



# Standard Guide for Minimizing Unwanted Electron Beam Effects in Auger Electron Spectroscopy<sup>1</sup>

This standard is issued under the fixed designation E 983; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This guide outlines the origins and manifestations of unwanted electron beam effects in Auger electron spectroscopy (AES).

1.2 Some general guidelines are provided concerning the electron beam parameters which are most likely to produce these effects.

1.3 General classes of materials are identified which are most likely to exhibit unwanted electron beam effects. In addition, a tabulation of some specific materials which have been observed to undergo electron damage effects is provided.

1.4 A simple method is outlined for establishing the existence and extent of these effects during routine AES analysis.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

E 673 Terminology Relating to Surface Analysis<sup>2</sup>

E 996 Practice for Reporting Data in Auger Electron Spectroscopy and X-Ray Photoelectron Spectroscopy<sup>2</sup>

## 3. Terminology

3.1 See Terminology E 673 for terms used in Auger electron spectroscopy.

NOTE 1—Electron beam effects and their consequences are widely referred to in the literature using any one or more of the following terms: electron beam damage, sample damage, specimen damage, beam effects, electron beam induced processes, and electron irradiation effects.

## 4. Significance and Use

4.1 When electron beam excitation is used in AES, the incident electron beam can interact with the specimen material causing physical and chemical changes. In general, these

effects are a hindrance to AES analysis because they cause localized specimen modification **(1, 2, 3)**.<sup>3</sup>

4.2 With specimens that have poor electrical conductivity the electron beam can stimulate the development of localized charge on the specimen surface. This effect is a hindrance to AES analysis because the potentials associated with the charge can either adversely affect the integrity of Auger data or make Auger data collection difficult.

## 5. Origins of Electron Beam Effects

5.1 Electron beam effects in AES may originate from one or more distinct processes.

5.1.1 Charge accumulation in materials with poor electrical conductivity leading to potentials that cause distortion of Auger data or make AES data collection difficult by virtue of:

5.1.1.1 Auger peak shift on energy scale.

5.1.1.2 Auger peak shape and size distortion.

5.1.1.3 Auger signal strength instability.

5.1.2 Electronic excitation of surface, subsurface, and bulk atoms and molecules leading to specimen changes **(4, 5, 6)** which include:

5.1.2.1 Dissociation.

5.1.2.2 Electron stimulated desorption (ESD) **(7)**.

5.1.2.3 Electron stimulated adsorption (ESA) **(8)**.

5.1.2.4 Polymerization **(9, 10)**.

5.1.2.5 Carburization **(11, 12, 13)**.

5.1.2.6 Oxidation **(14, 15)**.

5.1.2.7 Reduction **(16)**.

5.1.2.8 Decomposition **(17, 18)**.

5.1.2.9 Erosion.

5.1.2.10 Diffusion.

5.1.3 Charge accumulation in materials of poor electrical conductivity leading to specimen changes which include **(19, 20)**:

5.1.3.1 Electric field enhanced diffusion.

5.1.3.2 Electromigration.

5.1.4 Heating which may cause:

5.1.4.1 Annealing.

5.1.4.2 Segregation.

5.1.4.3 Volatilization.

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee E-42 on Surface Analysis and is the direct responsibility of Subcommittee E42.03 on Auger Electron Spectroscopy and XPS.

Current edition approved Sept. 15, 1994. Published November 1994. Originally published as E 983 – 84. Last previous edition E 983 – 89.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 03.06.

<sup>3</sup> The boldface numbers in parentheses refer to the references listed at the end of this standard.

#### 5.1.4.4 Chemical reaction.

### 6. Practical Manifestations of Electron Beam Effects

6.1 Electron dose dependent changes in the intensity, energy, peak shape of one or more Auger transitions; depending upon the material, these changes may be complete within a fraction of a second or they may progress for hours.

6.2 Discoloration of the specimen at the electron beam irradiated region.

6.3 Physical damage to the specimen such as erosion, cracking, blistering, or densification.

6.4 Pressure rises in the analytical vacuum chamber during electron irradiation.

6.5 Localized electric charge dependent changes in the intensity, energy, or peak shape of all Auger transitions. These changes may be stable but often are erratic resulting in unstable AES signals which may preclude AES data collection.

### 7. Electron Beam Parameters

#### 7.1 Electron Dose and Current Density:

7.1.1 Electron dose and current density were previously defined using units of  $C/cm^2$  and  $mA/cm^2$ , respectively. These units are not consistent with the SI system. To keep from changing the magnitude of the numbers appearing in the literature (from which Table 1 is adapted), the multipliers of the terms are being changed. A dose of  $C/cm^2$  is equivalent to  $10^4 C/m^2$ , while  $1mA/cm^2$  is equivalent to  $10A/m^2$ .

7.1.2 Specimen material modification can often be related to the electron dose ( $D$ ); that is, the number of electrons incident on a unit area of the specimen, expressed in coulombs per square centimeter ( $C/cm^2$ ) (1).

7.1.3 A number of materials, (for example, see Table 1), exhibit dose-dependent effects when the electron dose exceeds a material specific critical dose  $D_c$ . The magnitude of the critical dose corresponds to the onset of detectable damage and the values may be subject to future revision. The material specific dose,  $D_c$  may be as low as  $10A/m^2$ .

TABLE 1 Electron Beam Damage in AES<sup>A</sup>

| Material            | Energy  | $10^4 C/m^2$         | T = time at 1<br>$10A/m^2$ | Refs |
|---------------------|---------|----------------------|----------------------------|------|
| $Si_3N_4$           | 2 keV   | stable               | ...                        | (21) |
| $Al_2O_3$           | 5 keV   | 10                   | 3 h                        | (2)  |
| Cu, Fe              | 1 keV   | 1                    | 15 min                     | (22) |
| Pthalocyanines      |         |                      |                            |      |
| $SiO_2$             | 2 keV   | 0.6                  | 10 min                     | (21) |
| $Li_2WO_4$          | 1 keV   | 0.05                 | 8 min                      | (23) |
| NaF, LiF            | 0.1 keV | 0.06                 | 60 s                       | (18) |
| $LiNO_3$ , $LiSO_4$ | 1 keV   | 0.05                 | 50 s                       | (23) |
| KCl                 | 1.5 keV | 0.03                 | 30 s                       | (18) |
| $TeO_2$             | 2 keV   | 0.02                 | 20 s                       | (24) |
| $H_2O(F)$           | 1.5 keV | 0.01                 | 10 s                       | (25) |
| Native oxides       | 5 keV   | $2 \times 10^{-3}$   | 2 s                        | (3)  |
| $C_6H_{12}(F)$      | 0.1 keV | $3 \times 10^{-4}$   | 0.3 s                      | (26) |
| $Na_3AlF_6$         | 3 keV   | $10^{-4} - 10^{-3}$  | 0.1 s                      | (27) |
| $CH_3OH(F)$         | 1.5 keV | $2.5 \times 10^{-4}$ | 0.3 s                      | (25) |

A

where:

$D_c$  = critical dose for detectable damage,

T = time of electron bombardment at  $10A/m^2$  without detectable damage,

and

F = frozen.

(Adapted from Ref. 1.)

7.1.4 In practice, the electron dose is directly dependent upon the electron beam current density,  $J_B$ , ( $A/m^2$ ), the time of electron irradiation in seconds,  $t$  (s); and the angle of incidence,  $\Theta$ , of the beam on the sample. That is,  $D_C (C/m^2) = J_B (A/m^2) \cdot t (s) \cdot \cos\Theta$ . Putting the electron beam current density into commonly used condition,  $10 A/m^2$  would be equivalent to using  $10^{-8} A$  incident beam current into a  $33 \mu m$  electron beam diameter at normal incidence.

7.1.5 The electron beam-induced heating of a given material of poor thermal conductivity and the accumulation of charge on a material of poor electrical conductivity are dependent upon the electron beam current density.

7.1.6 Current densities for a static electron beam should be of the order  $10^4 A/m^2$  or less for susceptible materials. In the case of rastered or gated electron beams, the time-averaged current density and the instantaneous current density must be considered. Even though the time-averaged current density may be small, the instantaneous current density may be sufficient to cause specimen damage or specimen charging.

7.1.7 In small-spot AES analysis, or scanning Auger microscopy, the use of electron probes with high current density is inherent. Obviously a trade-off between signal-to-noise and the perturbing effects of the electron beam is required (2).

#### 7.2 Electron Energy:

7.2.1 The electron beam effects which involve electronic excitation are not strong functions of electron beam energies used for AES (1 to 25 keV). Changes in electron beam energy will affect the depth, and therefore the volume, in which such changes occur.

7.2.2 Electron beam effects due to heating can be minimized by reducing the electron beam power since this reduces the power dissipation within the specimen.

7.2.3 Electron beam effects arising due to charging and electric fields in the surface can be minimized by appropriate empirical choices of electron beam condition (accelerating voltage, current, and current density). It should be noted that the electron beam angle of incidence (the angle between the electron beam and the specimen normal, as defined in Terminology E 673) influences the electron emission coefficient of the specimen surface.

### 8. Susceptible Materials

8.1 *Nonmetallic Materials*, particularly oxides, fluorides, chlorides, alkali halides, carbonates, and organics are most prone to decomposition under electron beam irradiation.

8.2 *Adsorbed Species*, particularly carbonaceous molecules, water and halogens, are usually desorbed, but in some cases may change their chemical form.

8.3 *Metal Surfaces (Clean)*, are most susceptible to ESA; the degree is, of course, enhanced by poor vacuum conditions and depends on the composition of residual gases. The type of specimen surface preparation is also an important factor.

8.4 *Insulators*, may undergo "unstable charging" wherein it is difficult to acquire an AES spectrum.

8.5 *Mobile Ionic Species*, particularly within oxides, nitrides, and other dielectric materials, are subject to electric field induced migration under the electron beam.

8.6 *Nonmetallic Powders*, fibers, and other specimen configurations which make poor thermal contact with the specimen

holder, are more susceptible to beam heating.

8.7 Table 1 is a list of some specific materials reported to undergo electron beam induced decomposition during AES. An estimate of the critical electron dose,  $D_c$  ( $C/cm^2$ ), is included as a guide to the electron beam current density and irradiation time which can be tolerated without detectable damage.

## 9. Methods of Observation and Minimization

9.1 Determine the existence and extent of electron beam effects for unfamiliar specimens by sequential acquisition of Auger spectra during continuous electron irradiation.

9.2 If the specimen is a bulk insulator, charging is generally reduced by decreasing the electron beam current, the current density (by defocusing the electron beam), lowering the accelerating voltage, and increasing the tilt angle (to increased electron emission).

9.3 If the specimen is a thin insulating film on a conductive substrate, charging is generally reduced by increasing the accelerating voltage and decreasing the angle of incidence. This has the effect of increasing the depth of penetration of the electron beam into the conductive layer.

9.4 Many electron beam effects involving diffusion processes may be minimized by cooling the specimen. This can be achieved using a variety of methods including the use of liquid

nitrogen cooled specimen holders. Maintain good thermal contact between the specimen and specimen holder.

9.5 For the analysis of insulators, maximize thermal and electrical contact to the specimen. Consider placing a grounded conducting foil or a conductive mask over the specimen near the analyzed region or a grid over the specimen surface to assist in charge dissipation. Consider the use of glancing incidence for the electron probe. If the insulating layer is thin consider using a higher energy electron probe.

## 10. Reporting Electron Beam Effects

10.1 The conditions that are used to control electron beam effects should be reported in a manner consistent with Practice E 996. This record should, at a minimum, include the electron beam conditions, such as accelerating voltage, incident current, current density, time of exposure, and incidence angle. If the electron beam was rastered over the specimen, state the raster speed, area, and beam diameter. Also, state if any thermal cooling or electrical contact to the specimen was used.

## 11. Keywords

11.1 Auger electron spectroscopy; charging; electron beam; electron beam damage

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