TEC MAX SYSTEM

OPERATION AND MAINTENANCE MANUAL FOR THE TEC MODEL 4005 MAX (MINIATURE APPARATUS FOR X-RAY DIFFRACTION SYSTEM)

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1 TEC MAX Introduction

The TEC MAX System is a miniaturized device for measuring surface residual stresses using x-ray diffraction. It is highly portable and designed to be placed into small areas.   The TEC MAX consists of both hardware and software. The hardware includes a fixed two-detector diffractometer, cable assembly, controller unit, and a host computer. The software includes two application programs: MAX Analysis Manager and MAXAcquisition, The system incorporates built-in safety features to protect both the operator and the equipment.

Figure 1 is a block diagram of the system showing the main components and their connections.
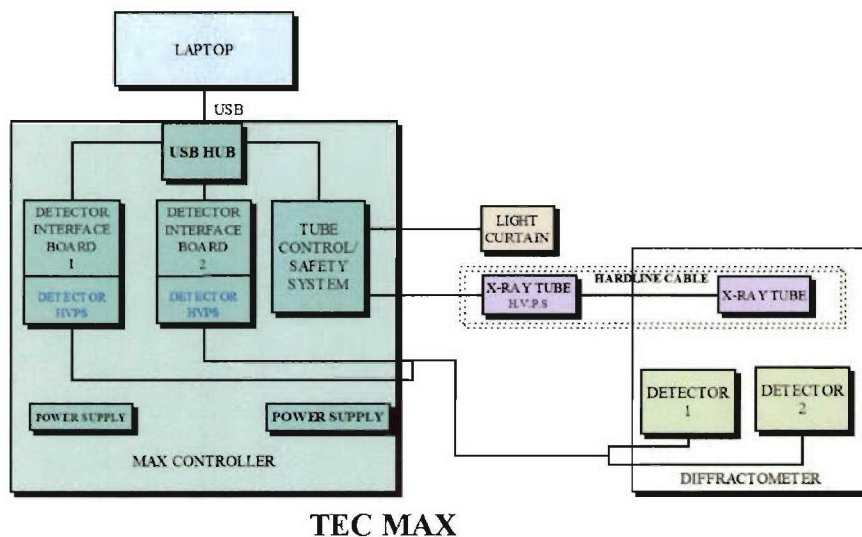
Figure 1 TEC MAX Block Diagram

1.1   TEC MAX Hardware

Figure 2; The TEC MAX System

1.1.1 Host Computer

A host computer is supplied with the TEC MAX System. This will normally be a laptop computer for increased flexibility in use of the system. The only specifications for the host computer are that it must have an NT-based operating system and a USB port, The TEC MAX host computer will come pre-loaded with the required application software for the system.
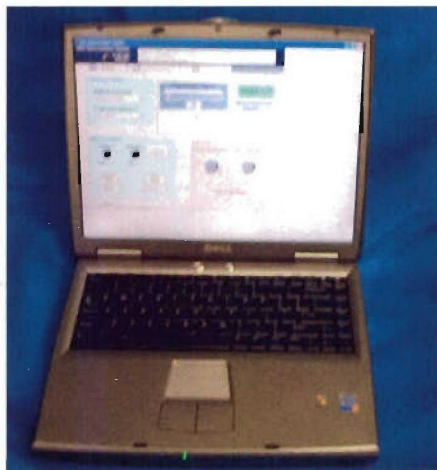
Figure 3 The TEC MAX Host Computer

1.1.2 MAX Controller

The TEC MAX Controller is the electronics and power supplies required to drive the x-ray tube and collect data from the detectors. It is assembled in a plastic transport case for portability. All connections to the diffractometer and host computer are made to the front panel on the controller once the cover is opened. Power for the controller is 110V/220V, 60HZ/50HZ.

Figure 4 The TEC MAX Controller

1.1.3 MAX Diffractometer

The TEC MAX diffractometer consists of two PSPC detectors, a miniature x-ray tube and the x-ray high voltage power supply. The diffractometer is unique for the type of material being measured by fixing the detectors and the x-ray tube target in the correct mechanical configuration for the material being measured. If a different material is to be measured, the diffractometer head can be easily changed to another mechanical fixture by the operator.
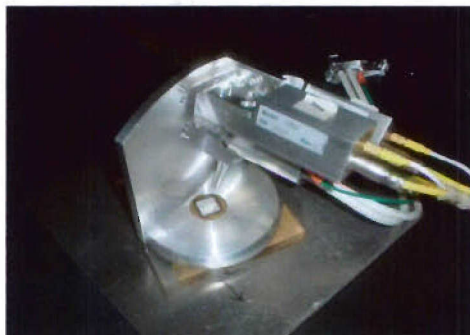
Figure 5 The TEC MAX Diffractometer

1.1.4 MAX Cable Assembly

The TEC MAX cable assembly connects the MAX controller to the diffractometer.

Figure 6 TEC MAX Cable Assembly

1.1.5 MAX Optical Beam Safety Device

Part of the safety features of the TEC MAX is an optical beam safety device provided with each system. This safety device should be set up to prevent anyone from gaining access to the radiation area around the x-ray tube port. When the optical beam is broken, the system will shut down the x-rays and inform the operator of the problem via screen message and audible alarm.
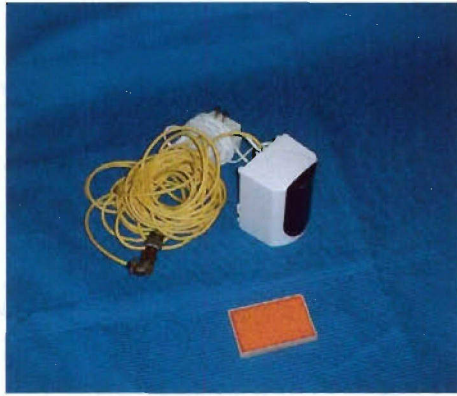
Figure 7 TEC MAX Optical Beam Safety Device

NOTE:

It is the responsibility of the operator to ensure the optical beam safety device is properly positioned for each measurement. The curtain should be placed so that it prevents any personnel from approaching within three feet of the diffractometer.

1.2 TEC MAX Software

The application software provided with the TEC MAX system includes two executable programs.

MAXAcquisition is the application that controls the hardware functions and collects data. MAX Analysis Manager is the application that processes the collected data and provides the residual stress results. While these are separate programs, they can be called by each other. That is, the operator can go directly to MAX Analysis Manager after MAXAcquisition collects the data.

Future upgrades to the basic software package will be added as they are released.

1.3 TEC MAX Optional Equipment

The Optional equipment for the TEC MAX system consists of different diffractometer assemblies pre-aligned for different materials and optional safety features that can be used in place of the optical beam safety device. Future potential enhancements include a movable diffractometer for multiple psi angle tilts and retained austenite measurement capabilities.

2 Theory of Operation

2.1 Theoretical Basis

This section briefly reviews the elementary theory of stress residual analysis using x-ray diffraction, and gives a few comments on materials to be tested and the condition of the material surface. However, measuring stress in some components produces data that cannot be interpreted in terms of the simple classical theory of biaxial stress, as would be commonly assumed. The data presented here clearly show that preferred orientation and grain-size effects must be considered.

2.2 Measuring Residual Stresses with X-Ray Diffraction

2.2.1 Bragg's Law for Diffraction

X-ray methods of analyzing residual stresses in crystalline materials (metals, alloys, and some ceramics) have been tested and compared to other methods and are used throughout the world. The basis of the technique is Bragg's law for diffraction.

A x-ray tube usually produces one very intense wavelength. In a crystal, the atoms are arranged in "planes" or sheets, with some spacing (d). Because x-rays are electromagnetic radiation, they are deflected or "scattered" by the charges on the electrons in the atoms. Waves scattered from successive planes add constructively for only a certain geometric condition, which is shown in Equation 2.1. This occurs when the difference in the paths of the scattered beams from these planes is a multiple (n) of the wavelength (λ). This results in Bragg's law:

Equation 2.1

$$n\lambda = (2d)\sin\theta$$

Or

Equation 2.2

$$\lambda = \left(2\frac{d}{n}\right)\sin\theta$$

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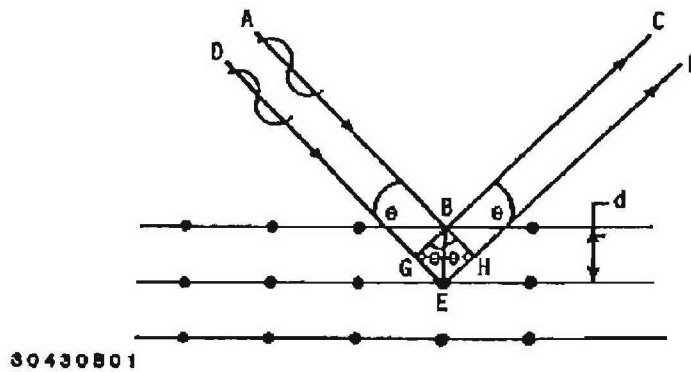
Figure 8 Scattering from Planes of Atoms.

The atomic planes are seen edge-on. The difference in path length of x-rays ABC and DEF is GE+EH and is equal to 2d sinθ.

Most materials are "polycrystalline" — that is, composed of many small crystals or grains. Assume an x-ray detector is moved over a range of angles 2θ to find the angle, θ, of the diffraction from grains that satisfy Bragg's law; that is, grains that have planes of atoms with interplanar d-spacing such that λ = (2d/n) sinθ. Grains that have planes with this d-spacing parallel to the surface will diffract as shown in Figure 9a.

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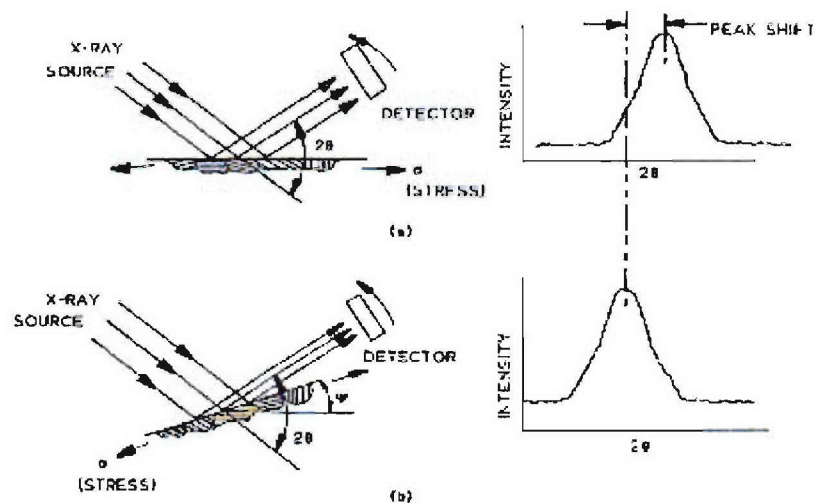
Figure 9 Principle of the sin²Ψ technique for measuring residual stress by x-ray diffraction.

This diffraction takes place from a thin surface layer about 20 μm thick, depending on the x-ray wavelength and atomic number of the sample. If the surface of the specimen is in compression, the d-spacing of these planes parallel to the surface is larger than in the unstressed state. Conversely, when tensile stresses are encountered, the d-spacings of these planes are smaller.

If the diffractometer is then tilted to an angle Ψ with respect to the incident diffraction vector (Figure 9b), new grains diffract, and the orientation of the diffraction planes is more nearly perpendicular to the stress direction. The result is that, with the tilt, the d-spacing decreases (because of the assumed compressibility) and the angle 2θ increases. In effect, the interplanar spacing acts as an internal strain gage.

The parameter of interest is the microstrain as given by Δd/d. If there is no stress in the sample, the diffraction curves in Figure 9a and 9b will superimpose. However, in the presence of surface stresses, the atomic planes represented in Figure 9a are compressed by a tensile stress with respect to those in Figure 9b due to the Poisson effect. Thus, a peak shift results. The stress (σ) can be determined from the shift through the relationship:

Equation 2.3

$$\sigma \simeq K \Delta 2\theta$$

where K is a constant determined by the elastic constants of the material under investigation.

Thus, an accurate and rapid measurement of the location of the diffraction peaks can be used to determine the presence of both residual and loading stresses. The use of a position-sensitive detector can increase the speed and accuracy of such determinations by over 100 times when compared to classical procedures.

The diffraction peak should be measured at as large a scattering angle as possible. This can be seen by differentiating Bragg's law:

Equation 2.4

$$d\lambda = 0 = \left(-2d\cos\theta\right)\Delta\theta + 2\Delta d\sin\theta \,;$$

Or

$$\Delta\theta = \left(\frac{\Delta d}{d}\right)\tan\theta$$

Thus, for a given strain $\Delta d/d$, the shift in the peak ($\Delta\theta$) is larger when 2θ is larger.

Equation 2.3 is a simplification that involves many assumptions, both mathematically and metallurgically. Metallurgical variables affecting the results include preferred orientation, grain size, normal stresses, stress gradients, and composition gradients. With the computer technology incorporated in the TEC MAX system, the mathematical simplifications are not made, and all of the currently available complex data analyses for metallurgical variables are incorporated into the system. As research in the field progresses, more analyses will be added to the software system.

The TEC MAX X-Ray Diffraction System is designed to make measurements as simply and as quickly as possible. The diffractometer is relatively compact because a miniature x-ray tube is used. The detector does not have to scan slowly over 2θ to measure the peak because it is a position-sensitive detector that literally "sees" a broad range of 2θ angles at once.

The location of each x-ray photon that strikes the detector is decoded electronically, providing a digital image of the diffracted x-rays in one dimension. **MAX ANALYSIS MANAGER**, the software for the TEC MAX X-Ray Diffraction system, locates the peak in this image by statistical curve-fitting after making corrections for geometric factors which affect the scattering process and which vary with θ. The error in measurement is affected by the amount of data acquired; **MAX ANALYSIS MANAGER** also computes this error. The result of the error computation is reported so that the value of the measurement can be judged. From these data, it is possible for the operator to estimate the counting time which will give an acceptable error in stress.

Since residual stresses are elastic, elastic theory is employed to convert the measured strain to stress. Consider the axial system shown in Figure 10 and note that the P_1 axes define the specimen, whereas the change in interplanar spacing is measured along L. The stresses or strains in the same coordinate system (P_1) without a prime and that along L as primed. Then, from elastic theory, and with σ_{ij} a stress component:

Equation 2.5

$$\frac{d_{\phi\psi} - d_o}{d_o} = \varepsilon_{\phi\psi} = 1/2\, S_2\,(hkl)\,[\,\sigma_{11}\cos^2\phi + \sigma_{12}\sin 2\phi + \sigma_{22}\sin^2\phi\,]\sin^2\psi$$

$$+ 1/2\, S_2\,(hkl)\,\sigma_{33}\cos^2\psi + S_1\,(hkl)\,[\,\sigma_{11} + \sigma_{22} + \sigma_{33}\,]$$

$$+ 1/2\, S_2\,(hkl)\,[\,\sigma_{13}\cos\phi + \sigma_{23}\sin\phi\,]\sin 2\psi.$$
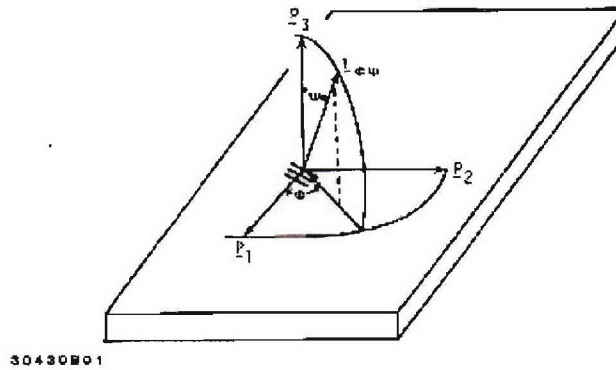
Figure 10 The definition of the specimen axes.

Where P, defines the surface, and $L_{\phi\psi}$, the measurement direction.

The term d_o is the unstressed interplanar spacing. Also, the terms 1/2 S_2(hkl) and S_1(hkl) are elastic constants which, for an isotropic medium, are $(1+v)/E$ and $-v/E$, respectively[1]. For an anisotropic material, these constants can be measured by applying known elastic loads to a piece of material as much like the sample as possible. It is also possible to calculate them from single crystal elastic constants. The commonly used values have been compiled for a wide range of materials; these values are included in **MAX ANALYSIS MANAGER**. The stresses and strains are averaged over the depth of penetration of the x-ray beam.

If one assumes $\sigma_{33} = \sigma_{13} = \sigma_{23} = 0$; that is, the stress components normal to the surface of the sample are negligible. Then Equation 2.5 reduces to:

Equation 2.5

$$\frac{d_{\phi\psi} - d_o}{d_o} = 1/2\ S_2\ (hkl)\ [\ \sigma_{11}\cos^2\phi + \sigma_{12}\sin 2\phi$$

$$+\ \sigma_{22}\sin^2\phi]\ \sin^2\psi + S_1\ (hkl)\ [\ \sigma_{11} + \sigma_{22}]$$

Calling the terms in the first bracket σ_Φ, the stress in the Φ direction, and realizing that at $\Psi = 0$:

---

1 E is Young's modulus; v is Poisson's ratio, and (hkl) are the Miller indices which identify the diffracting planes of spacing d.

Equation 2.6

$$\frac{d_{\phi,\psi=0} - d_o}{d_o} = S_1(hkl)\,[\,\sigma_{11} + \sigma_{22}\,],$$

Equation 2.5 reduces to:

Equation 2.7

$$\frac{d_{\phi,\psi} - d_{\phi,\psi=0}}{d_o} = 1/2\,S_2(hkl)\,\sigma_\phi \sin^2\psi.$$

Since the changes in d-spacing are in the fourth or fifth decimal place, d_o can be replaced by $d_{\phi,\psi=0}$, and does not need to be known. Then

Equation 2.8

$$\frac{d_{\phi,\psi} - d_{\phi,\psi=0}}{d_{\phi,\psi=0}} = 1/2\,S_2(hkl)\,\sigma_\phi \sin^2\psi.$$

Equation 2.8 indicates that a plot of $d_{\Phi,\psi}$ versus $\sin^2\psi$ should be linear, with a $\Psi = 0$ intercept at $d_{\Phi,0}$ and a slope:

Equation 2.9

$$½\,S_2(hkl)\,\sigma_\Phi d_{\Phi,\psi=0}$$

If $½\,S_2(hkl)$ is known, the stress may be obtained. This is the standard procedure for stress analysis. It is important to keep in mind that this procedure *assumes* $\sigma_{33} = \sigma_{13} = \sigma_{23} = 0$, assumptions often not valid in real components

If the intensities of the Ψ angles vary significantly, this may be a result of strong preferred orientation or texture in the component. There is no point in analyzing the stress in such a case. An indication of potential preferred-orientation problems is significant variation in the (corrected) integrated intensity of the diffraction peak as a function of Ψ angle. **MAX ANALYSIS MANAGER** computes the integrated intensity. Sometimes switching to an (h00) or (hhh) reflection or using a more penetrating radiation will reduce this effect.

One of the assumptions in this method is that sufficient grains are reflecting to obtain a good average. This can be judged by moving the sample relative to the x-ray beam. Large variations in the intensity imply too coarse a grain size. This can occur in welds or castings, particularly with Al- and Ni-base alloys and with austenitic stainless steel products. Use of a more penetrating radiation may reduce this problem.

The x-ray diffraction method (or "XRD") measures the sum of two kinds of stresses. The first of these is the stress pattern that arises due to the difference in plastic extension of the inside and outside of the part, as might occur during quenching or some surface treatment like peening. These stresses are called "macrostresses." The other type of stress pattern is called "interaction" or "pseudo-macro" stresses. These stresses arise because of the different elastic and plastic behavior of the different phases in a multiphase solid.

There are two ramifications of this fact. The first is that care is needed in comparing XRD results to mechanical methods because the latter are designed to measure only the first kind of stress. Methods exist for separating the XRD-determined stressed into both these components if such a comparison is desired.

The second ramification is that the XRD stresses are usually determined in only one phase. It is well known that residual stresses must balance. The macrostresses measured in one phase will balance if measurements versus depth are made. However, the interaction of stresses in one phase will be balanced by stresses in the other phases. If the latter are not analyzed, it may appear strange that the stresses in the one phase do not vary versus depth, for example.

What is the accuracy and precision of this technique? Accuracy is largely dependent on the elastic constants. As for precision, comparisons have been made between laboratories, and repeated measurements have been reported. Typically the uncertainty is 10-20 MPa (±1500-3000 PSI). The value can be larger for particularly difficult geometries. The source of this error is twofold. First, there are counting statistics, which can be controlled by varying the data acquisition time.

Second, there are various geometric errors, some of which can be corrected (such as the variation in scattering of the atoms with θ, absorption, and x-ray beam polarization). Others can only be estimated, the most important of which are the beam's divergence on the sample and sample positioning.

Any measurement should also include an estimate of the uncorrected statistical errors. The latter is an important feature that has always been implemented in the **MAX ANALYSIS MANAGER** software.

Finally, while it is currently true that most residual stress analysis is made in metals and alloys because of their wide use and plastic behavior, the XRD method *is* applicable to any crystalline material for which a high-angle diffraction peak can be located. There has been considerable interest in the very large stresses that can occur in ceramics as a result of grinding, especially in transformation-toughened products. For example, TEC has successfully measured stresses in polycrystalline Al_2O_3, Si_3N_4, and graphite components.

2.3   Single Exposure Technique (SET)

This technique uses two detectors to measure residual stress by obtaining two diffraction peaks with a single exposure. Before performing the stress calculations, however, the normal corrections must be made for the peak position (Lorentz-polarization, absorption, scattering factor, background).

The detectors are positioned at the required 2θ position on either side of the x-ray tube in the Ω configuration. The x-ray tube makes an angle, β, with the sample perpendicular (β cannot be equal to zero). The desired β angle sets one detector near Ψ=0 and the other detector at Ψ=Ψ.

The basic equation is:

Equation 2.10

$$\sigma_\phi = \left(\frac{\pi}{180}\right)\left(\frac{E}{1+v}\right)\left(\frac{1}{2\sin 2\beta \sin^2 \theta}\right)\Delta\theta$$

where:

$\dfrac{E}{1+v}$ is the elastic constant

β = angle between the tube and sample perpendicular

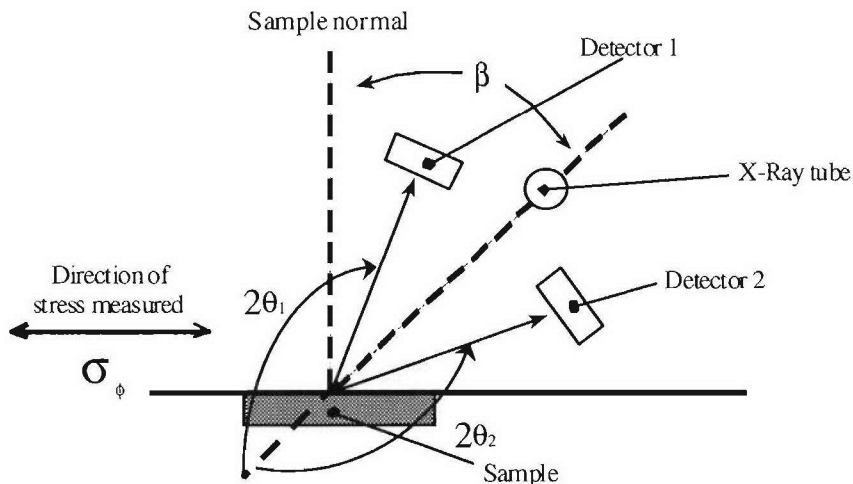$\theta =$ diffraction angle, $\approx \dfrac{\theta_1 + \theta_2}{2}$

Figure 11 Diagram of Single Exposure Technique (SET)

2.4 Statistical Error Analysis

2.4.1 Introduction

There are two classes of error in the residual stress measurements discussed previously or in the retained austenite measurements. These are systematic errors which result from various aspects of the x-ray optics of the instrument and counting errors which result from the statistical nature of the x-rays counted in the detector. The following two sections are concerned only with the counting errors.

Systematic errors include such effects as the following:

- Geometric and collimation errors that arise from the finite size of the incident x-ray beam (both horizontal and vertical)

- Displacement of the detector from the x-ray focusing circle

- Displacement of the sample surface from the center of rotation of the diffractometer

- Noncoincidence of the zero of ψ-angle with the normal to the sample surface

- Peak asymmetries arising from variation of background with angle and Kα doublet resolution

- Bragg angle-dependent corrections such as Lorentz polarization, sample absorption, and variation of atomic scattering factors

Great care has been taken in the design of the diffractometer to eliminate systematic errors due to geometrical effects. The use of small, pin-hole collimators reduces (but does not eliminate) collimation errors. **MAX ANALYSIS MANAGER** makes as many of the systematic corrections as is reasonably possible. Such corrections include sample absorption, Lorentz polarization, Kα doublet, and variation of background with angle.

2.4.2 Counting Statistics — Biaxial Analysis

The error in the measured stress which results exclusively from the counting statistics of the recorded x-ray data is a result of the following chain. Fluctuations in the recorded intensities at each Bragg angle result in an error in determining the Bragg peak location. An error in Bragg peak location results in an error in the d-spacing, which in turn leads to an error in the slope of d-spacing versus $\sin^2\psi$ and, hence, the stress. In this analysis, a linear relation between the slope and stress is assumed.

The XRD peak profile, as recorded with a position-sensitive proportional counter (PSPC), is acquired in equally spaced 2θ increments for a fixed time, t. Let the

2θ increment be δ and the I_j be the power (counts/second) recorded in the jth interval. A parabola of the form

$$a + b\delta + cj^2\delta^2 = I_j$$

is fitted to the top fraction of the diffraction peak. Here the Bragg angle of the jth detector element is given by $x_j = j\delta$. The fraction of the peak which is fitted should be in the neighborhood of 15% but may be as high as 50% in some unusual cases. In the following analysis, it will be assumed that there are an odd number, n, of detector resolution elements used in the fit. The coefficients a, b, and c are found by standard least squares techniques. The peak location is determined by the maximum in the parabola, and is given by

$$\frac{dI_j}{d(j\delta)} = 0$$

where

$$2\theta_p = 2\theta_o - \frac{b}{2c}.$$

In Equation 2.13, $2\theta_o$ is the 2θ position corresponding to j=0.

Following Wilson [British J. Appl. Phys. 16, 665 (1965)] and Cohen, the variance in $2\theta_p$ is given by

$$\sigma^2(Y_p) = \sum_{j=-n}^{n}\left[\left(\frac{\partial Y_p}{\partial M_o}\cdot\frac{\partial M_o}{\partial I_j}\right)^2 + \left(\frac{\partial Y_p}{\partial M_1}\cdot\frac{\partial M_1}{\partial I_j}\right)^2 + \left(\frac{\partial Y_p}{\partial M_2}\cdot\frac{\partial M_2}{\partial I_j}\right)^2\right]V_{I_j}$$

where

$$Y_p = 2\theta_p$$

and

$$M_i = \delta^{i+l} \sum_{j = \_n}^{n} j^i I_j$$

The least squares fit to the data shows that the constants in Equation 2.13 are functions of the moments of the diffraction peak, M_i

Equation 2.15.a

$$b = \frac{M_1}{\delta^3 n_2}$$

and

Equation 2.15.b

$$c = \frac{M_o n_2 \delta^2 - M_2 n_o}{\delta^5 (n_2^2 2 - n_4 n_o)}$$

where

$$n_i = \sum_{j=-n}^{n} J^i .$$

Performing the derivatives, substituting into 2.15b and remembering that $I_j = C_j/t$ where C_j is the number of photons counted in the jth element in the time t, it may be shown that

Equation 2.16

$$\sigma^2{}_{2_{\theta_p}} = \frac{\delta^4 (n_2 - n_0 n_4)^2}{4 n_2^2 (n_2 \delta^2 M_0 - n_0 M_2)^4} x$$

$$\sum_{j--n}^{n} \left[(n_0 \delta^3 M_1 j^2)^2 + (n_2 \delta^2 M_0 - n_0 M_2)^{2\delta^4{}_l{}^2} + (n_2 M_1)^2 \right] x \frac{C_j}{t^2}$$

Equation 2.16 thus describes the variance in the least squares estimate of the peak position which results exclusively from fluctuations in counting statistics. All

other sources of error, such as those discussed previously in this appendix, are excluded from this consideration.

Cohen has experimentally verified that Equation 2.18 indeed does describe the variance in $2\theta_p$, and has shown that the parabolic fit to the diffraction peak (Equation 2.15) gives the best estimate of the peak location.

With an estimate of the effect of counting statistics on peak location in hand, we may now estimate the resulting error in the d-spacing. From Bragg's Law

$$d = \frac{\lambda}{2 \sin \theta}$$

and

$$\sigma_d^2 = \frac{1}{2} \left(\frac{\lambda \cos \theta}{2 \sin^2 \theta} \right)^2 \sigma_{2\theta_p}^2$$

where

$$\sigma \frac{2}{2\theta}$$

is given by Equation 2.16.

Next is determining the variance of the stress computed from the known variances in d-spacing as a function of ψ. Assume that the stresses are biaxial. From Equation 2.8, a plot of the d-spacing versus $\sin^2\psi$ is a straight line with slope $\sigma(1+v)/E\ d_{\Phi,o}$ where $d_{\Phi,o}$ is the zero intercept of the straight line fit of d-spacing versus $\sin^2\psi$. The stress, σ, is then given by

$$\sigma = \frac{E}{1+v} \frac{m}{d_{\phi,o}}$$

where m is the slope of the fit and $d_{\Phi,o}$ is the zero intercept. Assume that N measurements of d are made at various Ψ angle as represented by the data points $(d_i, \sin^2\psi_i)$. Let $x_i = \sin^2\psi_i$ and $y_i = d_i$. Then, from least squares theory

Equation 2.20

$$\sigma = \frac{E}{1+v} \frac{N \Sigma x_i\, y_i - \Sigma x_i \Sigma y_i}{\Sigma x_i^2 \Sigma y_1 - \Sigma x_i \Sigma x_i\, y_i}$$

In Equation 2.20, the summing index i runs from 1 to N.

Assume that there are no errors in determining the Ψ angle at each measurement location and there are no errors in E/(1+v). Then

Equation 2.21

$$\sigma_o^{\,2} = Z \left(\frac{\delta\,\sigma}{\delta\,y_i} \right)^2 \sigma_{d_i}^2$$

where

$$\sigma_{d_i}^2$$

is the variance of the d-spacing determined at the ith location.

Performing the differentiations of 2.20 as required in 2.21, it may be shown that the variance in the stresses that result only from counting statistics are

Equation 2.22

$$\sigma_o^2 = \left(\frac{E}{1+v} \right)^2 \frac{1}{\left(F_3 - F_4 \right)^2} \sum_{i=1}^{N} \left[N^2 D_1^2 + A_1^2 + \frac{\left(A_2^2 + A_1^2\, D_1^2 \right)\left(F_1 - F_2 \right)^2}{\left(F_3 - F_4 \right)^2} \right] \sigma_{d_i}^2$$

where

$A_1 = \Sigma \sin^2\psi_1$

$A_2 = \Sigma \sin^4\psi_i$

$D_1 = \sin^2\psi_i$

$F_1 = N \Sigma \sin^2\psi_i d_i$

$F_2 = A_1 \Sigma d_i$

$F_3 = A_2 \Sigma d_i$

and

$F_4 = A_1 \Sigma \sin^2 \psi_i d_i$

All summation range $1 \le i \le N$.

The error in stress as computed from Equations 2.16, 2.17, and 2.22 is presented in the final results output report under the heading, "Counting Statistics Error." It must be remembered that this is the contribution to the error from counting statistics only and assumes that there are no systematic errors from such metallurgical sources as grain size, preferred orientation or from deviation from a biaxial stress state [i.e., terms such as σ_{33} or shear stresses normal to the sample surface (σ_{13}, σ_{23})].

2.5 Stress Constants

TEC has compiled a library of x-ray stress constant libraries. These values are contained in the MATERIALS LIBRARY maintained in MAX ANALYSIS MANAGER.

2.6 Surface Considerations

The depth of penetration of x-rays into various metals and alloys depends both on the wavelength of the incident radiation and the composition and atomic number of the alloy constituents.

When the path length, Φ, of the radiation is $1/\mu$, given by

$$\varphi = 2x \sin\theta$$

where x is the distance normal to the surface, then the scattered intensity is reduced in intensity to 37% of its incident value. This means that 63% of the scattered intensity comes from a layer of material $x = 1/2\mu \sin\theta$ thick; 86% comes from a layer 2x thick, and 95% from a layer 3x thick.

Because the x-ray beam penetrates typically only a few tens of μm, it is important to recognize that one is really measuring surface and near-surface stresses. Thus, any smoothing, brushing, or disrupting of the surface before measurement probably will affect the stresses. Therefore, it is important to avoid mechanical abrasion or deformation of the surface before measurement be avoided. You must consider carefully the nature of the results desired and the effect of such modifications.

Recall that the XRD technique measures the microstrain in only specifically selected atomic planes (Equation 2.1). Thus, measurements may be made through thin, low-absorption layers of other material on the surface. Because such layers of different materials have different crystal structures, they will only coincidentally have diffraction peaks near the diffraction peak in use for stress determinations, and their sole effect usually will be to decrease the signal-to-noise ratio of the desired diffraction peak.

TEC has found that residual stress measurements can be made through thin layers of paint on steel surfaces (but not lead-based paints!), through rust on steel parts, and through anodized passive layers on aluminum. In other cases, it is essential to remove surface contaminants by techniques such as chemical etching or electropolishing. You must take care that the surface preparation technique does not introduce significant surface stresses itself. Because every situation is different, all TEC can do is recommend extreme care in measuring and interpreting results on modified surfaces.

2.7   Position-Sensitive Proportional Counters

In traditional x-ray diffractometry, a proportional or scintillation counter, preceded by a narrow slit, is scanned through a range of 2θ angles. The resultant scattered intensity is recorded in analog form on a strip chart or in digital form in a scaler/printer or a computer. This procedure requires a precision diffractometer to control the detector/slit motion and is time-consuming, as data at each 2θ angle are recorded serially.

In recent years, devices known as position-sensitive proportional counters (PSPCs) have been developed. These devices can record not only the presence of a photon, but also its location along a line. Typically, spatial resolutions from 50 to 500 μm can be achieved. Thus, a PSPC with a wire length of 50 mm and a resolution of 50 μm can record data at 1000 locations (scattering angles) simultaneously

Two significant improvements in residual stress analyses result from these devices: (1) a diffractometer with no 2θ motion can be built because the PSPC subtends a sufficiently large (~16°) 2θ-angular range to record the entire Bragg peak for a particular class of materials, and (2) all the data are recorded simultaneously with a decrease in data acquisition time of up to 100 times.

Measurement times as small as a few seconds are now possible, even with low-power x-ray sources. This is true because the cosmic and electronic noise of a PSPC is no more than that of a simple proportional or scintillation counter. However, this noise is now spread uniformly over all channels (scattering angles), with an attendant improvement of the signal-to-noise ratio of the PSPC system of from 100 to 1000 over the simple detectors. To take advantage of these technological developments, additional sophisticated electronics and data acquisition systems are required

TEC developed a high-resolution PSPC detector for residual stress analysis; the signal processing electronics have been carefully selected and matched to perform the necessary position decoding and shaping functions.

3 Radiation Safety Requirements

3.1 Introduction

Read this section carefully before you apply power to the TEC MAX (Model 4005 Miniaturized Apparatus for X-Ray Diffraction) System . This chapter will give you adequate information about radiation safety so you will know how to operate your x-ray diffraction system safely. The chapter also lists federal regulatory requirements for safely operating analytical x-ray equipment.

> **NOTE**: *The information in this document is not intended to replace regulations published by state or other regulatory bodies having jurisdiction over any users analytical x-ray equipment. It is the owner's/operator's responsibility to become familiar with, and to adhere to, the appropriate state and federal regulations for the control and safe use of radiation.*

Federal radiation safety requirements for x-ray diffraction and fluorescence analysis equipment are published in ANSI N43.2-1977. The TEC MAX (Model 4005 Miniaturized Apparatus for X-Ray Diffraction) System is designed to meet all regulatory requirements published therein.

State regulations regarding licensing, registration, and radiation safety vary from state to state. Therefore, *the owner/operator must become familiar with and meet the requirements of your state.*

> **IMPORTANT!** *Before the owner/operator turns on the x-ray beam, they must obtain the necessary registration or licensing in the state in which they will operate the system. You can obtain the name and address of your state agency from TEC. If you need assistance in obtaining your license or registration, please call TEC at (865) 966-5856. Ask for the Materials Testing Contract Manager.*

The radiation safety requirements discussed in this section are applicable to analytical x-ray equipment in almost all states. In keeping with many official publications, this manual uses the word *shall* to indicate a provision that is a requirement in most states.

When not underlined in this section, the term "exposure" has a more general meaning than that given by its dictionary definition.

3.2 Definitions

Analytical x-ray equipment — any devices that use x-rays to examine the microstructure of materials.

Analytical x-ray system — analysis equipment composed of local and remote components. Local components; i.e., those that are struck by x-rays, including the radiation source housings, port and shutter assemblies, collimators, sample holders, cameras, goniometers, detectors, and shields. Remote components include the power supplies, transformers, amplifiers, readout devices, and control panels.

Calendar quarter — a period of time of not less than twelve consecutive weeks or not more than fourteen consecutive weeks. The first calendar quarter of each year shall begin in January, and subsequent calendar quarters shall be arranged so that no day is included in more than one calendar quarter, and no day in any one year is omitted from inclusion in a calendar quarter.

Closed-beam configuration — a system containing shields or enclosures that prevent the accidental exposure of any personnel or any of their body parts to the x-ray beam.

Failsafe characteristics — features designed into the system that cause the beam port shutters to close or otherwise prevent emergence of the primary beam upon the failure of a safety or warning device.

Certificate of Registration — unless otherwise specified, a certificate issued in accordance with the appropriate state regulations.

Absorbed dose — the amount of energy imparted to matter by ionizing radiation in a volume element divided by the mass of irradiated material in that volume element. The unit of absorbed dose is the "rad" (see *Rad*).

Dose equivalent — describes the postulated effect, expressed on a scale common to all radiation, of radiation on a given organ. The dose equivalent is defined as the absorbed dose in rads times certain modifying factors. The unit used to describe the dose equivalent is the "rem" (see *Rem*).

Exposure — the measure of the ionization produced in air by x- or gamma radiation. It is the sum of the electrical charges on all of the ions of one sign that are produced in air, when all electrons liberated by photons in a volume element of air are completely stopped in air, divided by the mass of the air in the volume element. The unit used for *exposure* is the "roentgen" ® (see *Roentgen*). When not *italicized* in this chapter, the word "exposure" has a more general meaning.

Exposure rate — the *exposure* per unit of time such as R/min or mR/h.

Gray (Gy) — The SI unit for absorbed dose; one rad equals 0.01 Gy (see *Rad*).

Human use — the internal or external administration of radiation or radioactive material to human beings for medical purposes.

Ionizing radiation — see *"Radiation"*

Normal operating procedures — procedures for operating x-ray equipment under conditions suitable for analytical purposes. To meet these conditions, you must have shielding and barriers in place. Normal operating procedures do not include maintenance, but do include routine alignment procedures. Routine and emergency radiation safety procedures are included in this definition.

Occupational dose — the exposure of an individual to radiation either (1) in a restricted area or (2) in the course of employment in which the individual's duties involve exposure to radiation. The occupational dose does not include any exposure of an individual to radiation for either medical diagnosis or therapy.

Open-beam configuration — an arrangement of the analytical x-ray producing device such that an individual could accidentally expose some part of the body in either the primary or diffracted beam path during normal operation.

Person — any individual, corporation, partnership, firm, association, trust, estate, public or private institution, group, agency, political subdivision of a state, any other state or political subdivision or agency thereof, any federal agency, and any legal successor, representative, agent, or agency of the foregoing.

Personnel monitoring equipment — any devices designed to be worn or carried by an individual so the dose received by the individual can be estimated. These devices include film badges, pocket dosimeters, and thermoluminescent dosimeters.

Primary beam — the ionizing radiation that passes through an aperture of the source housing by a direct path from the x-ray tube. The x-ray tube is located in the radiation source housing.

Rad — the unit of the absorbed dose. One rad is the dose corresponding to the absorption of 100 ergs per gram of material.

Radiation — ionizing radiation. This radiation includes gamma rays and x-rays, alpha and beta particles, high-speed electrons, neutrons (indirectly ionizing), and other nuclear particles.

Radiation area — any area that is accessible to individuals in which there is radiation at such levels that a major portion of the body could receive in any one hour an absorbed dose in excess of five millirems or in any five consecutive days a dose in excess of 100 millirems.

Radiation machine — any device capable of producing radiation. This definition does not include a device that produces radiation via the use of radioactive materials.

Radiation safety officer — an individual who has a knowledge of, and the responsibility for, applying appropriate radiation-protection regulations, standards, and practices.

Registration — documented status with the appropriate state agency in accordance with the applicable state requirements.

Rem — the unit used to describe the dose equivalent. It is the measure of any radiation to body tissue measured by the estimated biological effect of an *exposure* to one roentgen ® of x-rays (one millirem [mrem] = 0.001 rem). Any of the following are considered to be equivalent to a dose of one rem:

- an *exposure* of one R of x-radiation or gamma radiation

- a dose of one rad due to x-radiation, gamma, or beta radiation

- a dose of 0.05 rad due to particles heavier than protons and with sufficient energy to reach the lens of the eye

- a dose of 0.1 rad due to neutrons or high-energy protons

Restricted area (or controlled area) — any area whose access is controlled by the licensee or registrant to protect individuals from exposure to radiation and radioactive material. A restricted area shall not include any area used for residential quarters, although a separate room or rooms in a residential building may be set apart as a restricted area.

Roentgen (R) — the unit of *exposure*. One roentgen equals 2.58×10^{-4} coulombs/kilogram of air.

SI — Système International [d'Unités]; International System [of units]. The international scientific standard of measurement.

Sievert (Sv) — The SI unit for dose equivalent; one rem equals 0.01 SV.

Source of radiation — any radioactive material or any device or piece of equipment that emits radiation or is capable of producing radiation.

Unrestricted area (or uncontrolled area) — any area whose access is not controlled by the licensee or registrant for the protection of individuals from exposure to radiation and radioactive material. Any area used for residential quarters must be unrestricted.

Worker — an individual who is engaged in work under a license or certificate of registration issued by the state department responsible for radiation protection. The license is controlled by the licensee. This definition does not include the licensee or registrant.

You/your -- refers to the owner/operator of the system.

3.3 Equipment Requirements

3.3.1 Safety Devices

In all open-beam configurations, the owner/operator shall employ a safety interlock that provides an exclusionary zone of 48 inches around the diffractometer whenever the x-ray tube is powered. This interlock should either prevent the insertion of a body part into the primary x-ray beam path or *cause the beam to be shut off upon entry of any object into its path.*

The owner/operator should use a tilt switch, broadcasting at any angle, that does not intersect the planar support member (i.e., table top) for the diffractometer during normal use. The tilt switch, cooperating with the planar support member, acts in lieu of a beam trap to prevent the transmitted primary beam from exceeding 0.25 mrem/hr.

3.3.2 Warning Devices

When operating in an open-beam configuration, the owner/operator shall provide the following readily discernible indicators:

1. an x-ray tube status indicator (ON/OFF) that is located near the radiation source housing if the primary beam is controlled in this manner

2. a shutter status indicator (OPEN/CLOSED) that is located near each port on the radiation source housing if the primary beam is controlled in this manner

Warning devices shall be labeled so that their purposes are easily distinguishable. Warning devices shall have failsafe characteristics.

3.3.3 Labeling

All analytical x-ray equipment shall be labeled with the following easily discernible sign or signs (these signs must include the radiation symbol):

1. On the x-ray source housing, place a sign that reads "CAUTION: HIGH-INTENSITY X-RAY BEAM." You may use similar wording that has the same intent.

2. Near any switch that energizes an x-ray tube, place a sign that reads "CAUTION: RADIATION — THIS EQUIPMENT PRODUCES RADIATION WHEN ENERGIZED." You may use similar wording that has the same intent.

3.3.4 Shutters

The port of the x-ray tube shall be provided with a beam shutter, This beam shutter shall have an integral collimation path interlocked such that the x-ray tube can be powered only when the shutter is installed.

3.3.5 Warning Lights

Near any switch that energizes an x-ray tube, an easily visible warning light shall be placed and labeled "X-RAY ON." Similar wording that has the same intent may be used. This light shall be illuminated only when the tube is energized. Warning lights shall have failsafe characteristics.

3.4 Requirements for Restricted Areas

3.4.1 Radiation Levels

When installing the local components of an analytical x-ray system, the owner/operator shall arrange, shield, or control access to them such that the radiation levels in any area surrounding the local component group could not result in a dose to an individual present therein in excess of either of the dose limits given below. This requirement shall be met at any specified tube rating.

1. Radiation levels that, if an individual were continuously present in the area, could result in that person receiving a dose in excess of two millirems in any one hour.

2. Radiation levels that, if an individual were continuously present in an area could result in that person receiving a dose in excess of 100 millirems in any seven consecutive days.

When using an analytical x-ray producing device in an open-beam configuration, the owner/operator must meet the following conditions:

1. Radiation levels in the vicinity of controls and the x-ray tube during the normal operation of the equipment shall be such that the operator's exposure shall not exceed, in one hour, 37 mrem to the hands or 2 mrem to the whole body, gonads, blood forming organs, or lens of the eye.

2. The exposure level at 5 centimeters from the x-ray tube housing shall not exceed 2.5 mR/hr during normal operations.

3.4.2 Surveys

In order to show compliance with the restrictions on radiation levels specified in the previous section ("Radiation Levels"), you shall perform radiation surveys of all analytical x-ray systems *at each* of the following times:

1. upon installation of the equipment

2. following any change in the initial arrangement, number, or type of local components in the system

3. following any maintenance that requires the disassembly or removal of a local component in the system

4. during the performance of maintenance and alignment procedures that require the presence of a primary x-ray beam when any local component in the system is disassembled or removed

5. anytime that a visual inspection of the local components in the system reveals an abnormal condition

6. whenever personnel monitoring devices show a significant increase over the previous monitoring period or if the readings are approaching the Radiation Protection Guides (radiation dose limits)

Radiation survey measurements may not be required in some states if a registrant can demonstrate to the satisfaction of the state regulatory agency that it complies with the restrictions on radiation levels specified in Section 3.4.1, "Radiation Levels."

3.5 Operating Requirements

3.5.1 Procedures

Normal operating procedures shall be written and available to all analytical x-ray equipment operators. No one shall be permitted to operate analytical x-ray equipment in any manner other than those specified in the procedures unless that person has obtained the written approval of the system owner's radiation safety officer.

3.5.2 Bypassing

No person shall bypass a safety device unless that person has obtained the written approval of the radiation safety officer. When a safety device has been bypassed, you shall place an easily discernible sign with the message "SAFETY DEVICE NOT WORKING" on the radiation source housing. You may change the wording of the sign, but the obvious intent must be the same.

You shall maintain a record of all such alterations, including date, duration, and person making the alterations, for inspection by the applicable regulatory agency.

3.5.3 Equipment in Operation

Except when the area containing the equipment is locked to prevent unauthorized or accidental entry, the operator shall be in immediate attendance of the equipment at all times when the equipment is in operation.

3.6 Personnel Requirements

3.6.1 Instruction

No person shall be permitted to operate or maintain analytical x-ray equipment unless that person has received instruction, and has demonstrated competence, *in each* of the following areas:

1. identification of radiation hazards associated with the use of the equipment

2. significance of the various radiation warning and safety devices incorporated into the equipment; or the reasons they have not been installed on certain pieces of equipment and the extra precautions required in such cases

3. proper operating procedures for the equipment

4. recognition of symptoms of an acute localized exposure

5. proper procedures for reporting an actual or suspected exposure

6. proper use of personnel monitoring equipment that is calibrated for the type and energies of radiation the worker may encounter

3.6.2 Personnel Monitoring

The following personnel shall be provided and shall use finger dosimetric devices:

1. analytical x-ray equipment workers who use systems that have an open-beam configuration and are not equipped with a safety device

2. personnel who maintain analytical x-ray equipment if the maintenance procedures require the presence of a primary x-ray beam when any local component in the analytical x-ray system is disassembled or removed

3.7 Detection and Measurement of Radiation from X-Ray Diffraction Equipment

The source for all the information in this section is the Appendix of the *American National Standard N43.2 Radiation Safety for X-Ray Diffraction And Fluorescence*, American National Standards Institute (ANSI) N43.2 - 1977. Citations in this section are the citations found in ANSI N43.2 — 1977. This section is included for information purposes only.

3.7.1 Nature of the Radiation (A1)

For diffraction tubes, typical acceleration potentials are 25 to 50 kVp. For tubes used in fluorescence analyses, typical acceleration potentials are 25 to 100 kVp.

([5], p. 1096) Therefore, the upper limit for the energy of x-ray photons is 50 or 100 keV.

In the white radiation continuum, there is no theoretical lower limit to the energy of the photons. The intensity below about 5 keV is low, and the x-rays are easily attenuated. We can assume that the continuum extends from 5 to 100 keV with an intensity maximum in the range of 20 to 30 keV, depending on the accelerating potential.

Superimposed on this continuum are the lines of the characteristic spectrum of the anode. These lines constitute less than half of the energy output when tubes are used for diffraction. The photon energies associated with these lines are generally between 5.4 and 17.5 keV.

In order to measure the dose from both the continuum and the characteristic spectrum, you should use a survey meter that has energy absorption characteristics similar to those of air over the 5 to 100 keV range.

3.7.2 Sources of Radiation (A2)

There are many sources of radiation associated with analytical x-ray machines. Hazardous radiation may come from any of the following sources:

1. the primary beam

2. leakage or scatter of the primary beam through cracks in ill-fitting or defective equipment

3. penetration of the primary beam through the tube housing, shutters, or diffraction apparatus

4. secondary emission from the sample or other material exposed to the primary beam

5. diffracted rays

6. radiation generated by rectifiers in the high-voltage power supply

The primary beam is the most hazardous of these sources because of the extremely high exposure rates of this beam. At the ports of ordinary diffraction tubes, exposure rates of 4×10^5 R/min have been reported. [2]

The leakage or scatter of the primary beam through openings in ill-fitting or defective equipment can produce very high-intensity beams that possibly have small and irregular cross sections.

For well-designed equipment, the hazard resulting from the penetration of the beam through shutters or the x-ray tube housing is small. At the energy levels

commonly used for diffraction and fluorescence analysis, it is easy to provide adequate shielding of the beam.

Diffracted beams also tend to be small and irregular in shape. They may be directed at almost any angle with respect to the main beam. Occasionally, and for short periods, the exposure rates of diffracted beams can be 80 R/hr.

Radiation that leaks from the high voltage power supply may come from gassy rectifiers. The effective potential is twice the potential of the x-ray tube, and this radiation is very penetrating. Radiation from the high voltage power supply can appear at any time. The only effective countermeasure is to shield the assembly that contains the rectifiers and to check at least twice a year for radiation leakage.

In addition to the six sources of radiation listed above, large quantities of primary radiation may be accidentally released as the result of removing parts of the system or improperly installing accessories. The only effective way to reduce the exposure of personnel is to immediately detect such conditions.

In installations in which you frequently disassemble the shielding components and change accessories, you should consider using an area monitor. This monitor should have audible and visible indicators that are activated when a pre-selected radiation level has been exceeded.

3.7.3 Choosing a Radiation Survey Meter (A3)

One of the major problems in carrying out a radiation survey program is obtaining a detector that has good energy independence at very low photon energies. ([6] p.105-127)

The type of instrument usually recommended for accurate measurements of exposure rates at low x-ray energies is an ionization chamber with a thin (1.0 mg/cm^2) plastic window. You may need to correct its calibration at low x-ray photon energy levels (<6.5 keV), but these energies are an important part of the spectrum only when you use an x-ray tube with an iron or chromium anode.

In general, for the accuracy required in radiation protection surveys, you will not be required to make corrections as long as the entire sensitive area of the detector is in a uniform radiation field. For this reason, the relatively large size of the ionization chamber (typically a diameter of 3.5 inches) is a disadvantage of this instrument. This disadvantage is because of the resulting low sensitivity when it is used to search for beams that have small cross-sections.

Instruments such as Geiger counters, scintillation detectors, and Si(Li) detectors offer high sensitivity in a small area. Generally, they are superior to ionization chambers for detecting beams that have small cross-sections. The responses of these instruments depend on the energy of the x-ray beam.

You must interpret the readings with care. Readings that you obtain with a Geiger counter in the soft x-ray range are particularly unreliable. However, since your primary need is to find and mechanically eliminate small beams by adding shields or correcting unsatisfactory assembly methods, the use of the simpler counters is quite acceptable for this purpose.

The primary beam generated by the TEC MAX is a small-volume beam. Whenever you use a large-volume detector to measure radiation emanating from the TEC MAX, you must use great care and make the necessary corrections.

3.7.4 Evaluating the Exposure Rate Due to Small Beams (A4)

3.7.4.1 Instrumental Methods (A4.1)

If an instrument is calibrated for radiation that would uniformly expose the entire active area of the instrument, it will give an erroneously low reading when it is exposed to a beam that has an area smaller than the active area of the instrument. ([6], p.155-162) The scale reading must be multiplied by the following factor:

$$f = \frac{\text{Area of Detector}}{\text{Area of Beam}}.$$

Around single-crystal diffraction equipment, it is common to encounter beams as small as 0.01 cm^2. You may need to make correction factors of 6000 or more for a 3.5 inch diameter ionization chamber. In such cases, it is difficult to detect beams in which the dose rate may be hundreds of times greater than permissible.

In order to resolve the difficulty in detecting and measuring the x-radiation from color television receivers ([4]), the NCRP accounted for the fact that the smaller the beam, the less the likelihood that the same area of an individual will be repeatedly or continuously exposed. The standard for home television receivers permits the dose rate to be averaged over an area of 10 square centimeters.

The same practice is acceptable for surveys around x-ray generator cabinets and system barriers. However, in the vicinity of x-ray tube housings, beam ports, collimators, and specimen chambers, where small, intense beams are likely to be encountered, it is recommended that you average the dose rate over an area no larger than 1 square centimeter.

We may assume that the largest correction factor that must be applied will be numerically equal to the sensitive area of the detector in square centimeters. In general,

$$\dot{X} = \dot{X}_{scale} \qquad\qquad \text{for } A_{beam} \mid A_{detector}$$

$$\dot{X} = \frac{A_{detector}}{A_{beam}} \qquad\qquad \text{for } 1 \text{ cm}^2 < A_{beam} < A_{detector}$$

$$\dot{X} = \frac{A_{detector}}{1 \text{ cm}^2} \cdot \dot{X}_{scale} \qquad\qquad \text{for } A_{beam} < 1 \text{ cm}$$

where $\dot{X}$ is the exposure rate and A is the area.

3.7.4.2 Film Methods (A4.2)

When you search for radiation leaks, a useful adjunct to the survey meter is x-ray film in a low absorption opaque envelope. With this method, you often can see recognizable shadows of features of the x-ray equipment, and thus can effectively locate the leak.

You can estimate the cross-section of the beam so you can correct the readings of a survey meter, or you can make the film method give a quantitative estimate of the dose if you have calibration data available. ([3], pp.185-212)

Another advantage of the film method is its suitability for integrating over a long period in order to detect very weak beams and for indicating the dose averaged over a time period that includes all the steps of a given procedure. The latter characteristic of the film sometimes leads to the detection of a transient situation that is possibly hazardous.

If you check equipment in the field where darkroom facilities may not be available, you should consider a cassette for use with high-speed film packets (ASA 3000). With this procedure, an air cushion forces the film into contact with a fluorescent screen during exposure. Exposure times are generally shorter than with conventional films. Development facilities are built into the cassette, so you can view the photograph after only 15 seconds.

3.7.4.3 Fluorescent Screen Method (A4.3)

Although fluorescent screens are occasionally useful, their low sensitivity generally limits their effectiveness to cases that involve a direct beam from the x-ray tube anode. Consequently, you should mount the screen on a long handle to minimize the risk of exposing your hands.

You should view the screen through a lead-glass shield. You can improve the sensitivity of the screen by darkening the room completely and allowing 20 minutes or longer for you eyes to become accustomed to the dark.

3.7.5 Calibration (A5)

The accuracy of any radiation detector is only as good as its calibration. You should periodically check all radiation survey meters against a reference instrument (either secondary standard or sub-standard) that has a calibration traceable to the NIST primary standard chamber. You should carry out this calibration check for all of the qualities of interest.

3.7.6 Using a Check Source (A6)

You should provide a check source to detect abrupt changes in calibration that result from deteriorating components or hidden damage. Iron-55, which decays by electron capture directly to the stable isotope manganese-55, is suggested for this application.

Iron-55, which emits the 5.9 keV x-ray characteristic of manganese-55, represents the extremely low energy range of the spectrum encountered in survey work. It also provides an excellent test of an instrument's capabilities in this most difficult range. A 50 μCi source is sufficient to provide exposures in the range 10-100 mR/h, and its half-life of 2.7 years is long enough to ensure a reasonably long useful life.

You should mount the source in a fixture that will ensure stable geometry. If possible, you should establish the exposure rate shortly after you have calibrated the survey instrument. Thereafter, it is necessary for you to correct only for the activity and, possibly, variations in air absorption due to changes in barometric pressure and relative humidity. The simplicity of the spectrum aids in making such corrections.

Using a check source also allows you to determine the stability of survey instruments. However, you may prefer to use a source of higher energy photons to avoid any possible influence of variations in air absorption.

3.8 Radiation Safety Training and Responsibilities

3.8.1 Training

Recommendations for installing and operating the equipment are not in themselves sufficient to guarantee adequate protection. Such protection depends largely on the expert knowledge of the staff. It also depends to a great extent on their cooperation in carrying out the instructions prepared by their supervisor for the purpose of protecting them from exposure to radiation.

The 16-mm film, "The Double Edged Sword," is excellent for training new users of diffraction and spectroscopic equipment. This film, which is 22 minutes in length, was produced by Durrin Films, Inc. under a contract with the National Institute of Standards and Technologies for the Bureau of Radiological Health.

You may purchase the film from the National Audiovisual Center (GSA), Washington, D. C. 20409. You may borrow it from Association Sterling Films, 600 Grand Avenue, Ridgefield, NJ 07657. A videotape version (3/4 inch or 1/2 inch cassette) is also available for free loan from the Training Resources Center (HFX-70) FDA, BRH, 5600 Fishers Lane, Rockville, MD 20852.

3.8.2 Injury Potential

X-ray diffraction analysis equipment generates high-intensity ionizing radiation. This radiation can cause severe and permanent injury if any part of the body is exposed to the primary beam, even for a few seconds.

In cases of accidental exposure, the most frequently reported injuries are severe burns that affect the upper extremities of the body. ([7], pp.245-249) These injuries are slow to heal, they can lead to cancer, and they sometimes require the amputation of one or more fingers. ([1], pp. 4 and 6)

Excessive exposure of the lens of the eye to x-rays can result in cataracts and other opacities. ([8], pp. 759-785) The damage may not become apparent until years later. You must also consider other injuries, such as genetic damage, that can affect the future offspring of irradiated persons.

3.8.3 Responsibilities — General

The owner or person in charge of a controlled area is responsible for evaluating needs and forming a radiation protection policy. This person is also responsible for the working conditions within the controlled area.

He or she is responsible for executing all requirements specified by the state department of radiological health for the use of the x-ray machine. He or she is also responsible for maintaining safe operating conditions and ensuring that personnel receive adequate medical examinations and radiation monitoring.

Each worker shall, upon the instruction of a qualified expert or responsible supervisor, follow the recommendations and instructions prepared for protecting workers from radiation. Each worker shall use the protective devices provided for him or her, and shall bring to the attention of those in charge any defect or deficiency in the radiation protection devices or procedures.

3.8.4 Responsibilities — Health Surveillance

The following guidance will help you develop or review a medical program for radiation workers. All personnel who are new to radiation work should have a

pre-placement medical examination. Notes should be made of the new worker's family history, previous occupational history, and previous x-ray diagnostic examinations or radiation therapy.

The pre-placement examination should include a complete blood count, which should measure the erythrocyte and leukocyte levels and a differential white blood cell count. The purpose of the examination is to determine the "normal" condition of the worker at the time of employment and to note any abnormalities that might later be confused with radiation damage.

In cases in which there has been previous occupational exposure, the examiner should record the total accumulated dose. Any appropriate additional medical examinations should be performed at this time. These examinations should include an examination of the skin and nails and an ophthalmologic examination with particular emphasis on changes in the lenses of the eyes.

You should ensure that adequate periodic medical examinations are conducted. The examiner should pay particular attention to the eyes and to the skin of the hands and face. Because of the nature of radiation hazards, you should not rely on blood counts and personnel monitoring alone because these might give rise to a false sense of security.

To ensure that no worker receives more than the maximum permissible dose, you should systematically check — with appropriate instruments — the doses received as a result of occupational exposures. (Refer to the sub-section "Choosing a Radiation Survey Meter (ANSI A3).")

If any worker is likely to accumulate, in any one calendar quarter, more than 25% of the maximum permissible dose per calendar quarter, then you shall keep an individual cumulative dose record for that worker. The following table gives the maximum permissible dose per calendar quarter for various body parts.

Table 1: Maximum Permissible Dose (MPD) Equivalent Values*

Exposed Areas	Maximum 13-Week Dose Rem[a]	Maximum Early Dose Rem[a]	Maximum Accumulated Dose Rem[a]
Control areas			
Whole body, gonads, lens of eye, red bone marrow	3	5	5(N-18)[b]
Skin (other than hands and forearms)	-	15	-
Hands	25	75	-
Forearms	10	30	-
Other Organs	5	15	-
			-
Non-controlled areas	-	0.5	-

Refer to the latest revision of the appropriate state/federal regulations for the acceptable maximum permissible dose equivalent values.

[a] For the purpose of this report, you may assume that the numerical value of the dose equivalent in rems is equal to the numerical value of the exposure in roentgens.

[b] N = age in years and is greater than 18. When the previous occupational history of an individual is not definitely known, you shall assume that he/she has already received the MPD permitted by the formula 5(N-18).

3.8.5 Personnel Monitoring

The deficiencies of personnel monitoring devices are well known. However, according to a report ([3]) in 17 cases of accidental exposure, the film badge gave the first indication that an accident had occurred in five of the cases. You must ensure that you use suitable personnel monitoring devices.

If the accumulated doses recorded for an individual exceed those recommended in Table 1, you should notify the person(s) exposed, the owner or person in charge of the controlled area, and any cognizant regulatory agency. You should make a report in keeping with the user organization's radiation safety rules and state regulations.

3.9 References

[1] *A Summary of Industrial Accidents in USAEC Facilities, 1965-1966.* United States Atomic Energy Commission, Division of Technical Information, Oak Ridge, Tennessee 37830 (TID-5360-Supplement-6).

[2] *Basic Radiation Protection Criteria.* National Council on Radiation Protection and Measurement, P.O. Box 30175, Washington, D.C. 20014, 1971 (NCRP Report 39), 135 pp.

[3] *Conference on Detection and Measurements of X-Radiation from Color Television Receivers.* United States Department of Health, Education and Welfare; and Electronic Industries Association; Bureau of Radiological Health, Washington, D.C. 20850, March 28-29, 1968 (Technical Papers), 365 pp.

[4] *X-Ray Protection Standards for Home Television Receivers*, Interim Statement of the National Council on Radiation Protection and Measurements (NCRP), Approved, February 23, 1968. Available from NCRP, 7910 Woodmont Avenue, Suite 1016, Washington, D.C. 20014.

[5] Attix, F. H. and Roesch, W. C. (Editors). *Radiation Dosimetry*, vol. 1-1968 (Fundamentals), vol. 2-1966 (Instrumentation) and vol. 3-1969 (Sources, Fields, Measurements and Applications). New York: Academic Press.

[6] Beu, K. E. (Chairman). *Safety Considerations in the Design of X-Ray Tube and Collimator Couplings on X-Ray Diffraction Equipment.* American Crystallographic Association, Polycrystal Book Service, P.O. Box 11567, Pittsburgh, PA 15238, 1962, (Apparatus and Standards Committee Report No. 1) 12 pp.

[7] Howley, J. R., and C. Robbins, *Radiation Hazards from X-Ray Diffraction Equipment*, Radiological Health Data and Reports, Vol. 8, No. 5 (May 1967).

[8] Merriam, G. R., and E. F. Focht, *Clinical Study of Radiation Cataracts and Relationship to Dose*, The American Journal of Roentgenology, Radium Therapy, and Nuclear Medicine, Vol. 77 (May 1957).

4 System Setup

The setup of the TEC MAX System consists of the following steps;

1) Determine which x-ray tube, diffractometer, and detector filters are required for the type of material to be measured.

2) Locate the MAX Controller close to the measurement point so the cable assembly when extended allows the diffractometer to sit directly above the measurement point.

Note: The MAX Cable Assembly should be placed carefully to ensure it is not damaged. Do not place it directly on sharp edges or allow it to be stepped on.

3) Connect the cable assembly to the diffractometer/x-ray tube selected and the front panel of the MAX Controller. Connect the USB cable from the host computer to the MAX Controller.

4) Connect the host computer and the MAX Controller to main power.

5) Connect the optical beam safety device to the MAX Controller. Place the optical beam safety device and reflector so that no personnel will be able to approach within three feet of the diffractometer without breaking the optical beam.

6) Make sure the diffractometer and the measurement point are aligned correctly. The exit port of the x-ray tube must be perpendicular to the sample surface at the measurement point. The distance is set correctly by the base mount when the sample surface at the measurement point is at the bottom of the base plate.

7) Turn on power to the host computer and allow it to boot up.

8) Turn on power to the MAX Controller.

9) Turn on the "X-ray Enable" key switch.

10) Start MAXAcquisition on the Host Computer and proceed with the measurements.

5 Making a Measurement

The process for taking a residual stress measurement involves both hardware and software. Basically, the operator sets up the diffractometer at the measurement point, then uses the software to take the measurement and to analyze the data.

Measurements are made using the MAXAcquisition software included with the system. The acquired data is then analyzed using MAX Analysis Manager, which provides the results and graphs in a printable format. It also allows for some changes to the measurement to improve the results,

The unit must have be calibrated prior to making a measurement, if it is not refer to Section 6.1

To make a measurement, the operator should perform the following steps:

1.   Set the diffractometer on the sample. Ensure that the setup is safe using the optical beam safety device or other means to prevent any personnel exposure to the x-ray beam, either direct or diffracted.

2.   Run MAXAcquisition and enter all parameters required. Start the acquisition and save the results.

3.   Run MAX Analysis Manager to obtain and/or print the results the results.

The following subsections provide detailed information on how to perform the above steps.

5.1   Diffractometer Setup

Place the diffractometer head directly over the measurement point. Align the head in the correct orientation for the measurement desired. The diffractometer may be held to the sample surface by a variety of means.

The surface of the sample must be parallel to the bottom plane of the bracket. The orientation of the diffractometer to the sample surface will determine the direction of the stress measurements.

> When the diffractometer is placed on the sample and before the measurement is made, ensure that the safety boundary is defined and the optical beam or optional method chosen is set up to prevent personnel from getting into the radiation area.
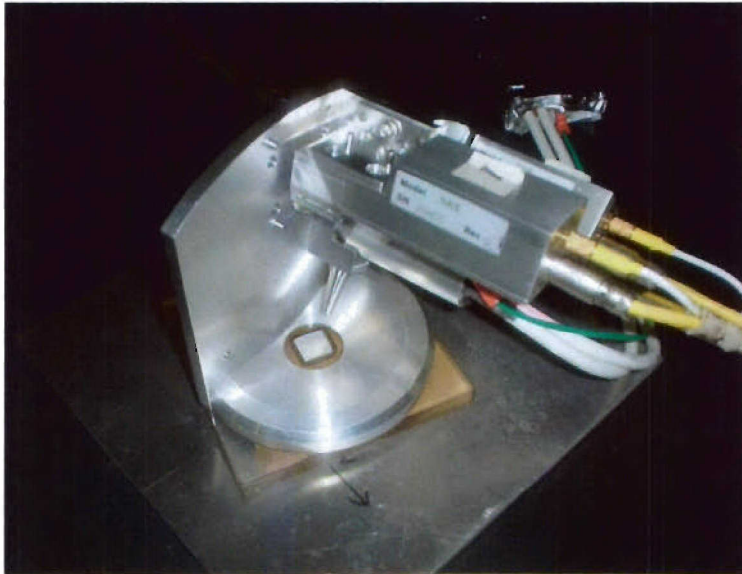
Figure 12 Max Diffractometer placed on sample

Make sure the MAX Cable Assembly is clear of obstacles and cannot be accidentally stepped on.

Power up the MAX Controller and ensure all cable connections are correct and tight.

Power up the MAX Host Computer and open the MAXAcquisition software.

5.2 MAXAcquisition

The MAXAcquisition software allows the operator to set up the MAX hardware and to collect spectrum data. When the program is opened, the Setup window is displayed.

5.2.1 Setup Tab
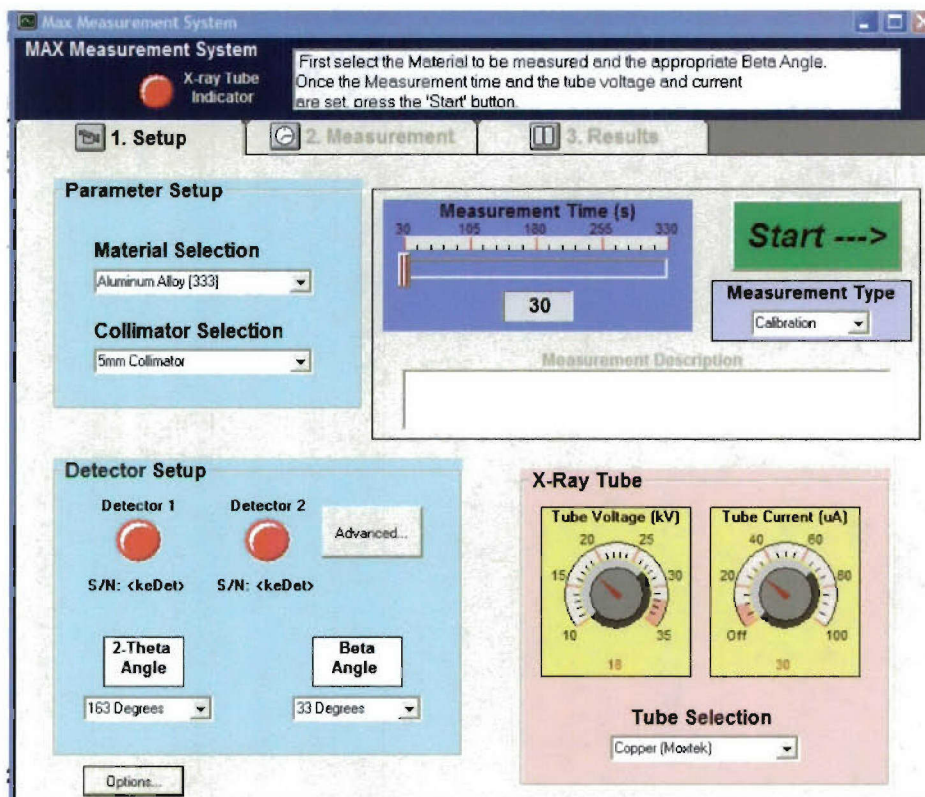
Figure 13 MAXAcquisition Setup Screen

5.2.1.1 Information Section

The information section includes the X-ray Tube Indicator lamp. This lamp will be lit RED when the tube is energized. There is also a text information window which informs the operator of current information, operating instructions, etc. The information displayed will change depending on tab selected.
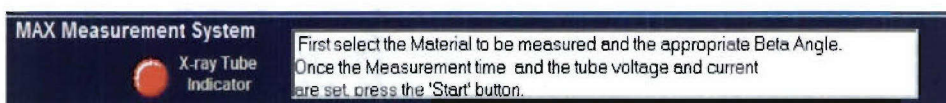
Figure 14 Information Section for Setup Tab
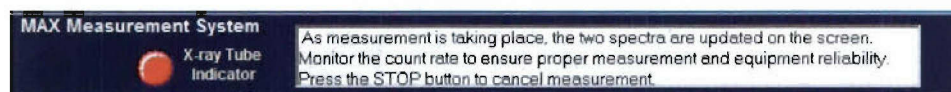
Figure 15 Information Section for Measurement Tab
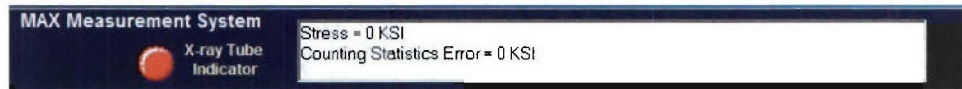
Figure 16 Information Section for Results Tab

5.2.1.2 Parameter Setup

The operator must enter the material to be measured and the size of the collimator mounted on the diffractometer.
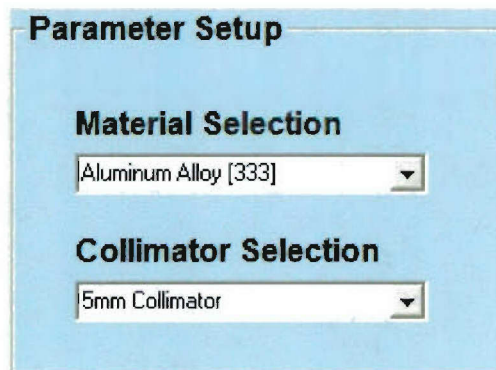
Figure 17 Parameter Setup Section

Material Selection allows operator to select the material being measured from the pull down list provided utilizing the down arrow.
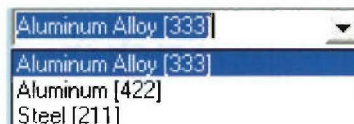
Figure 18 Material Selection Pull-down List

Collimator Selection allows the operator to choose from a pull down list of collimators to select for the measurement. Remember that the hardware does not sense the collimator size, so the selection must match what is physically installed on the diffractometer,

Figure 19 Collimator Selection Pull-down List

5.2.1.3 Detector Setup

The operator must correctly set up the detectors for operation as well as select the bracket dependent 2 theta angle and beta angle.
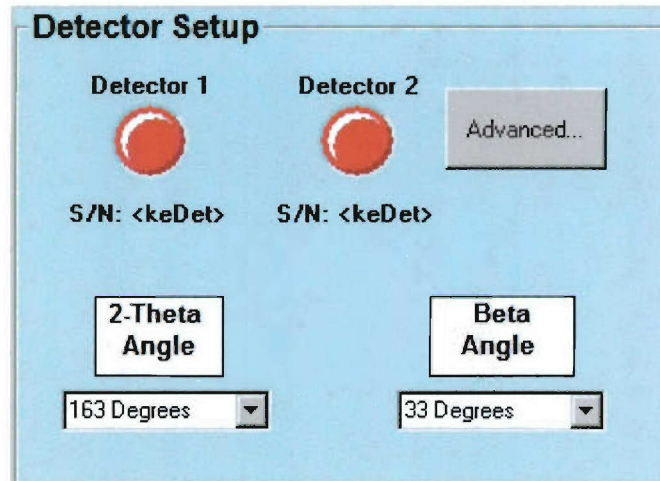
Figure 20 Detector Setup Section

When the system is correctly calibrated, the detector serial numbers will appear below the detector lamps. If the detectors are selected the Detector Lamps 1 and 2 will be lit. If they are not, ensure the USB cable is connected and the MAX Controller is powered up.

Select the 2 Theta Angle for the mounting fixture from the pull down list.
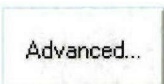
Figure 21 Detector Setup 2 Theta Angle Pull-down List

Select the Beta Angle for the mounting fixture from the pull down list.

Figure 22 Detector Setup Beta Angle Pull-down List

The Advanced Button [Advanced...] brings up the following window.
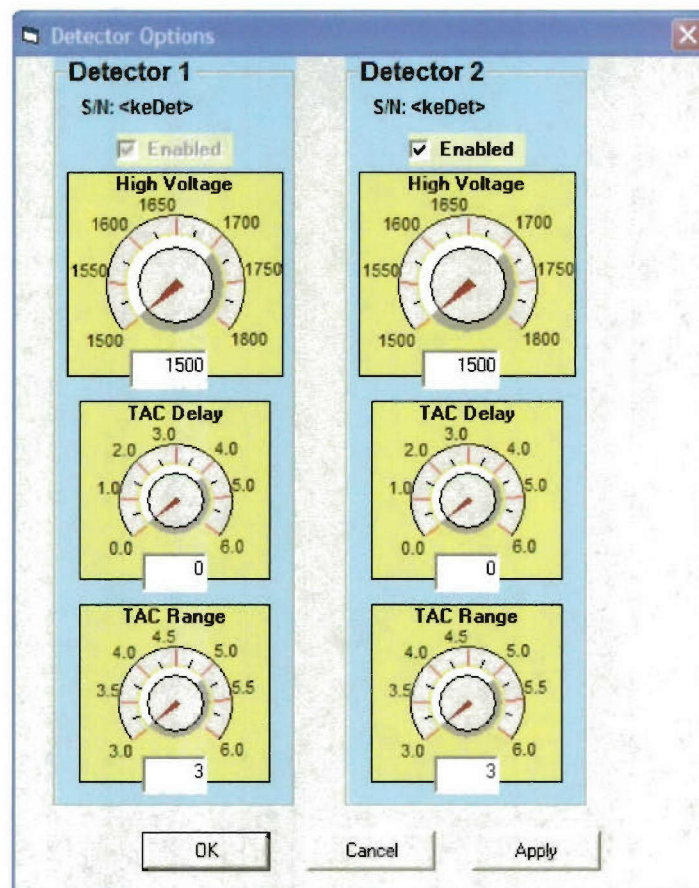
Figure 23 Detector Setup Advanced Detector Options

The Detector Setup Advanced Detector options panel should only be used during a measurement to enable the detectors. Any other adjustments after a system has been calibrated will invalidate the measurement data. See Section 6 for complete operation of this section during calibration.

5.2.1.4 Options Button

Clicking on the options button [Options...] brings up the following screen.
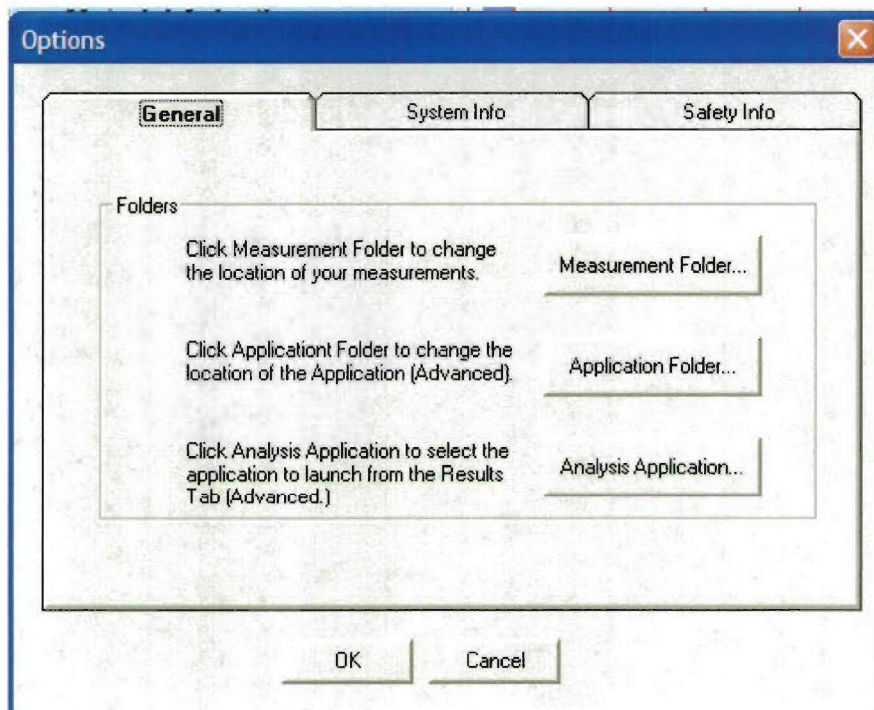
Figure 24 MAXAcquisition Options General Tab

Analysis Application

Look in: Analysis Manager

MaxAnalysis.exe

My Recent Documents

Desktop

My Documents

My Computer

File name: MaxAnalysis

Files of type: Executable Files (*.exe)

Open

Cancel

Options

General | System Info | Safety Info

Detector 1 Information

SN: FakeDet
Firmware Version: 0
Configured: True
Efficiency Done: False
Calibration Done: False

Update

Detector 2 Information

SN: FakeDet
Firmware Version: 0
Configured: True
Efficiency Done: False
Calibration Done: False

OK   Cancel

5.2.1.5 X-ray tube Setup

The operator uses the X-ray Tube Setup section to set the tube high voltage and current for the measurement. It also allows the tube selection.
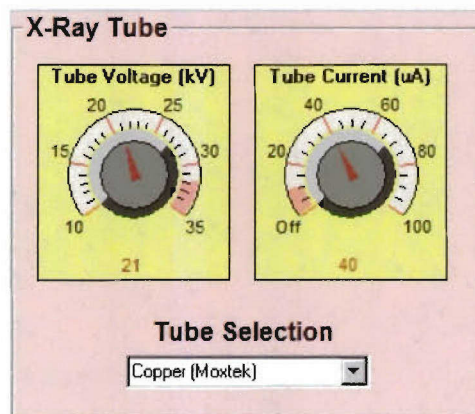
Figure 25 X-ray Tube Section

To set the Tube Voltage or Tube Current, position the mouse over the dial, left click and adjust to desired setting. The final setting will be indicated at the bottom of the dial. The red areas indicate areas where the tube should not be operated.

To select the proper tube, use the Tube Selection Pull-down list.

Figure 26 Tube Selection Pull Down List

5.2.1.6 Start Box

The Start Box allows the operator to start the measurement. It provides for setting the measurement time, the measurement type, and a description of the measurement. When the START button is pushed the screen changes to the Measurement Tab.
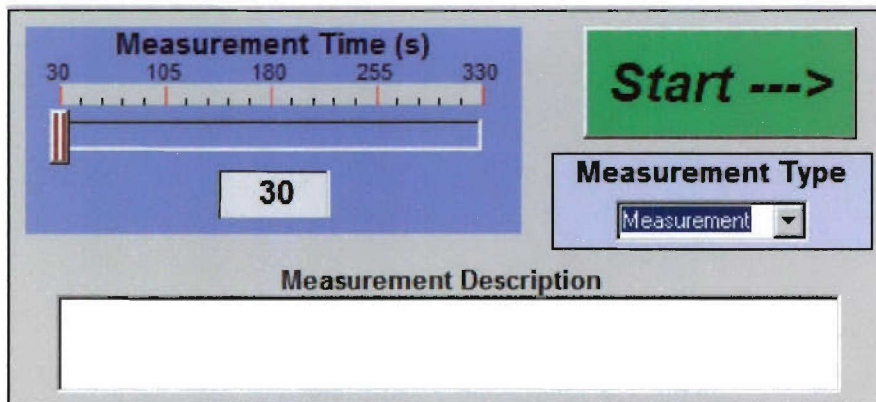
Figure 27 Start Box Section

Measurement Time (s) is entered by dragging the mouse on the slide bar to set the time from 30 to 330 seconds. The number selected is displayed in the lower window. The operator can also enter the desired time directly in the window.

Measurement Type is selected by the operator from a pull down list provided. Notice that when any other measurement type is selected other than measurement, the software will automatically set some parameters such as the high voltage and current settings for the x-ray tube.

Figure 28 Measurement Type Pull-Down List

Measurement Description. Enter a description of the test you will perform. This field, which is for your information, should contain enough identifying details for you to recognize the sample later. The minimum information you should include

is the sample name and/or identifying number, and the location on the sample and direction of the measurement.

Divide a long description into separate lines so it will fit on the printed report (the report generator will attempt to place the information on a single line otherwise). To do this, press the **ENTER** key to force a hard return in the text.

The Start Button should be pushed after all parameters are entered. This will start the measurement and the system will collect a data spectrum.

5.2.2   Measurement Tab

The Measurement tab appears automatically when the measurement is started.
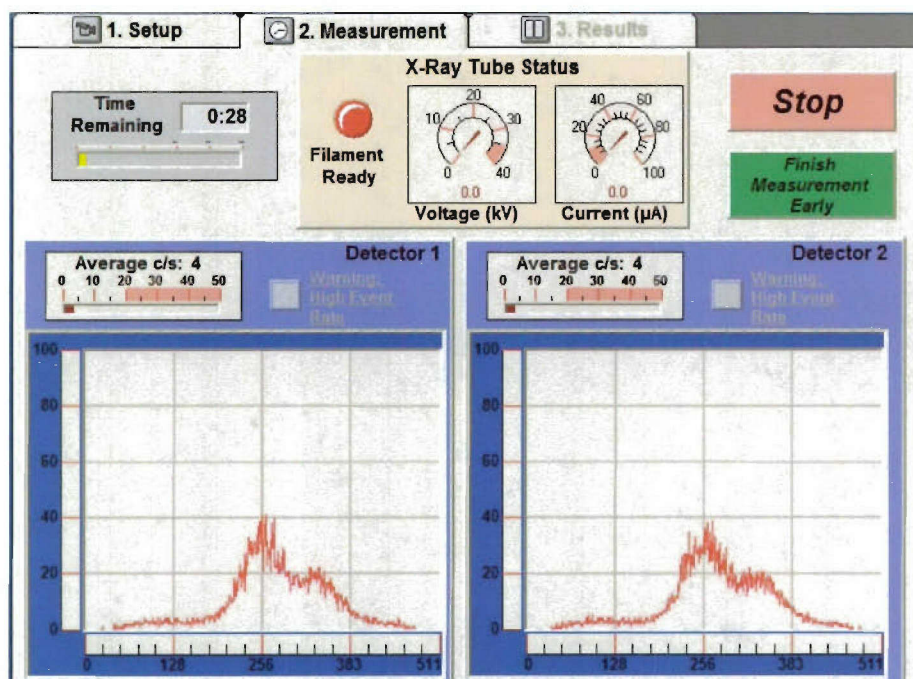
Figure 29 MAXAcquisition Measurement Window

While the data collection is taking place, the Time Remaining section shows a progress bar and the time remaining in minutes and seconds.

Figure 30 Time Remaining Section

The X-ray Tube Status section indicates the monitored high voltage and current readings and has a Filament Ready indicator lamp
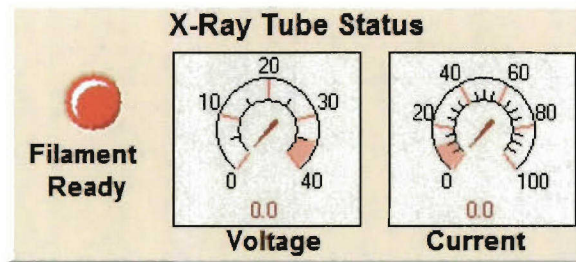
Figure 31 X-Ray Tube Status

The Detector 1 and 2 Spectra Display windows show the accumulating data spectra. It displays the average counts per second and has a warning lamp if the count rate exceeds the recommended rate of 20 cps.
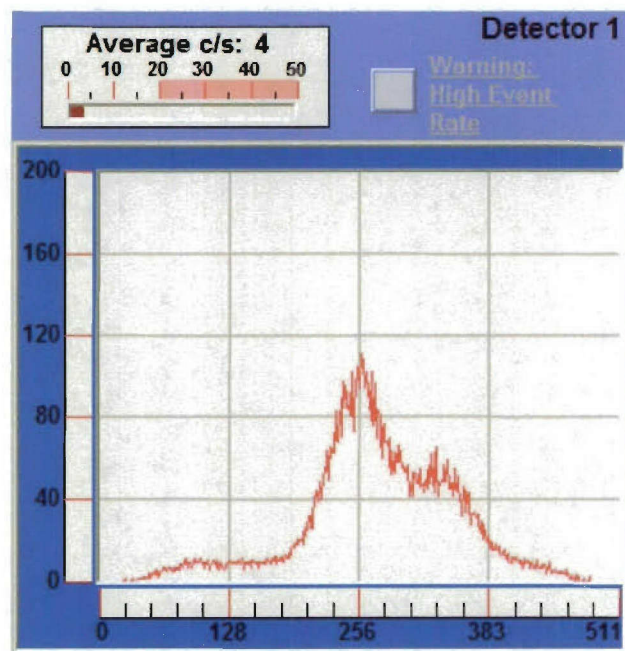
Figure 32 Detector 1 Spectra Display Window

While the data is being collected, the operator may elect to stop the run completely by pressing the stop button **Stop**. This action takes the operator back to the setup tab.

The operator may instead press the Finish Measurement Early button **Finish Measurement Early** which will cause the measurement to terminate normally but at a shorter time period.

When the measurement completes it open the standard windows save dialog and allows the operator to save the spectra as auto-named by the software or with the customers preference.
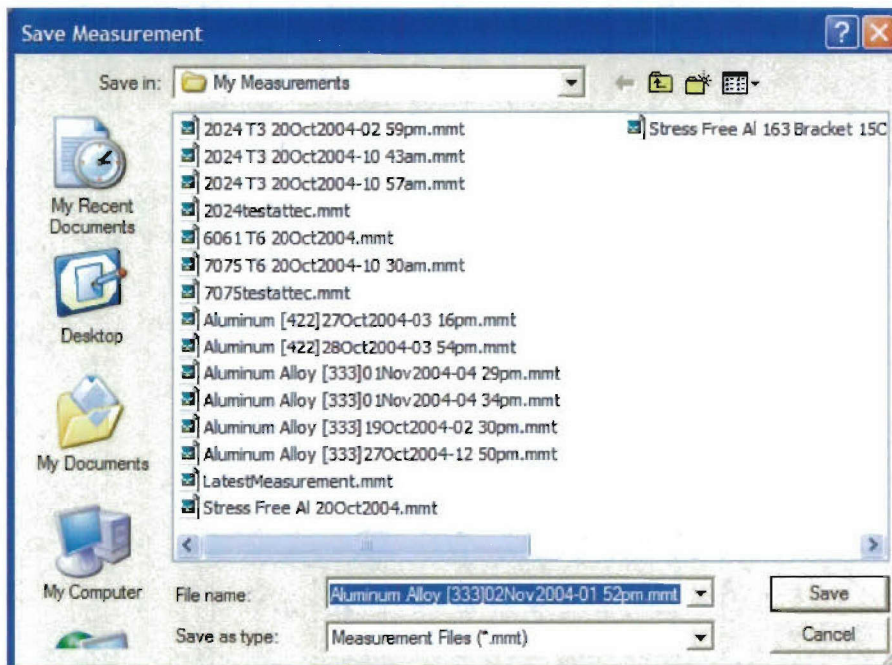
Figure 33 Save Measurement Dialog Box

5.2.3 Results Tab

The Results Tab will appear automatically when the measurement has completed.
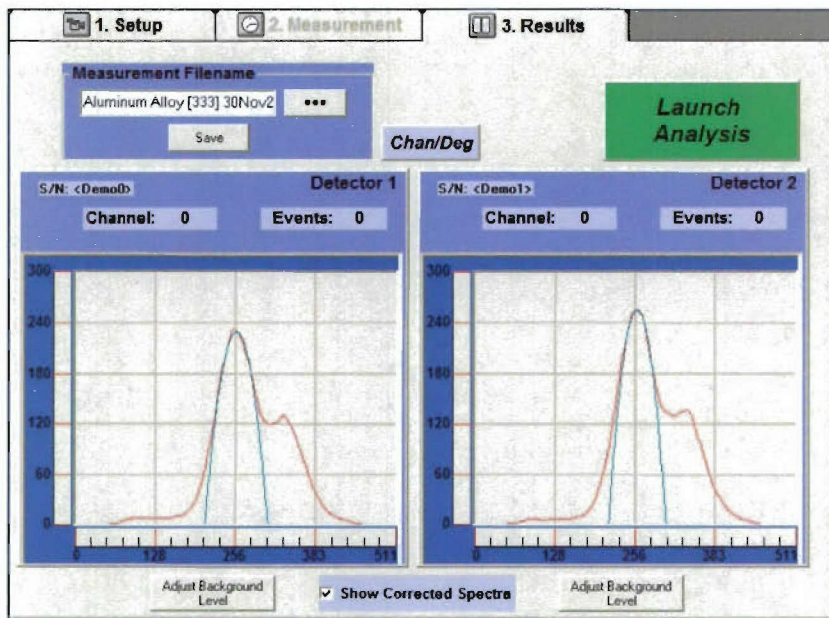
Figure 34 MAXAcquisition Results Window

When the measurement has completed, the operator can store the spectra at a different location than previously selected using the Measurement Filename section. The new file name can be entered directly in the window. If a different directory is desired, it can be selected by clicking the [•••] button.

Figure 35 Measurement Filename Section
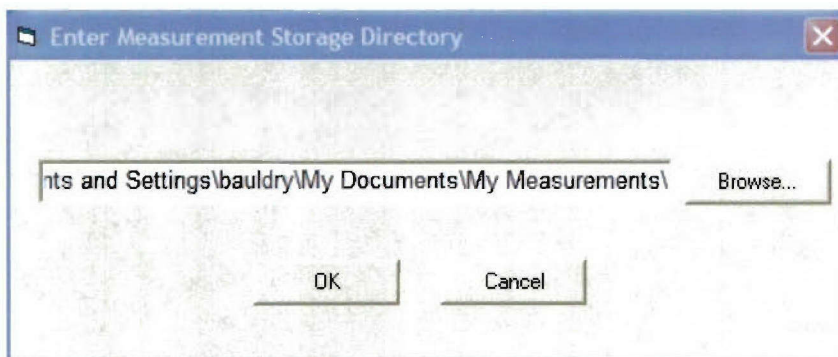
Figure 36 Enter Measurement Storage Directory Window

Clicking the [Browse...] will open the Browse for Folder window.

Figure 37 Browse for Folder Window

After naming the file and selecting a folder, the operator clicks the [Save] button to save the file.

The channel 1 and 2 spectra displays show the operator the final data spectra.
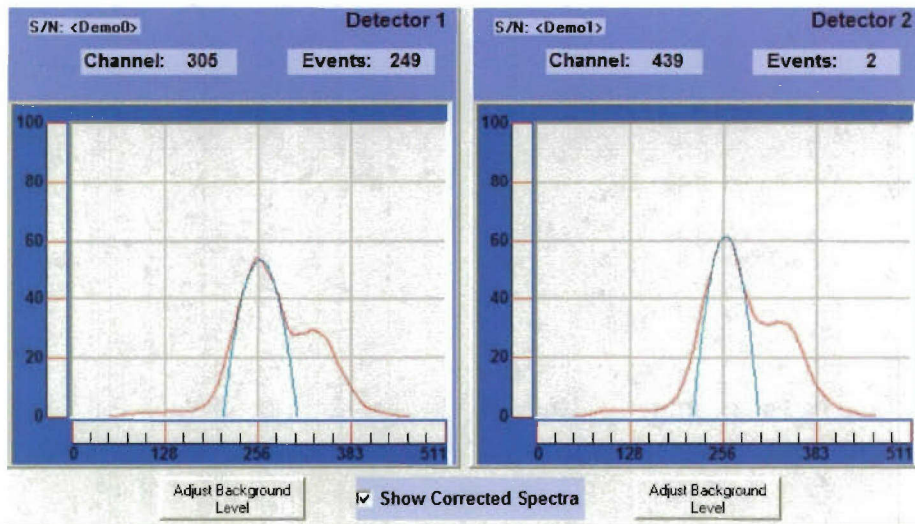
Figure 38 Detector Spectra Display

The operator may examine the data using the mouse to position a cursor on the display, The displayed data can be shown in channel numbers or degree 2 theta by clicking the **Chan/Deg** toggle button.

The operator can perform some limited data manipulations by using the buttons at the bottom of the Results Window.

The Show Corrected Spectra check box ☑ **Show Corrected Spectra** allows the operator to view either the raw or corrected spectra as shown below. This will only appear if the system has been calibrated.
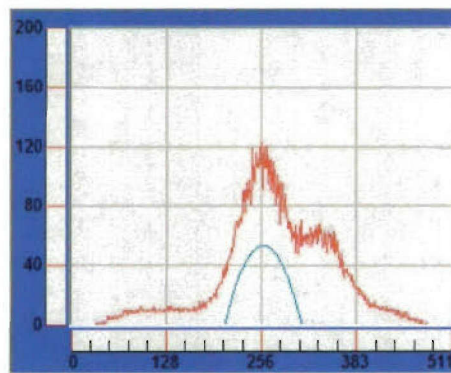
Figure 39 Uncorrected Spectra
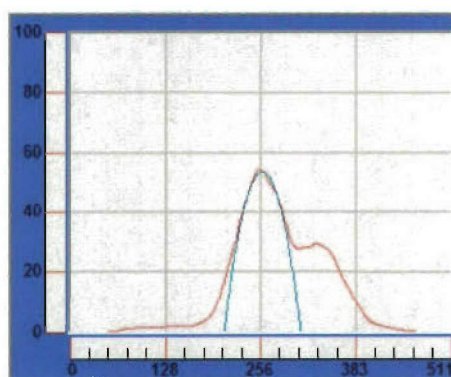
Figure 40 Corrected Spectra

If the operator wishes to make adjustments to the spectra background, he can press the Adjust Background Level button, Adjust Background Level This will display the Background Subtraction window. This function is only enabled for calibrated system.
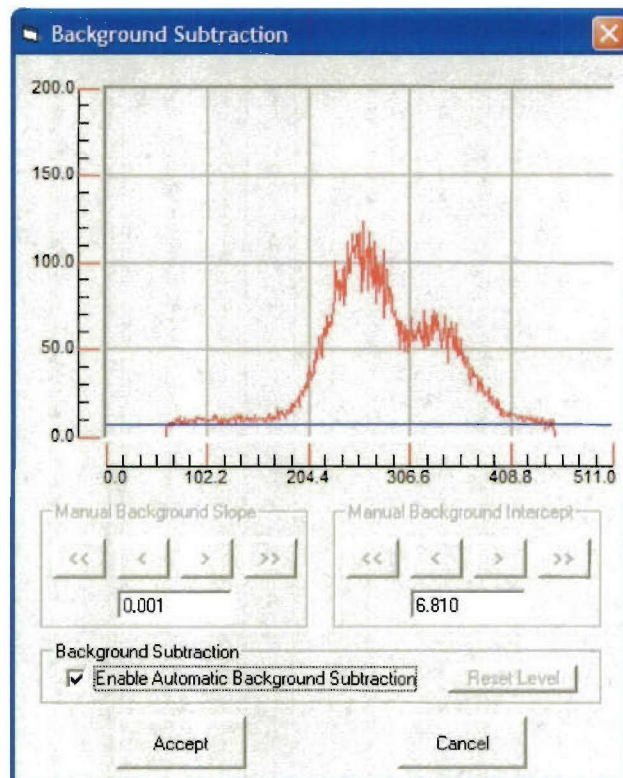
Figure 41 Background Subtraction Window

If the operator desires to change the background subtraction manually, he first deselects the Enable Automatic Background Subtraction. This enable the Manual Background Slope and Intercept sections. The operator can then adjust the background to his requirements. He then presses the Accept button to have his selection used in the spectra peak fitting.

After the file is saved, the operator can launch MAX Analysis Manager by clicking the Launch Analysis button.

Figure 42 Launch Analysis Button

5.2.4 Error Messages and Displays

The following are error messages and displays that may appear while running MAXAcquisition, A brief explanation accompanies each error.
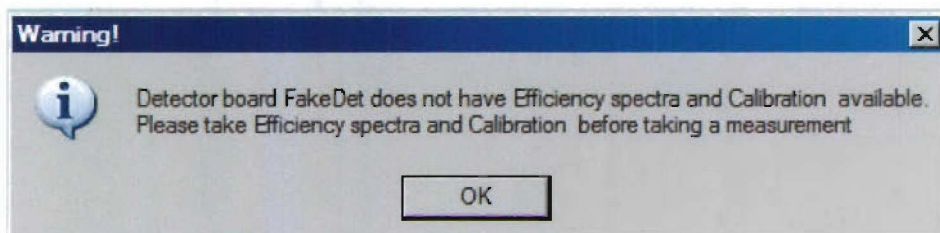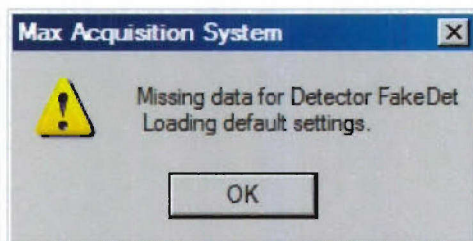
5.3 MAX Analysis Manager Program

In order to analyze data and obtain results, the operator must run the MAX Analysis Manager program. This can be launched from MAXAcquisition after a completed measurement or separately from its icon. When the program is launched the main window will appear.
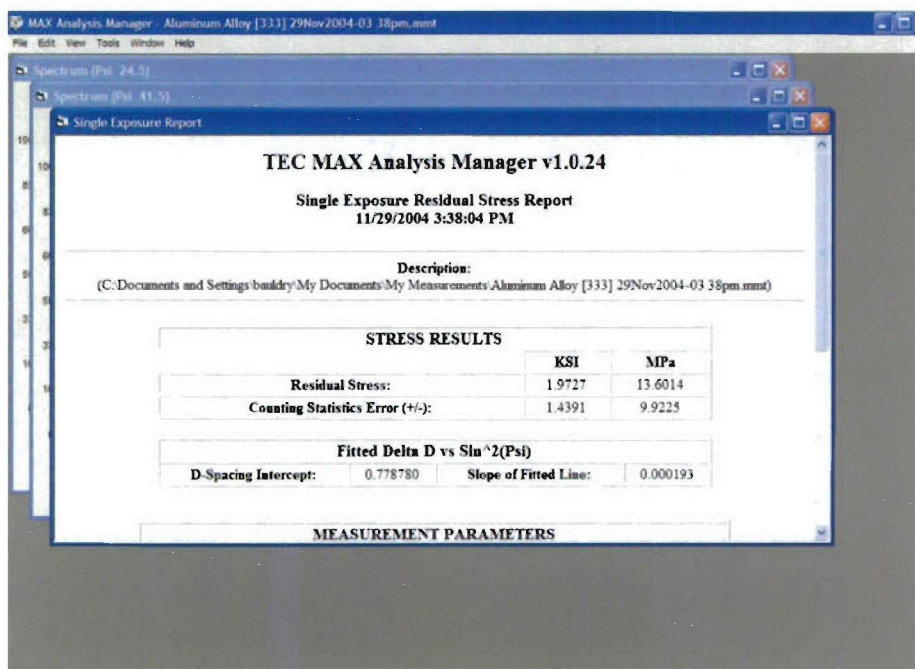
Figure 43 MAX Analysis Manager Main Window

Once a measurement run is completed the results page and the two spectrum pages will be displayed as in Figure 43 MAX Analysis Manager Main Window. If desired, the operator can print the pages, examine the data, or close the program to return to MAXAcquisition.

The following sections describe the MAX Analysis Manager Menu and options available to the operator.

5.3.1 File Menu
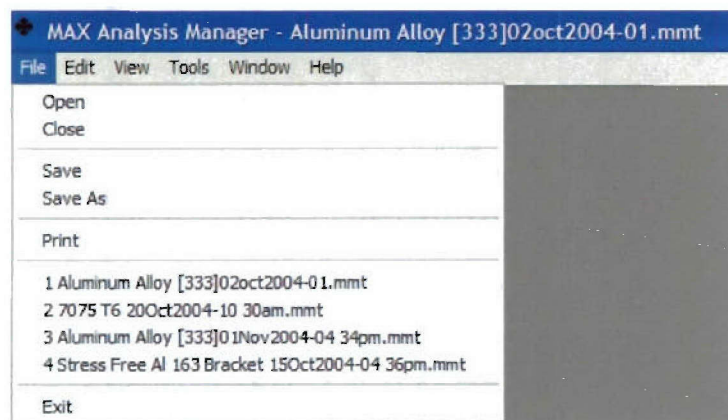
Figure 44 MAX Analysis Manager File Menu

The FILE menu allows you to open and close data files, save measurements in new data files, print spectra, graphs, and reports. You can access the following commands from the FILE menu.

5.3.1.1 File | Open

The FILE | OPEN command, which generates the standard Windows OPEN dialog box, allows you to open existing data files. Typically, existing measurement data files – which have suffixes such as *.mmt, – are stored in the storage directory selected by the operator.
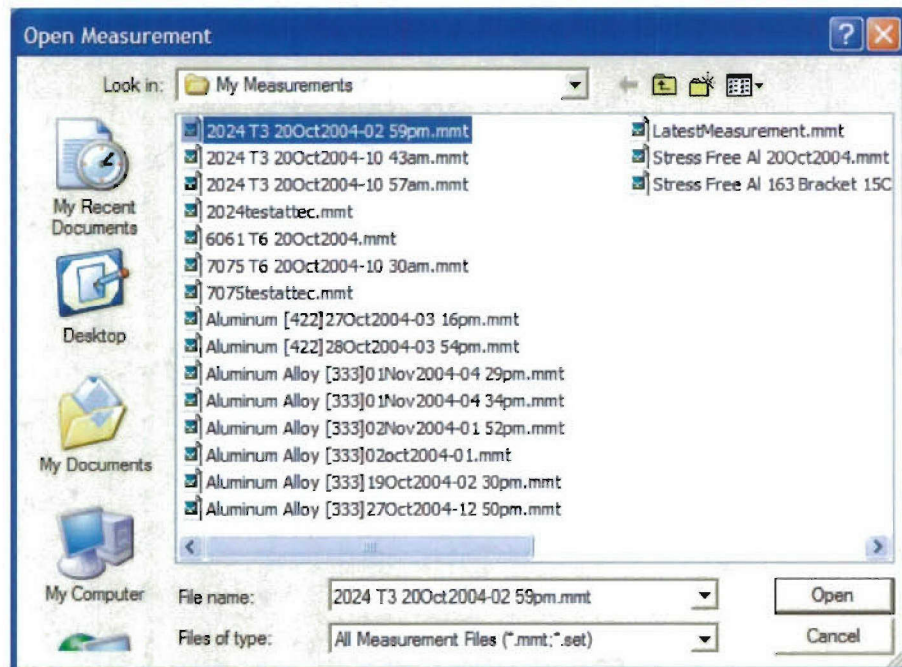
Figure 45 MAX Analysis Manager File | Open Window

Highlight the file you wish to open and click the OPEN button. If the name of the file you wish to open is not visible in this window, browse in your directory tree for the folder that contains your file.

If another file is currently open when you click the OPEN button, **MAX ANALYSIS MANAGER** displays a dialog box that asks you if you wish to save the current measurement. If you do not wish to save the file, click the **No** button. The newly opened file will replace it in your workspace.

If you need to save the file, click the **YES** button. The standard SAVE AS dialog box will appear to allow you to save the file. After the file is saved, the newly opened file will replace it in your workspace.

5.3.1.2 File | Close

Select FILE | CLOSE command to close the current *measurement* file. The **MAX ANALYSIS MANAGER** main window remains open.

> **NOTE:** *If you wish to close an active window, click the X in the upper-right corner of the window. To close all open windows, select WINDOW | CLOSE ALL. To close MAX ANALYSIS MANAGER, select FILE | EXIT or click the X on the MAX ANALYSIS MANAGER main window.*

If you have made changes to the file, **MAX ANALYSIS MANAGER** will prompt you to save the file.

Click the YES button if you wish to save the changes to the existing file. Click the No button if you wish to discard changes

5.3.1.3 File | Save

The FILE | SAVE command allows you to save changes you have made to a file. When you save changes, the file that contains your changes will replace the existing file.

> **NOTE:** *As with most software, when you make changes to an existing file then select FILE | SAVE, MAX ANALYSIS MANAGER overwrites your existing file with new data. To ensure you preserve the integrity of your original measurement data, ALWAYS keep your original file and make changes to a COPY of the original.*

Select FILE | SAVE. (If you are working with a measurement that has never been saved, the SAVE AS dialog box will appear on your screen.)

In the FILE NAME: text box near the bottom of the dialog box, type the name you wish to give the file.

If you wish to save the file in a directory different from the one shown in this box, use the folder buttons at the top of the box to move up and down the directory tree.

Click the SAVE button to save the file.

Click the CANCEL button to exit out of the FILE | SAVE command.
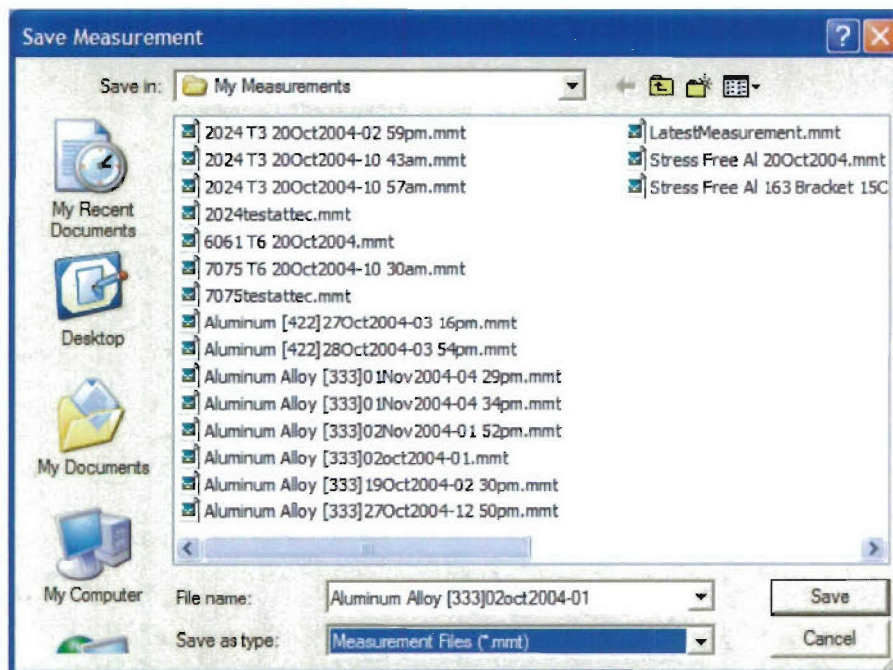
Figure 46 MAX Analysis Manager File | Save or | Save As Window

5.3.1.4 File | Save As

The FILE | SAVE AS command allows you to save a file for the first time or to save the file with a new name.

1.   Select FILE | SAVE AS.

2   In the FILE NAME: text box, enter the name you wish to give the file.

3   Be sure the SAVE IN text box shows the correct folder. If you wish to change folders, use the folder buttons to move up and down the directory tree.

4   Click the SAVE button to save the file.

Click the CANCEL button to exit out of the FILE | SAVE AS command.

5.3.1.5 File | Print

The FILE | PRINT command allows you to print data analysis, and spectra. When you execute this command, it prints the currently active window *only*.
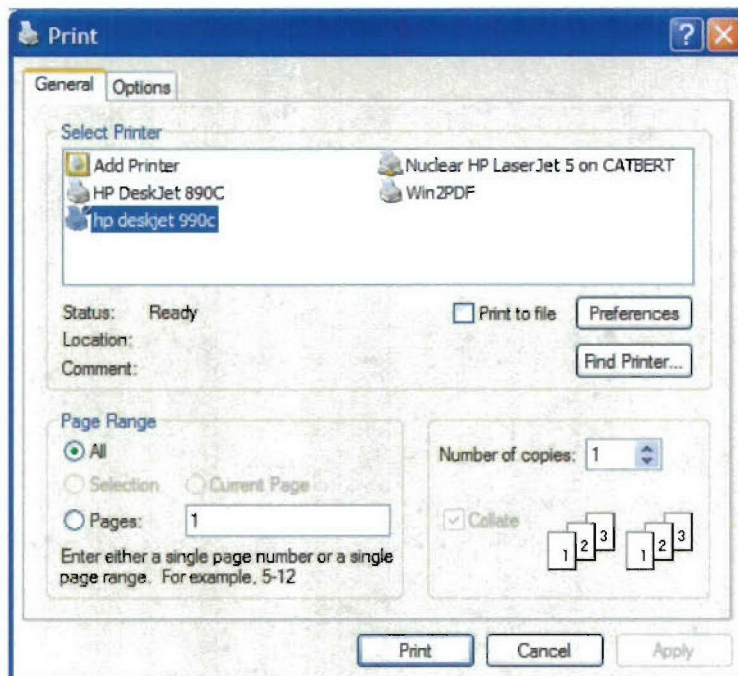
Figure 47 MAX Analysis Manager File | Print Menu

5.3.1.6 File | [Recent Files List]

This standard Windows item lists the most recently opened files. Click on the name of the file that you wish to open.

5.3.1.7 File | Exit

This command allows you to exit **MAX ANALYSIS MANAGER**. If you are in the process of taking a measurement, or if an open file contains any unsaved data, **MAX ANALYSIS MANAGER** will prompt you to save the current measurement data.
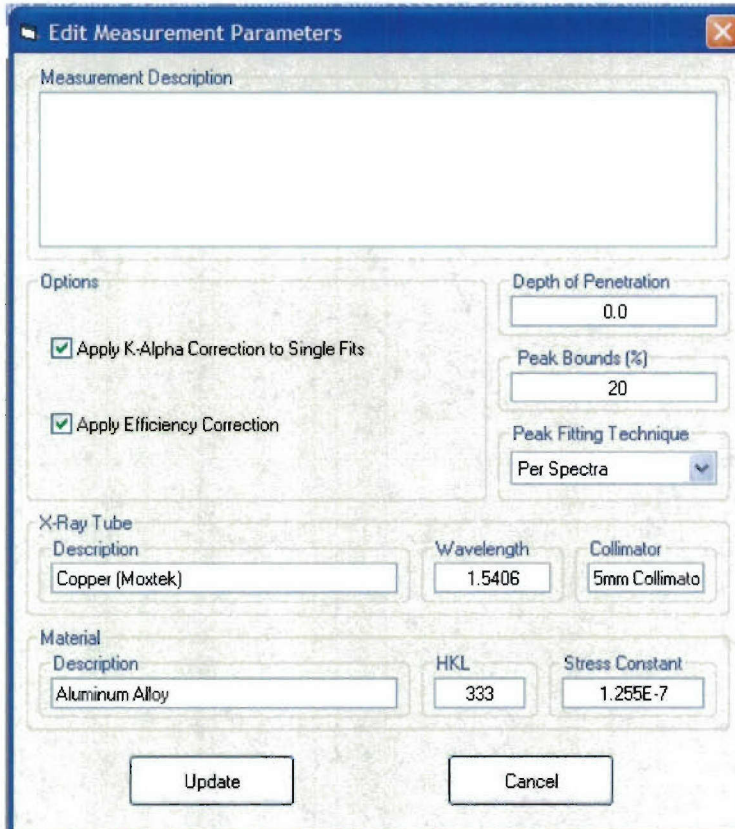
5.3.2 Edit Menu

Figure 48 MAX Analysis Manager Edit Menu

5.3.2.1 Edit | Measurement

The Edit | Measurement command allows the operator access to the file parameters of the opened file. The operator can make changes to the parameters and update the file by clicking the [Update] button.

Figure 49 MAX Analysis Manager Edit | Measurement Window

5.3.3 View Menu

The View Menu allows the operator to view the reports and graphs.
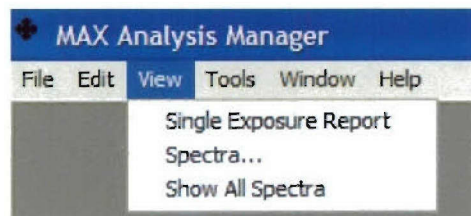
Figure 50 MAX Analysis Manager View Menu

5.3.3.1 View | Single Exposure Report

This command calls up on the screen the Single Exposure Residual Stress Report.
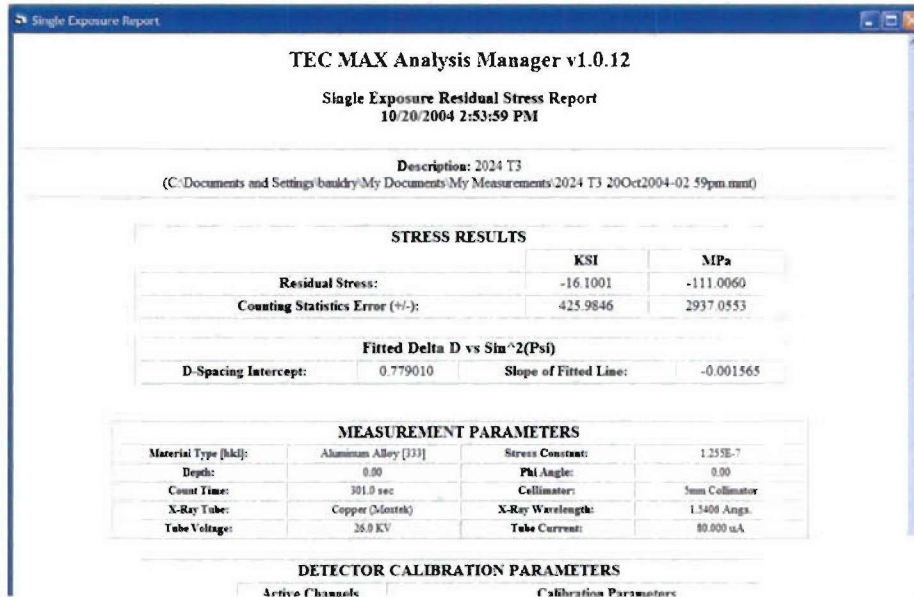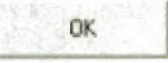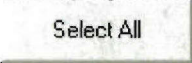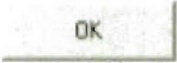
Figure 51 Single Exposure Residual Stress Report

5.3.3.2 View | Spectra

The View | Spectra command opens a selection window which allows the operator to select one or all of the spectra associated with the current open measurement to be displayed. Use the cursor to highlight an individual spectra to be displayed and click the [OK]. The operator can click on the [Select All] button to highlight all listed spectra and then press the [OK]. If the operator does not want to open a spectrum, he can click on the [Cancel] button.
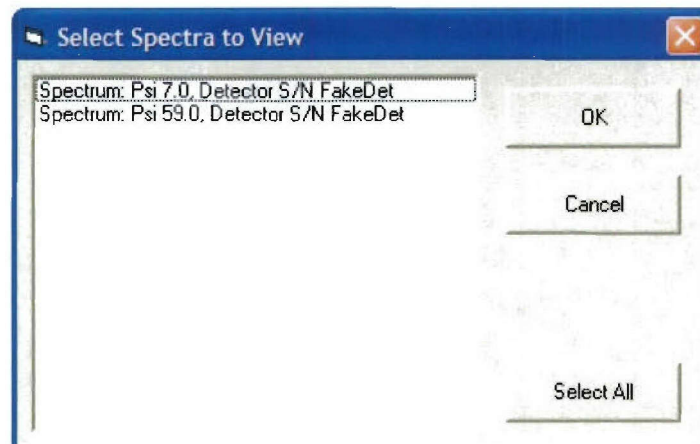
Figure 52 View | Spectra Selection Window

The below figure is a typical Spectrum View.
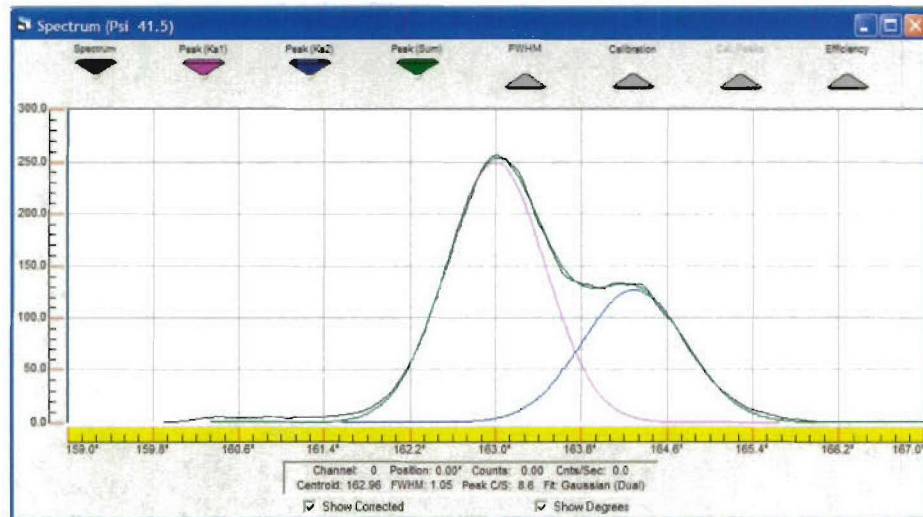
Figure 53 Typical Spectrum View

5.3.3.3 View | Show All Spectra

This command will display all spectra views for the currently opened file.

5.3.4 Spectrum Menu
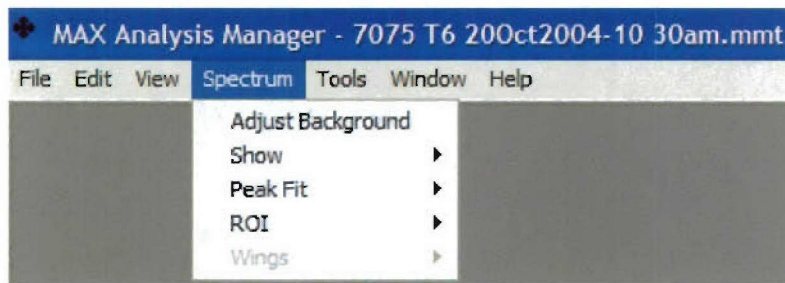
Figure 54 MAX Analysis Manager Spectrum Menu

5.3.4.1 Spectrum | Adjust Background

The Spectrum | Adjust Background opens the Background Subtraction window. This allows the operator to manipulate the background levels on the highlighted spectrum in an attempt to improve the results. If the Enable Automatic Background Subtraction checkbox is selected, the manual functions for slope and intercept are grayed out. Clicking on the ⟦Reset Level⟧ button restores the background subtraction configuration from the last time the file was stored.
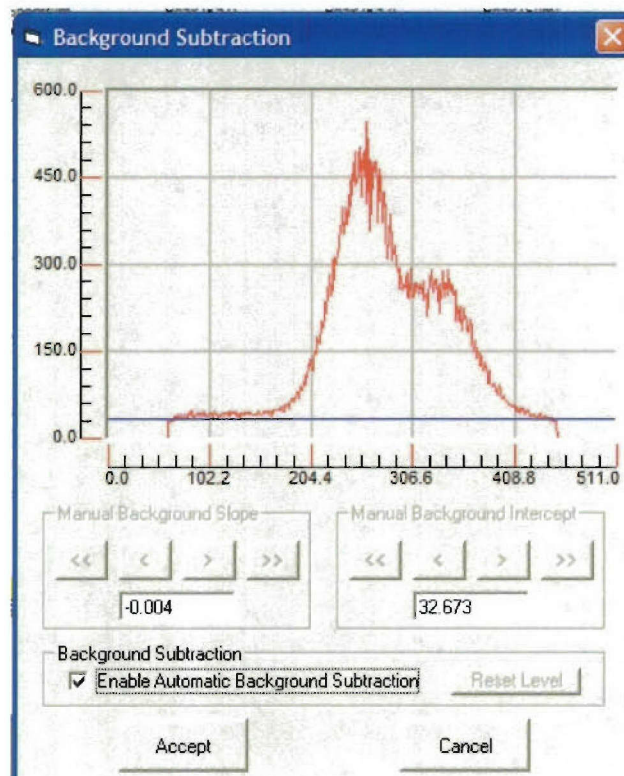
Figure 55 MAX Analysis Manager Spectrum | Background Subtraction Window

5.3.4.2 Spectrum | Show

The Spectrum | Show menu allows the operator to display various qualities of the spectrum. The operator can chose from measurement spectra data, calibration data spectra, or to toggle between degrees and channels displayed on the spectra. All the Spectrum | Show functions are also available on the spectrum display as toggle switches .

Going from to performs the same function as Spectrum | Show | Measurement | Spectrum.
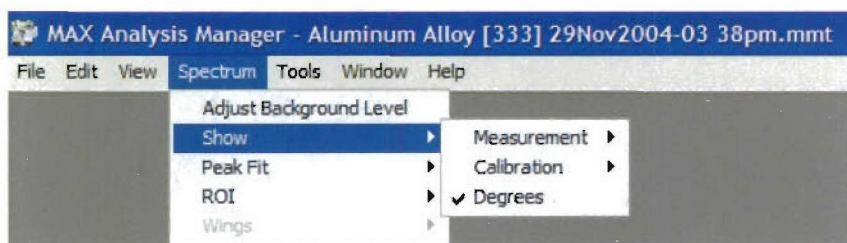
Figure 56 MAX Analysis Manager Spectrum.| Show Menu

5.3.4.2.1   Spectrum | Show | Measurement

This function allows the operator to display the Spectrum, the Corrected Spectrum, the FWHM (full width half max) and the Peaks on the spectrum display.
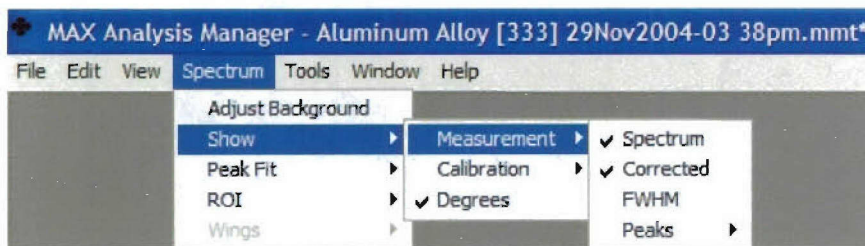
Figure 57 MAX Analysis Manager Spectrum | Show | Measurement Menu

5.3.4.2.1.1   Spectrum | Show | Measurement |Peaks
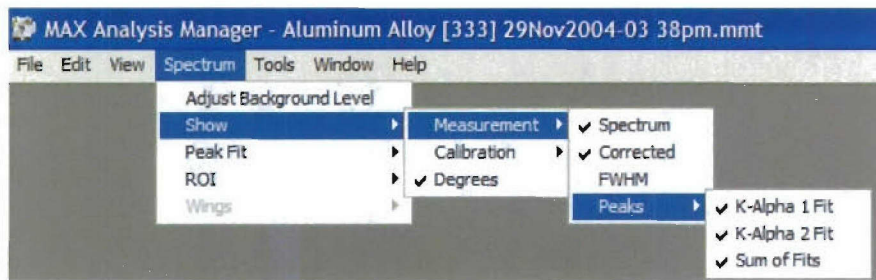
Figure 58 MAX Analysis Manager Spectrum | Show | Measurement | Peaks Menu

This function allows the operator to display various information on the Spectra display. He can select to display, the K-Alpha 1 Fit, the K-Alpha 2 Fit, and/or the Sum of Fits. Each fit is displayed in a different color for ease of viewing.

5.3.4.2.2 Spectrum | Show | Calibration

This function allows the operator to display the Calibration Spectrum (scaled), the fitted Peaks (scaled) of the calibration spectra, and the Efficiency Response Spectrum of the calibration on the same screen as the measurement data.
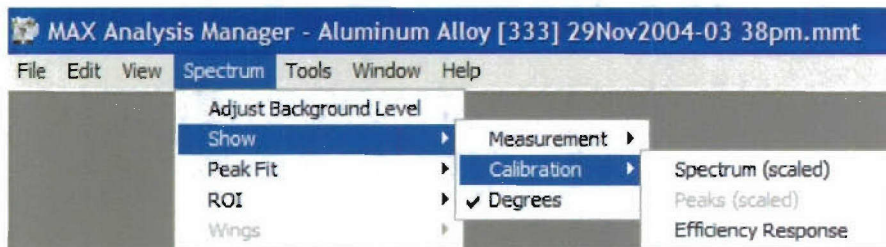
Figure 59 MAX Analysis Manager Spectrum/Show/Calibration Menu

5.3.4.2.3 Spectrum | Show | Degrees

This function allows the operator to change the x axis display from degrees two theta to channel numbers.

5.3.4.3 Spectrum | Peak Fit

Currently the Spectrum | Peak Fit function allows the operator to select either parabolic peak fitting, Guassian peak fitting, Guassian (dual) peak fitting, Cauchy peak fitting, Cauchy (Dual) peak fitting, Pearson VII peak fitting, Pearson VII (dual) peak fitting, or automatic selection of peak fitting. The selected technique will be applied to the current data.
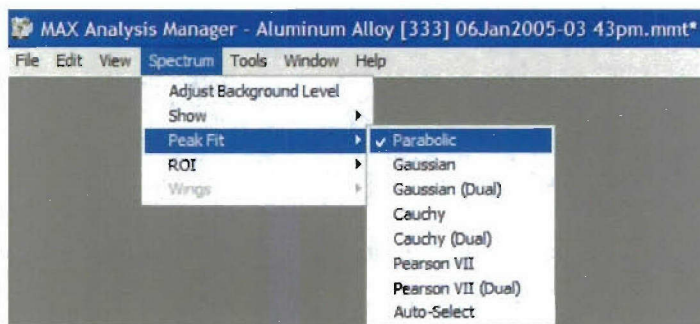
Figure 60 MAX Analysis Spectrum | Peak Fit Menu

5.3.4.4 Spectrum | ROI

This function allows the operator to set a region of interest (ROI) on the spectra display to limit the area used to determine the peak location. The operator positions the cursor on the display and selects Spectrum | ROI | Set Left. A marker is placed on the spectra at that location and the bottom line shows the area excluded. The operator then positions the cursor on the display and selects Spectrum | ROI | Set Right. A marker is placed on the spectra at that location and the bottom line shows the area excluded. The analysis will now only consider the area inside the region of interest, This is most useful for spectra that have multiple peaks to insure the correct peak is used for analysis.
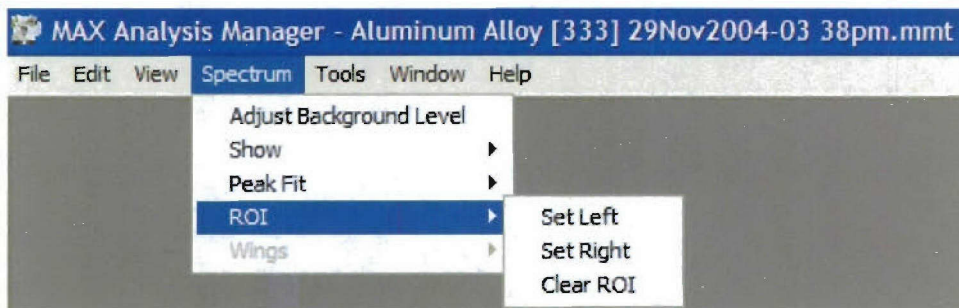
Figure 61 MAX Analysis Manager Spectrum | ROI Menu

5.3.4.5 Spectrum | Wings

5.3.5 Tools Menu

The Tools menu allows the operator to set several options that control what is automatically displayed when MAX Analysis Manager is run. It also allows the operator to select the color scheme he displays on the screen.
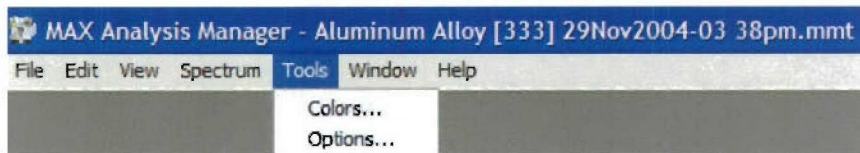
Figure 62 MAX Analysis Manager Tools Menu

5.3.5.1 Tools | Colors

The colors menu item, when selected, opens the Color Selection dialog box. The operator may select and change the color of the displayed items. Once the item

is highlighted, pressing the Edit Menu button [Edit Color] will bring up the color choice dialog box.
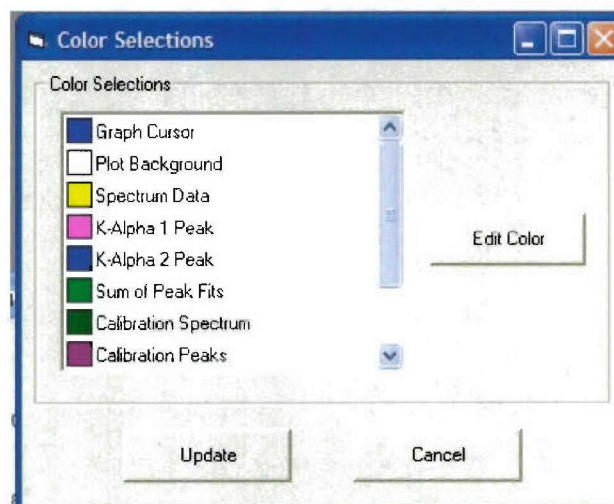
Figure 63 Color Selection Dialog Box

The operator select a fixed color or clicks on the color from the large chart. He can press Update [Update] to implement the change or cancel [Cancel] to leave the dialog box.
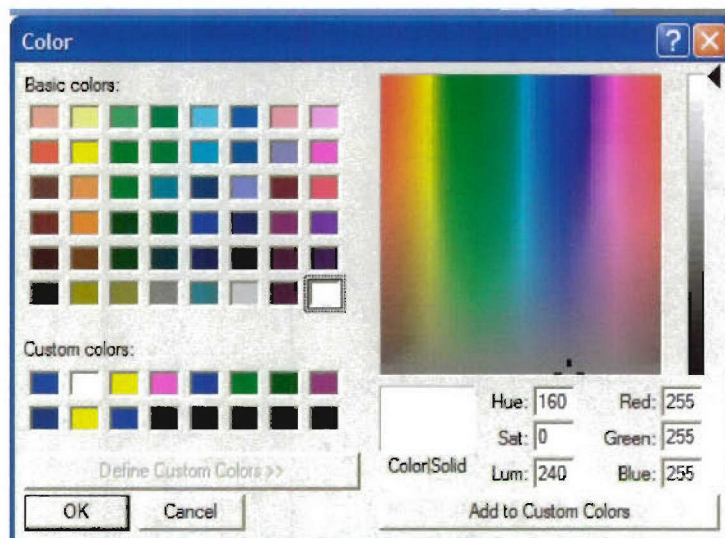
Figure 64 Color Choice Dialog Box

The operator in the Options dialog box can select to Auto Show Report, Auto Show All Spectra, Show Peak Fit, and Show Degrees. If Show Peak Fit is selected, the operator can choose to display K-Alpha 1, K-Alpha 2, Sum of the two in any combination. These selections will become the defaults when a new measurement file is opened.
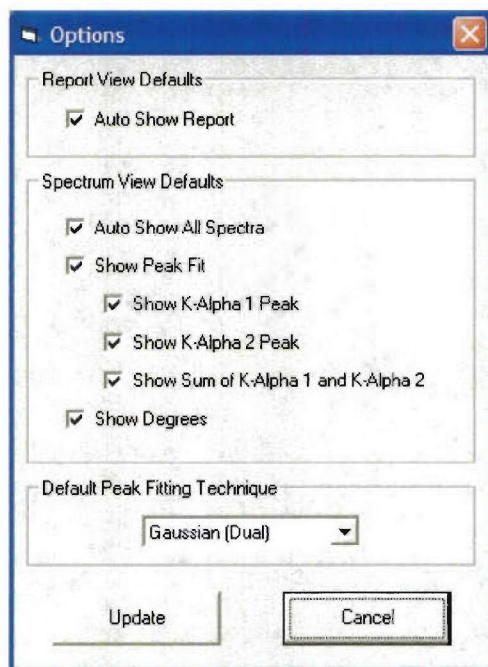
Figure 65 Tools | Options Dialog Box

The operator can also select the Default Peak Fitting Technique from the pull down menu.

Figure 66 Default Peak Fitting Technique Pull Down Menu

Once the operator has made his selection, he can press the Update button

 to implement his selections or the Cancel button to leave the selections unchanged.

5.3.6 Window Menu

This menu item allows you to manage various open windows while you are working with the **MAX ANALYSIS MANAGER** software. The **WINDOW** menu is a standard Windows and Windows XP menu and works in the usual manner. (For more information about these items, see your Windows XP user manual.)

You can execute the following commands from the **WINDOW** menu (none of these items generates a dialog box).
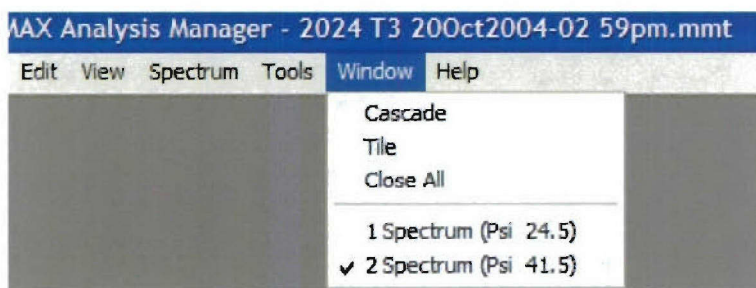
Figure 67 MAX Analysis Manager Window Menu

5.3.6.1 Window | Cascade

When you have more than one window open of any type, **WINDOW | CASCADE** resizes the windows to a uniform size and stacks them in a cascade from the upper left of your screen toward the bottom right. Each window overlaps a portion of the ones beneath it.

5.3.6.2 Window | Tile

When you have more than one window open of any type, WINDOW | TILE resizes the windows to a uniform size and tiles them (places them one beside the other) across your screen. No window overlaps another.

5.3.6.3 Window | Close All

When you select WINDOW | CLOSE ALL, MAX ANALYSIS MANAGER closes all open windows at once.

5.3.6.4 Window | [list of all open windows]

MAX ANALYSIS MANAGER displays the name of each open window in a list. You can navigate from one window to any other by selecting the title of that window.

5.3.7 Help Menu

5.3.7.1 Help | About MAX

This menu item brings up the following dialog box, which gives you information about the current version of the MAX ANALYSIS MANAGER software. To dismiss the dialog box, click the **OK** button.

Figure 68 MAX Analysis Manager Help Menu
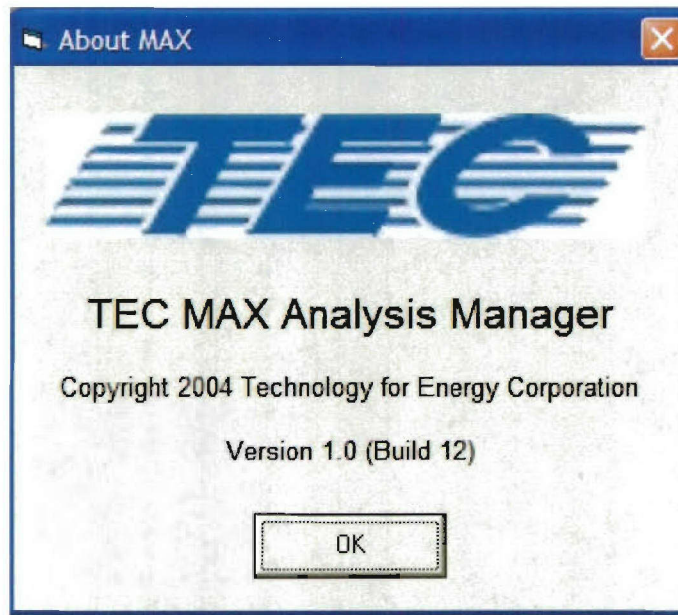
Figure 69 MAX Analysis Manager Help | About Window

6 System Maintenance

System maintenance consists of preventative and corrective maintenance actions. Corrective actions will be discussed in more detail in Section 7 of this manual. Preventative maintenance consists of the following periodic actions.

6.1 *Diffractometer Calibration*

The operator must perform a detector calibration:

1.   Whenever a detector is changed.

2.   Whenever a detector electronics board is replaced.

3.   Whenever measurements of the standards indicate a problem because of unacceptable results.

6.1.1 Diffractometer Calibration Setup

1.   Perform test procedure 4005-TP-01 to align and calibrate both Detector Interface Boards and Detectors.

2.   Set diffractometer on the standard for the type of head installed.

6.1.2 Efficiency Measurement

1.   Run MAXAcquisition.

2.   In the SETUP window, select the correct parameters.

3.   Select the efficiency measurement.

4.   Select Start and when the measurement completes store the efficiency data.

5.   Return to SETUP window and proceed with calibration measurement.

6.1.3 Calibration Measurement

1.   In the SETUP window, select the correct parameters.

2.   Select the calibration measurement.

3.   Select Start and when the measurement completes store the calibration data.

4.   Perform measurements on the test standards to validate the calibration.

6.2   Diffractometer PM

1   Clean all surfaces of the diffractometer assembly

2   Ensure that the collimator holder and collimators are clean and clear.

3   Check the detector filters for correct fit and lack of tears or holes in the filter material.

4   Inspect the connectors for signs of wear and improper fit.

5   Check to ensure all mounting screws are tight.

6.   Check High Voltage power supply for wear and tear.

6.3   Workstation PM

1   Check all connectors for signs of wear and correct fit

2.   Check for correct operation of all switches and lamps/LEDs.

3.   Clean case and front panel.

6.4   Cable Assembly PM

1   Check all connectors for signs of wear and correct fit.

2   Check all cables for signs of wear or fraying.

7 Troubleshooting

+problems that may occur with the MAX system. The intent is not to provide component level replacement options, other than major assemblies.. The circuit boards may be replaced and returned to factory for repair.
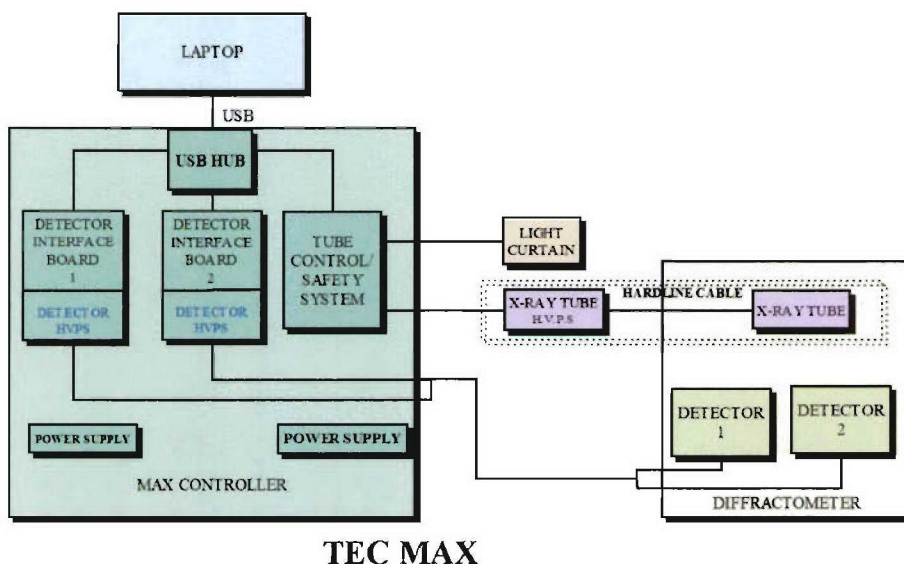
Figure 70 TEC MAX Block Diagram

7.1 Failure Symptoms

Table 2 Failure Symptoms

Failure Symptom	Probable Causes	Repair Actions
No power to Workstation	Power cord not connected or bad.	Check and replace
	Blown Main Fuse	Check and replace
	Failed power supply	Check and replace
No Spectra on one detector	Shielding by sample	
	Cabling	Check all

Failure Symptom	Probable Causes	Repair Actions
		cables
	Failed detector	Replace Detector
		Replace PCB
	Failed detector interface PCB	
No Spectra on both detectors	x-ray tube	Check all cables
		Replace tube
	Workstation power supply	Check voltages
Optical beam safety device does not stop system operation	Too much light in room	Adjust unit to operate
	Failed Safety System PCB	Replace Safety System PCB
Optical beam safety device does not allow measurement	Cable disconnected or loose	Check cable
	Red Target not aligned	Align target
	Failed optical unit	Replace optical unit
	Failed Safety System PCB	Replace Safety System PCB

7.2 Assembly Replacement Procedures

The following sub-sections provide guidelines for replacement of the MAX assemblies and sub-assemblies.

7.2.1 Diffractometer Assemblies Replacement

7.2.1.1 Diffractometer replacement

To replace the diffractometer assembly:

1.   Power the system down.

2.   Disconnect the five cables and the ground wire at the diffractometer.

3.   Connect the new diffractometer by connecting the five cables and the ground wire.

7.2.1.2 Detector Replacement

To replace a detector:

1.   Power the system down.

2.   Disconnect the data and high voltage connectors on the detector to replace.

Figure 71 MAX PSPC Detector

3.   Remove the hex head screw holding the detector bracket to the diffractometer,

4.   Remove the four Phillips screws hold the detector to the bracket.

5.   If the detector being replaced has a filter installed, remove it.

6.   Reverse the procedure to install the new detector.

7.   The system must be calibrated for the new detector (see Diffractometer Calibration 6-1)

7.2.1.3 X-ray tube and High Voltage Power supply Replacement

Figure 72 MAX High Voltage Power supply

To replace the x-ray tube and high voltage power supply:

1.   Power the system down.

2.   Disconnect the high voltage power supply connector.

3.   Disconnect the ground wire from the tube housing.

4.   Remove both detectors by removing the hex head screws holding the detector brackets to the diffractometer.

5.   Remove the four Phillips screws holding the tube in the tube holder and slide the tube out.

6.   Replace the new tube by following the above in reverse.

It is important that the new tube is aligned with the collimator exit hole.

7.2.1.4 Cable Assembly Replacement

To replace the cable assembly:

1.   Power the system down.

2.   Disconnect the five cables and the ground strap at the diffractometer.

3.   Disconnect the five cables and the ground strap at the MAX Workstation.

4.   Reverse to install the new cable assembly.

7.2.2 Workstation Assemblies Replacement

The following sub-sections require that the MAX Workstation be opened for access to the circuit boards and power supplies. To open the MAX Workstation:

1. Power the system down and remove the ac power cord from the workstation.

2. Disconnect all cables from the front panel.

3. Remove the eight Phillips screws holding the front panel in the case.

4. Using the handles, pull the chassis out of the case.

5. Reverse to install chassis in case.
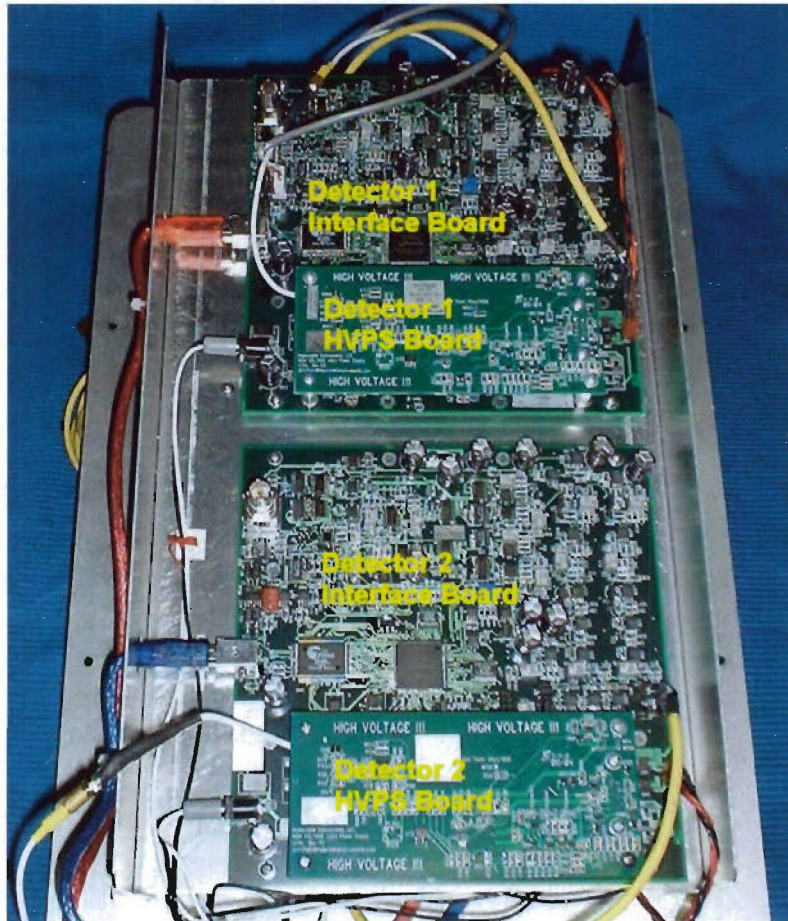
Figure 73 MAX Circuit Board Locations

7.2.2.1 Detector Interface Board (DIB) Replacement

1. Disconnect the six cables connected to the Detector Interface Board to be replaced.

2. Remove the four Phillips head screws holding the board to the chassis.

3. Remove the four Phillips head screws holding the Detector High Voltage Board (DHVB) on the DIB and remove the board.

4. Place the DHVB on the replacement DIB and replace the four screws.

5. Place the replacement DIB on the chassis and replace the four screws.

6. Reconnect the six cables to the DIB.

7. Perform test procedure 4005-tp-01 to calibrate and align the new DIB.

7.2.2.2 Detector High Voltage Board (DHVB)

1. Remove the high voltage connector from the DHVB to be replaced.

2. Remove the four Phillips head screws holding the (DHVB) on the DIB and remove the board.

3. Place the new board on the DIB and replace the four Phillips head screws.

4. Reconnect the high voltage connector.

7.2.2.3 Tube Control/Safety System PCB Replacement

1. Remove the five Phillips screws and ground lug that hold the chassis to the front panel.

2. Disconnect the cables from the board.

3. Remove the four screws holding the board to the standoffs and remove the board.

4. Reverse the procedure to install the replacement board.

7.2.2.4 Power Supply Replacement

1. Remove the five Phillips screws and ground lug that hold the chassis to the front panel.

2. Remove the wiring from the power supply to be replaced.
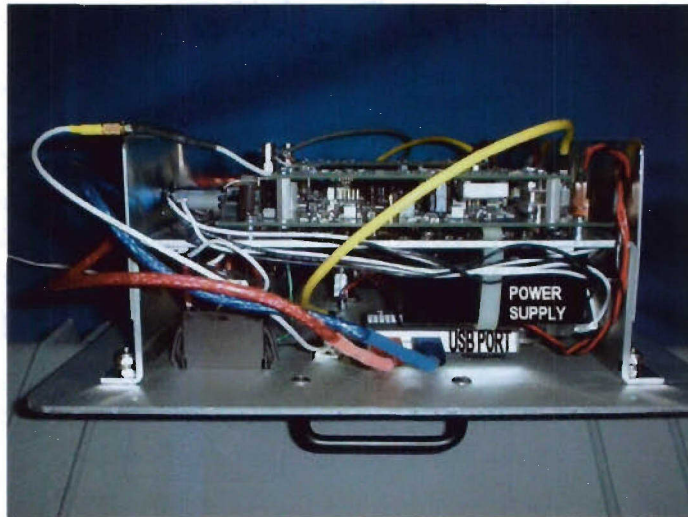
Figure 74 MAX Workstation Side View

3. Cut the tie wrap and remove and pry the power supply off the chassis.

4. Place a piece of double-sided tape on the replacement power supply.

5. Put the replacement in place and secure with a tie wrap.

6. Reconnect the wiring.

7.3 Technical Support

TEC will provide technical support for the MAX System. Problems which the operator cannot resolve should be referred to TEC. The system has a return to factory warranty.

<div align="center">

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8 List Of Tables And Figures

8.1 List of Tables

8.2 List of Figures