatures.

Polycrystalline surface [Ti-4 to Ti-7]. Simultaneous argon-ion bombardment and annealing at 1070 K were found to result in a surface free from sulfur in one case [Ti-6] but not in another [Ti-4]; slight amounts of carbon and oxygen contamination were detected in each case. Argon-ion sputtering at 300 K coupled with brief anneals was reported to produce a sulfur-free surface, but extended annealing caused the sulfur to reappear [Ti-7]. Argon-ion etching (600 eV, 10 μA/cm^2) coupled with annealing at 1020 K reduced, but did not eliminate, sulfur and chlorine contamination [Ti-5].

(0001) Surface [Ti-1 to Ti-3, Ti-8 to Ti-10]. The most successful procedure for the cleaning of the basal plane of titanium consisted of a cycle of sputtering with argon ions (600 eV, 4 μA/cm^2, 300 K) and annealing in UHV at 1020 K until 50 h of sputtering time was accumulated; this cycle was followed by sputtering with argon ions (600 eV, 4 μA/cm^2, 1020 K, ~ 14 h) and annealing in UHV (1020 K, 4 h) [Ti-8, Ti-9]. Subsequent recleaning was achieved by sputtering with argon ions at 300 K (600 eV, 30 min) or at 1020 K (500 eV, 1.2 h) followed by annealing (1020 K, 1 h). One author was able to obrain a sulfur-free (0001) surface by cycles of ion sputtering and annealing [Ti-10], while others were not [Ti-1 to Ti-3].

(10$\bar{1}$0) and (10$\bar{1}$1) surfaces [Ti-11, Ti-12]. One author generated a clean (10$\bar{1}$0) surface by argon-ion etching at 970–1070 K [Ti-11] while another achieved the same result on the (10$\bar{1}$1) plane by: (a) argon-ion bombardment; (b) annealing at 820 K (several hours); (c) repeating (a) and (b) until no impurities could be detected by XPS [Ti-12].

Tungsten, W (bcc, T_m = 3683 K)

Carbon, which originates in the bulk and segregates at the surface when tungsten is heated, is the most difficult contaminant to remove. The two most widely used techniques for the initial cleaning of a tungsten surface are (1) prolonged heating at a high temperature in UHV, and (2) reaction with oxyen to remove the carbon in the form of CO [W-1], followed by flashing at high temperature in UHV to desorb the oxygen as tungsten oxides. The effectiveness of the oxygen–anneal treatment varied considerably among crystals from different suppliers [W-2]. Also, there has been evidence that the anneal step removed pits that were formed at the surface during the oxygen treatment [W-2].

Polycrystalline surface [W-3 to W-14], Repeated flashings or prolonged heating to > 2500 K in UHV have yielded clean surfaces in some instances [W-1, W-10 to W-12]. However, in most studies such simple heating procedures have not yielded atomically clean surfaces. In fact, heating for up to 10 min at 3000 K in UHV did not remove carbon once it had segregated at the surface [W-7]. The equilibrium concentration of carbon segregated at the surface was a function of temperature, but carbon dissolved into the bulk above 2200 K [W-4]. The carbon concentration was quite large at temperatures of 1500–1800 K; efficient removal of the carbon from the sample could be effected by reaction at these temperatures with oxygen at ~ 10^{-5} to ~ 10^{-4} Pa. The tungsten oxides remaining on the surface were removed by one of the three procedures: (1) flashing to above 2400 K in UHV [W-6 to W-9, W-13], (2) heating in 7×10^{-5} Pa hydrogen for 2 min at 2200 K [W-4], or (3) sputtering with argon ions followed by anneals at 1800 K [W-14].

(100) Surface [W-2, W-9, W-15 to W-39]. Some evidence exists that prolonged heating at 2200–2500 K followed by flashing to 2800–3000 K yields

clean (100) surfaces [W-15, W-17, W-21]; however, in most cases the oxygen-anneal treatment described for the polycrystalline surfaces was required to initially prepare a clean surface [W-9, W-16, W-18, W-19, W-22 to W-39]. A maximum in carbon segregation onto the surface has been reported to occur at 1500 ± 100 K [W-27], which is consistent with results on polycrystalline tungsten. The ineffectiveness of the oxygen-anneal treatment has been encountered in one study [W-20]; twenty cycles of sputtering, using krypton ions on the sample at 1300 K with flashes to 2500 K between 1 h bombardments, were required to remove the carbon.

(110) Surface [W-1, W-28, W-34, W-39 to W-49, W-56]. Heating these surfaces to high temperatures in UHV is not sufficient treatment for the generation of clean surfaces. The (110) and vicinal surfaces on the same samples have been cleaned by the oxygen-anneal treatment detailed for the polycrystalline surfaces [W-42, W-43, W-47]. A sputter-anneal treatment has also been reported to be effective in cleaning these surfaces [W-41].

(111) Surface [W-39, W-50, W-51]. Carbon was removed successfully from these surfaces by heating in UHV at 2500 K [W-51]. However, the oxygen-anneal treatment was preferred [W-39, W-50].

(112) Surface [W-48, W-52, W-54]. This face has been cleaned by either sputter-anneal or oxygen-anneal treatments as outlined above.

Uranium, U (orth; tetr >941 K; bcc > 1048 K; T_m = 1405 K)

All the clean-surface studies reviewed were performed using polycrystalline material. Interstitial impurities (i.e. sulfur, chlorine, phosphorous, oxygen and carbon) have been the most common contaminants, but small amounts of calcium and iron were also commonly present. The most difficult contaminants to remove were carbon and oxygen. Heating to 1073 K in UHV was not sufficient to eliminate surface impurities [U-1 to U-4]. Many investigators found that uranium could be cleaned in two stages: (a) argon-ion sputtering (0.5–5 keV, 10–40 μA/cm^2, 300 K, 1–3 days), and (b) cycles of argon-ion sputtering at high temperatures (300–850 K) and annealing in UHV (800–1170 K) [U-5 to U-7]; however, this procedure was usually repeated several times. One group created a clean surface by argon-ion bombardment alone (5 keV, 10 μA/cm^2, 300 K, several h) [U-8]. In another study, a small amount of iron (~0.1 monolayer) was observed to segregate to the surface upon cooling from 850 to 300 K; this was removed by a gentle argon-ion bombardment (500 eV, 1 μA/cm^2, 300 K, 15–30 s) [U-7]. A different procedure consisted of a chemical treatment in oxygen (10^{-5} Pa, 1070 K, 30 min) and subsequent reduction in hydrogen (10^{-5} Pa, 1070 K, 10 min); this procedure effectively removed phosphorous and sulfur but some oxygen remained [U-9]. Clean surfaces have also been produced by in-situ abrasion with a diamond file [U-10]. For single crystal α uranium, we recommend the two-stage sputter-annealing cycle outlined above with the sample temperature never exceeding

850 K during annealing (avoids the orthorhombic to tetragonal phase transition at ~ 941 K). Maintaining a clean surface on uranium has been difficult because of rapid chemisorption of carbon monoxide, a residual gas in the UHV system.

Vanadium, V (bcc, $T_m = 2163$ K)

Inert-gas ion bombardment at high temperatures has been necessary to produce clean vanadium surfaces on single crystals of nominal 99.99% purity [V-1, V-2]. A majority of the investigators reporting on this metal were unable to produce surfaces free of oxygen, sulfur, or carbon [V-3 to V-10]; these elements were suspected of stabilizing the (100)-(1 × 1) structure. Interpretation of the Auger spectrum of vanadium is complicated by the overlap of the vanadium LVV transition at ~ 509 eV with the oxygen KVV at ~ 512 eV.

(100) Surface [V-1, V-5 to V-9] A clean (100) surface was obtained after approximately 200 h of neon-ion bombardment with the sample at 800 K; a (5 × 1) restructured surface was produced. The (5 × 1) structure undergoes a reversible phase transition to the normal (1 × 1) structure at 630 K [V-1]. Previous LEED investigations [V-5 to V-9] reported normal (1 × 1) structures but also reported significant amounts of sulfur on the surface which may have stabilized this structure.

(110) Surface [V-2, V-10]. Clean (110) surfaces have been prepared by sputtering with argon ions (2 keV, 20 μA/cm^2, 670 K, ~ 80 h), and annealing in UHV (970–1070 K). Shortened sputter–anneal cycles were repeated until evidence of oxygen segregation (by AES or the appearance of the oxygen induced (6 × 2) LEED pattern) was not observed after prolonged anneals [V-2].

Ytterbium, Yb (fcc, $T_m = 1092$ K)

The only procedure documented to yield clean surfaces on bulk polycrystalline ytterbium was mechanical removal of a macroscopic surface layer in UHV with a tungsten carbide blade [Yb-1]. Argon-ion sputtering and annealing have been tried but some oxygen was left on the surface [Yb-2]. However, repeated cycles of this last procedure should eventually lead to a clean surface.

Yttrium, Y (hcp, $T_m = 1795$ K)

The major contaminants found in yttrium foil samples were sulfur, carbon, chlorine and oxygen [Y-1]. These could be removed by argon-ion bombardment, but chlorine always reappeared on the surface after annealing [Y-2]. One author found yttrium to be very reactive towards oxygen and reported that it was not possible to clean this metal by argon-ion etching (900 eV, 11 μA/cm^2) alone [Y-3].

Zinc, Zn (hcp, $T_m = 693$ K)

The low melting point of zinc (~690 K) coupled with its high vapor pressure (~ 10^{-5} Pa at 420 K) cause it to be incompatible with accepted UHV bake-out procedures. This problem was circumvented by electroplating a zinc single crystal with a heavy nickel coating which prevented evaporation even at bake-out temperatures of 520 K. The crystal was cleaved after the bake-out was terminated and after the vacuum system reached its base pressure [Zn-1].

Polycrystalline surface [Zn-2 to Zn-7]. Treatments using argon-ion etching (900–1500 eV, ~200 μA/cm^2, 300 K) were sufficient to produce clean zinc surfaces.

(0001) Surface (Zn-1, Zn-8 to Zn-11). The basal plane of zinc is commonly prepared by cleaving in UHV [Zn-1, Zn-8, Zn-9] or air [Zn-9]. In the latter case, argon-ion bombardments was necessary to remove the surface oxide layer [Zn-9 to Zn-11]. The cleaving itself has been done at both ambient temperature [Zn-1, Zn-8] or at 77 K [Zn-9]. Annealing at 390–425 K was reported to produce a well-ordered surface as determined by LEED [Zn-9].

Zirconium, Zr (hex, $T_m = 2125$ K)

Zirconium is an efficient gettering agent for common gases and, in this respect, is chemically similar to titanium. Consequently, even zone-refined material contains appreciable levels of oxygen, carbon and sulfur. Segregation of these bulk impurities to the surface during heating constitutes the most troublesome aspect of cleaning zirconium surfaces. This problem is further complicated by the high solubility for oxygen (29 at% at 700 K), which increases with temperature.

Polycrystalline surface [Zr-1 to Zr-4]. After insertion into the vacuum system and brief heating to 900 K the Auger spectrum showed that the polycrystalline surface was contaminated with sulfur, chlorine, nitrogen and oxygen. Sputter cleaning (1 μA/cm^2, 500 eV, 300 K, 1 h) produced a clean surface, but subsequent annealing led to the reappearance of sulfur, presumably due to diffusion from the bulk. It is difficult to ascertain the completeness of sulfur removal using AES because there is a significant overlap of the sulfur transition at 150 eV and the zirconium transitions at 145 eV. Argon-ion sputtering at 900 K for 10 h reduced the sulfur level to the extent that the ratio of the 145 eV Auger peak (Zr + S) to the 95 eV peak (Zr only) reached a leveling value of 1.3 [Zr-1]. Other authors, who did not anneal in situ but cleaned by argon-ion bombardment only [Zr-2] or by in-situ micro-milling with a rotating diamond edge [Zr-3], easily eliminated the sulfur. The latter mentioned that it was not possible to keep the zirconium surface free of oxygen at a pressure of 10^{-7} Pa in the analyzing chamber.

(0001) Surface [Zr-5]. An Auger spectrum taken after an initial anneal at 873 K revealed large quantities of carbon and oxygen together with smaller amounts of nitrogen, boron (179 eV) and/or chlorine (181 eV). The carbon contamination proved most difficult to remove; it could not be reduced below

detectable limits. The cleanest surface obtained corresponded to an Auger peak ratio C_{274}/Zr_{170} of around 0.05 to 0.1. It was achieved after approximately 50 h of argon-ion bombardment at room temperature followed by a number of cycles of bombardment at 823–873 K for several hours and 30 min anneals at the same temperature. The annealing temperature of 823–873 K appeared to be optimal because more carbon segregated to the surface at both higher and lower temperatures.

3. Discussion and recommendations

Assessment of the information presented in the review section leads to several observations. Carbon, oxygen and sulfur were most often the difficult impurities to remove from elemental surfaces. Considering the number of elements affected, the relative importance of these impurities was C:O:S = 6:4:3 with carbon being a key impurity for thirty of the reviewed elements. Often, more than one of these impurities proved to be the main barriers to achieving a clean surface. Generally, carbon and sulfur contamination resulted from segregation of bulk impurities to the surface during heating; whereas the troublesome source of oxygen contamination was adsorption of oxygen-bearing gases during, and immediately after, the cleaning process. Elimination of carbon and sulfur was usually accomplished by heating in a reactive gas or heating during ion bombardment. The heating ensured a continuous flow of the impurities from the bulk to the surface, and the reaction and bombardment removed the impurities from the surface; thus, the bulk impurities were steadily depleted. Ion bombardment was generally used to remove oxygen, but repeated flashings in UHV to temperatures sufficient to desorb oxides were sometimes effective.

The reviewed information, tempered by our own knowledge and experience in surface cleaning, was condensed into a set of recommended procedures (table 1). These recommendations have been arranged alphabetically by element with differences for crystallographic planes detailed wherever appropriate. The procedures recommended are those we would use if faced with the need to prepare a clean surface of a particular element. Generally, the selected procedures are a consensus of the reviewed literature; however, possibilities for widespread implementation of the procedure were also considered. Careful adherence to the details of a recommended procedure does not per se ensure the creation of an atomically clean surface. Surface analysis with an element-specific technique is still required to determine the level of surface cleanliness.

Several aspects of the entries in table 1 deserve comment. Since most of these elements can be evaporated in UHV to form films with clean surfaces, evaporation is listed as a recommended procedure only if a procedure was not found for bulk specimens. Specific ion energies and current densities for the ion bombardment steps should be considered nominal values, because sputtering phenomena are not strong functions of these parameters in the ranges of interest. Unless otherwise specified, ion bombardment conditions should consist of an energy of approximately 1 keV, a current density of a few $\mu A/cm^2$, and ambient temperaure. All unspecified annealing temperatures can be assumed to be approximately two-thirds of the melting-point temperature (K) for the elements. All annealings and heatings should be performed under UHV conditions, except where contrary specifics have been given.

A variety of conclusions can be drawn from the information presented in table 1. As expected, repeated cycles of ion bombardment and annealing were recommended more often than any other type of procedure; in fact, such cycles were recommended for 39 of the 54 elements having procedures for bulk specimens. Heating in reactive gas(es) and annealing in UHV was the recommendation for four elements (Ir, Mo, Re, W). For five other elements (Ni, Pd, Pt, Rh, Ru) a combination of bombardment-annealing cycles and reaction-annealing cycles were recommended. Only four elemental surfaces (As, C [graphite and diamond], Nb, Ta) have been unambiguously cleaned by simply heating in UHV. In-situ scraping was the recommended procedure for two elements (Li, Tl); however, cycles of ion bombardment and annealing would probably yield clean surfaces of these elements. Unfortunately, we did not find any documented surface studies on bulk specimens for twenty of the elements reviewed. Nevertheless, application of the procedure recommended for chemically similar elements should produce atomically clean surfaces for these twenty elements.

Only one clearly systematic classification has been noted from table 1. All elements having gas reaction-annealing cycles as part of the recommended procedure are grouped near the center of the periodic table of elements. Osmium was the only reviewed element within the grouping for which reaction-annealing cycles were not included in the recommended procedure. Considering osmium's location within the periodic table and the fact that the recommended procedure was based on only one reference, the preferred method for cleaning osmium should probably include reaction-annealing cycles.

Although a variety of procedures may eventually produce a clean surface on those elements for which we did not list a recommended procedure, we suggest the following strategy for cleaning bulk specimens in the absence of a recommended procedure. Begin by outgassing the sample at temperatures slightly below the melting point. Using the nominal ion-bombardment conditions given above (1 keV, few $\mu A/cm^2$, 300 K), sputter with argon ions until in-situ element-specific analysis reveals that the surface is atomically clean. Anneal the sample at two-thirds of its melting point while monitoring the surface composition. If any contaminant (e.g., carbon or sulfur) segregates on the surface, determine the temperature corresponding to the maximum concentration of contamination. Then incorporate this temperature into the ion-bombardment conditions and repeat the bombardment-annealing cycle until the annealed surface is atomically clean.

Table 1
Recommended surface cleaning procedures

Element	Surface planes	Recommended procedure
Actinum (Ac)	–	No information found
Aluminum (Al)	Poly	Outgas in UHV (673 K, 48 h); repeat cycles of Ar$^+$ bombardment (5 keV, 1.6 μA/cm^2, 300–500 K, 10 h first time then 30 min) and annealing (673 K, 30 min)
	(100), (110), (111)	Outgas in UHV (673 K, 48 h); repeat cycles of Ar$^+$ bombardment ($\leq$2 keV, 5 μA/cm^2, $\leq$673 K, 15 min) and annealing ($\leq$700 K, 1 h)
Americium (Am)	–	No information found
Antimony (Sb)	(0001)	Cleave under UHV
	(0001), (01$\bar{1}$2), (11$\bar{2}$0)	Repeat cycles of Ar$^+$ bombardment (200 eV, few μA/cm^2, 300 K, few min) and annealing (520 K, few h)
Arsenic (As)	(0001)	Heat air-cleaved crystal in UHV (493 K)
	(111)	Cleave under UHV
Barium (Ba)	–	Evaporate in UHV to form films
Beryllium (Be)	Poly, (0001)	Repeat cycles of Ar$^+$ bombardment (500 eV, few μA/cm^2, 300 K, 1–2 h) and annealing (1000 K, 1 h)
Bismuth (Bi)	(0001), (01$\bar{1}$2) [a], (11$\bar{2}$0) [a]	Bombard with Ar$^+$ (150–300 eV, 1–5 μA/cm^2, 6 h) and anneal (510–520 K)
Boron (B)	Poly	Repeat cycles of Ar$^+$ bombardment and annealing (< 1600 K)
	β(100), β(111)	Heat (1723 K, 1 min)
Cadmium (Cd)	Poly	Repeat cycles of Ar$^+$ bombardment (3 keV)
	(0001)	Bombard with Ar$^+$ (700 eV, 3 μA/cm^2, 295 K, 24 h)
Calcium (Ca)	–	Evaporate in UHV to form films
Carbon (C)	Amorphous	Evaporate in UHV to form films
	Glassy	Fracture under UHV
	Graphite (0001)	Cleave in air; place immediately into ion- and electron-free

		vacuum system; anneal (723 K, 5 h); anneal ($T >$ 1273 K) if ion conc. is high
	Graphite (10$\bar{1}$0), (11$\bar{2}$0)	Fracture under UHV
	Diamond (100), (110), (111)	Heat in UHV (1173–1573 K, 10 min)
Cerium (Ce)	Poly	Repeat cycles of inert-gas ion bombardment (1 keV, 10 $\mu A/cm^2$) and annealing
Chromium (Cr)	(100)	Repeat cycles of Ar^+ bombardment (500 eV, 3–5 μA) and annealing (900 K)
	(110)	Repeat cycles of Ar^+ bombardment (500 eV, 25 μA, 300 K, 30 min) and annealing (670–870 K, 15 min)
	(111)	Repeat cycles of Ar^+ bombardment (2 keV, 5 $\mu A/cm^2$, 300 K) and annealing (1173 K)
Cobalt (Co)	Poly	Heat in oxygen (900 K, 10^{-4} Pa); repeat cycles of Ne^+ bombardment (530 eV, 1 $\mu A/cm^2$, 600 K) and annealing (1000 K, 15 h)
	(100)	Repeat cycles of Ar^+ bombardment (150 eV, 1 $\mu A/cm^2$, 420 K) and annealing (420 K)
	(0001), (10$\bar{1}$0), (10$\bar{1}$2)	Repeat cycles of Ne^+ bombardment (500 eV, 1 $\mu A/cm^2$, 600 K) and annealing (650–800 K, very brief)
Copper (Cu)	Poly [a], (100), (110), (111), (311)	Repeat cycles of Ar^+ bombardment (600 eV, few $\mu A/cm^2$, 300 K) and annealing (723 K)
Dysprosium (Dy)	–	Evaporate in UHV to form films
Erbium (Er)	–	Evaporate in UHV to form films
Europium (Eu)	–	Evaporate in UHV to form films
Gadolinium (Gd)	Poly	Repeat cycles of inert-gas ion bombardment (1 keV, 10 $\mu A/cm^2$) and annealing
Gallium (Ga)	Liquid	Repeat cycles of Ar^+ bombardment (3 keV, 20 $\mu A/cm^2$) and heating (600 K)
Germanium (Ge)	Poly	No information found
	(100), (110)	Repeat cycles of inert-gas ion bombardment (500 eV) and annealing (1073 K)
	(111)	Cleave in UHV

Table 1 (continued)

Element	Surface planes	Recommended procedure
Gold (Au)	Poly (Ca-free)	Heat in UHV (1000 K, 6 h) and in oxygen (1000 K, 6×10^{-3} Pa, 8–24 h) and anneal (1000 K)
	Poly, (100), (111), vicinals	Repeat cycles of inert-gas ion bombardment (340 eV, 3 μA, 300 K, 2–10 h) and annealing (≤973 K)
Hafnium (Hf)	Poly	Repeat cycles of Ar$^+$ bombardment (500 eV, 1 μA/cm^2, 1070–1370 K) and annealing (1370 K)
Holmium (Ho)	Poly	Repeat cycles of inert-gas ion bombardment (1 keV, 10 μA/cm^2) and annealing
Indium (In)	Poly	Repeat cycles of Ar$^+$ bombardment of liquid (>430 K) and solidification
Iridium (Ir)	(100)-5×1	Repeat cycles of heating in oxygen (1400–1500 K, $\sim 10^{-5}$ Pa, 5–10 min) and annealing (1500 K, 1 min)
	(100)-1×1	Generate clean (5×1) surface structure; heat in oxygen (475 K, $\sim 10^{-5}$ Pa, 200–400 s) and anneal (660 K); heat in hydrogen (400–700 K, $\sim 10^{-5}$ Pa, 300 s) or in carbon monoxide (400 K, $\sim 10^{-5}$ Pa, 300 s); anneal (800 K)
	(110)-2×1, (111)	Bombard with Ar$^+$ to remove initial contamination; repeat cycles of heating in oxygen (800 K, $\sim 10^{-5}$ Pa) and annealing (1200–1600 K)
	(755)	Repeat cycles of heating in oxygen (770–870 K, $\sim 10^{-5}$ Pa, several min) and flash annealing (1470 K)
Iron (Fe)	Poly, (100), (110), (111)	Bombard with Ar$^+$ (500 eV, 1–10 μA/cm^2, 300 K, 1–150 h) to remove oxide layer; repeat cycles of Ar$^+$ bombardment (500 eV, 20 μA/cm^2, 300 or 650 K, 15–30 min) and annealing (450–700 K, ~1 h); flash anneal (970 K)
Lanthanum (La)	Poly	Repeat cycles of Ar$^+$ bombardment (900 eV, 10 μA) and annealing
Lead	Poly, (100), (110)	Repeat cycles of Ar$^+$ bombardment (700 eV, 5–10 μA, several h) and annealing (430 K)
Lithium (Li)	Poly	Scrape bulk sample in UHV; bombard with Ar$^+$ (1 keV, 5 μA/cm^2, 300 K)

Lutetium (Lu)	-	Evaporate in UHV to form films
Magnesium (Mg)	Poly	Repeat cycles of Ar^+ bombardment and annealing
Manganese (Mn)	-	Evaporate in UHV to form films
Molybdenum (Mo)	Poly, (100), (110), (111), (112)	Repeat cycles of heating in oxygen (~1500 K, ~10^{-4} Pa, 3-6 h) and flash annealing (2300 K)
Neodymium (Nd)	Poly	Repeat cycles of inert-gas ion bombardment (1 keV, 10 $\mu A/cm^2$) and annealing
Neptunium (Np)	-	No information *found*
Nickel (Ni)	Poly, (100), (110), (111)	Repeat cycles of Ar^+ bombardment (500 eV, 5 $\mu A/cm^2$, 300 K, 15 *min)* and annealing (900 K, 15 min) until surface composition stabilizes; heat in oxygen (900 K, 10^{-4} Pa); heat in hydrogen (1000 K, 10^{-3} Pa); anneal (900 K, 15 min)
Niobium (Nb)	Poly, (100), (110), (111)	Heat in UHV (2300 K, several h)
Osmium (Os)	Poly	Repeat cycles of Ar^+ bombardment (2 keV, 5 $\mu A/cm^2$) and annealing flashes (1670 K)
Palladium (Pd)	Poly, (100), (110), (111), (210), (311)	Repeat cycles of inert-gas ion bombardment (~500 eV, 5 $\mu A/cm^2$, 900 K, ~300 h per mm thickness) and annealing (1100-1300 K); *heat in oxygen* (>1000 K, ~10^{-4} Pa) and flash anneal (1300 K)
Phosphorus (P)	-	No *information* found
Platinum (Pt)	Poly, (100), (110), (111), (112), (113), (133), (122), (012)	Repeat cycles of Ar^+ bombardment (~400 eV), heating in oxygen (1000-1300 K, ~10^{-4} Pa), and annealing (1300 K)
Plutonium (Pu)	Poly	Bombard with inert-gas ions (0.5-5 keV, 10-50 $\mu A/cm^2$) to remove gross contamination; repeat cycles of inert-gas ion bombardment (676 K) and annealing (676 K, <10^{-8} Pa, several min)
Polonium (Po)	-	No information found
Praseodymium (Pr)	-	Evaporate in UHV to form films
Promethium (Pm)	-	No information found

Table 1 (continued)

Element	Surface planes	Recommended procedure
Protactinium (Pa)	–	No information found
Radium (Ra)	–	No information found
Rhenium (Re)	Poly, (0001)	Repeat cycles of heating in oxygen (2000–2500 K, 10^{-3} Pa) and annealing (2200 K, 2 min)
Rhodium (Rh)	(100), (111), (775), (331)	Bombard with inert-gas ions (0.5–1 keV, 1–10 $\mu A/cm^2$, 300 K) to remove gross contamination; anneal (1250–1300 K); repeat cycles of Ar^+ bombardment (500 ev, 1–10 $\mu A/cm^2$, 300 K), heating in oxygen (700–1300 K, 10^{-5}–10^{-4} Pa), and annealing (900–1300 K, few min); flash anneal [1300 K for (100) and (111), 1070 K for (331) and (775)]
Ruthenium (Ru)	(0001), (110)	Repeat cycles of heating in oxygen (1300–1500 K, $\sim 10^{-5}$ Pa, 10–15 s) and annealing (1500–1600 K, 10–60 s)
	$(10\bar{1}0)$	Repeat cycles of Ar^+ bombardment (500 eV, 5 $\mu A/cm^2$, 1270 K, 5 min) and annealing (1270 K)
Samarium (Sm)	Poly	Repeat cycles of inert-gas ion bombardment (1 keV, 10 $\mu A/cm^2$) and annealing
Scandium (Sc)	Poly, (100), (001)	Repeat cycles of Ar^+ bombardment (500 eV, few $\mu A/cm^2$, 850 K) and annealing (1100 K) [a]
Selenium (Se)	–	No information found
Silicon (Si)	Poly	Ar^+ bombardment (2.5 $\mu A/cm^2$, 10 s)
	(100), *(110)*, *(100)* vicinals	Repeat cycles of Ar^+ bombardment (350–500 eV) and annealing (1200–1400 K, 2–30 min)
	(111)	Flash anneal (1473–1523 K)
Silver (Ag)	Poly, (100), (110), (111), (331)	Repeat cycles of Ar^+ bombardment (300–600 eV, 1–3 $\mu A/cm^2$, 300 K) and annealing (670–750 K)
Sodium (Na)	–	Evaporate in UHV to form films
Strontium (Sr)	Poly	Repeat cycles of Ar^+ bombardment (3 keV, 1 A s/cm^2) and annealing

Tantalum (Ta)	Poly, (100), (110)	Heat in UHV (2700–3000 K)
Tellurium (Te)	(0001), $(1\bar{2}10)$, air-cleaved $(10\bar{1}0)$	Repeat cycles of Ar^+ bombardment (~300 eV, ~2 $\mu A/cm^2$, ~30 min) and annealing (470 K, 1 h)
	$(10\bar{1}0)$	Cleave in UHV
Terbium (Tb)	Poly	Repeat cycles of inert-gas ion bombardment (1 keV, 10 $\mu A/cm^2$) and annealing
Thallium (Tl)	Poly	Scrape mechanically
Thorium	Poly, (100), (111)	Bombard with inert-gas ions (0.5–5 keV, 10–50 $\mu A/cm^2$) to remove gross contamination; repeat cycles of inert-gas ion bombardment (1000 K) and annealing (1000 K, < 10^{-8} Pa, several min)
Thulium (Tm)	–	Evaporate in UHV to form films
Tin (Sn)	Poly, (100)	Repeat cycles of Ar^+ bombardment (1–2 keV, 20 $\mu A/cm^2$) and annealing
Titanium (Ti)	Poly, (0001), $(10\bar{1}0)$	Repeat cycles of Ar^+ bombardment (600 eV, 4 $\mu A/cm^2$, 300 K) and flash annealing (1020 K) until 50 h of bombardment are accumulated; bombard with Ar^+ (600 eV, 4 $\mu A/cm^2$, 1020 K, ~14 h); anneal (1020 K, 1 h); repeat cycles of Ar^+ bombardment (600 eV, 4 $\mu A/cm^2$, 300 K for 1/2 h and 1020 K for 1/2 h) and annealing (1020 K, 1 h)
Tungsten (W)	Poly, (100), (110), (110) vicinals, (111), (112), (210)	Heat in oxygen (1550 K, ~7×10^{-5} Pa, 100–300 h per mm thickness); repeat flash anneals (>2400 K)
Uranium (U)	Poly	Bombard with inert-gas ions (0.5–5 keV, 10–50 $\mu A/cm^2$) to remove gross contamination; repeat cycles of inert-gas ion bombardment (1000 K) and annealing (1000 K, < 10^{-8} Pa, several min)
Vanadium (V)	(100)-5×1 (110)	Ne^+ bombardment (500 eV, 5 $\mu A/cm^2$, 800 K, 200 h) Ar^+ bombardment (2 keV, 20 $\mu A/cm^2$, 670 K, 80 h); anneal (970–1070 K); repeat cycles of Ar^+ bombardment (2 keV, 20 $\mu A/cm^2$, 670 K, 5 *min*) and annealing (970–1070 K)

Table I (continued)

Element	Surface planes	Recommended procedure
Ytterbium	Poly	Repeat cycles of Ar^+ bombardment and annealing [a]
Yttrium (Y)	Poly	Repeat cycles of Ar^+ bombardment (500 eV, 5 $\mu A/cm^2$) and annealing
Zinc (Zn)	Poly	Repeat cycles of Ar^+ bombardment (1 keV, 20 $\mu A/cm^2$, 300 K) and annealing
	(0001)	Cleave in UHV
	Air-cleaved (0001)	Repeat cycles of Ar^+ bombardment (1 keV, 20 $\mu A/cm^2$, 300 K) and annealing (390–425 K)
Zirconium (Zr)	Poly	Bombard with Ar^+ (500 eV, ~1 $\mu A/cm^2$, 300 K, 1 h) to remove gross contamination; repeat cycles of Ar^+ bombardment (900 K, 10 h) and annealing
	(0001)	Bombard with Ar^+ (300 K, 50 h); repeat cycles of Ar^+ bombardment (~850 K, several h) and annealing (~850 K, 30 min)

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[a] Procedure has not been documented by element-specific techniques to produce atomically clean surfaces.

4. Concluding remarks

Two remarks are appropriate with regard to this review. First, a need exists for documented cleaning procedures for more than twenty elements. The recommended procedures for these elements have been listed in table 1 as "no information found" or "evaporate in UHV to form films". We would be most pleased to receive copies of published papers that may help eliminate the information-gap in an up-dated version of this review. Second, we strongly suggest that future authors of surface studies provide either (1) details of their cleaning methods, including some measure of the degree of surface cleanliness from an element-specific analysis technique, or (2) reference to another paper that did provide details and documentation. The level of detail should be sufficient to permit reproduction of the surface conditions employed. A useful qualitative measure of surface cleanliness is specification of the peak amplitudes of the contaminants relative to those for the element, under given analysis conditions. Both these remarks are consistent with achievement of orderly scientific progress in the surface science of the elements.

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