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MOSSBAUER EFFECT
USING BACKSCATTER GEOMETRY
AS APPLIED TO NONDESTRUCTIVE TESTING

December 1969

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AEC RESEARCH &
DEVELOPMENT REPORT

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MOSSBAUER EFFECT USING BACKSCATTER GEOMETRY
AS APPLIED TO NONDESTRUCTIVE TESTING

By

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ABSTRACT

A Mossbauer effect spectrometer using a backscatter geometry has been developed which is capable of over 165% peak resonant signal to nonresonant background. This high percentage effect reduces the radiation counting time to minutes as compared to hours using conventional methods and leads the way to valuable nondestructive techniques. This improvement was achieved primarily by special shielding procedures and increased source intensity matched to an appropriate gas-filled proportional counter detector. Cobalt-57 was used as the source material, however, the technique can be applied to other Mossbauer elements.

A number of spectra are shown for different applications in such fields as surface physics, geology and metallurgy.

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MOSSBAUER EFFECT USING BACKSCATTER GEOMETRY
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INTRODUCTION

This report describes a Mossbauer spectroscopy system⁽¹⁾ which has various applications for nondestructive testing. Discussions are presented of the principle of the Mossbauer effect, the test equipment needed for the backscatter technique, and the interpretation of the spectra. To suggest the scope of applications for this technique, spectra are shown in different applications in such fields as surface physics, metallurgy, geology and biology.

SUMMARY AND CONCLUSIONS

The large increase in the percent resonance effect using the Mossbauer backscatter geometry described in this report opens the way to many important nondestructive applications. These applications cover a large number of disciplines including metallurgy, surface physics, mineralogy, and biology. The principal advantage of the technique is the ability to analyze the sample "as is"; i.e., the sample can be analyzed without being ground into particles or removed from place. The large percent resonance effect decreases the counting time from hours to minutes and allows the use of automatic testing equipment for the control and analysis of complex mixtures including metal containing ore.

MOSSBAUER EFFECT

The resonant absorption of γ -rays and the recoil-free emission by certain nuclei was discovered in 1957 by Rudolph L. Mossbauer during his graduate work in Heidelberg, Germany. In Mossbauer spectroscopy, a radioactive source consisting of excited nuclei, decays to the ground state and in doing so emits highly monochromatic γ -radiation. The emitted γ -rays are resonantly absorbed in nuclei within a sample where the nuclei are characteristic of the ground state nuclei of the radioactive source. The probability of a recoilless event (emission or absorption) is given by the Debye-Waller factor. It will be shown in this report that the resonance line is so narrow that its energy can be shifted significantly by means of the Doppler effect, that is, by moving the source (or absorber) at velocities of the order of a few mm/sec. Doppler shifting the γ -ray photon is analogous to the use of a dispersive device in optical spectroscopy.

The conventional technique used in Mossbauer spectroscopy is to locate a thin sample between the radioactive source and an appropriate detector. In this report we describe a backscatter technique which has many advantages over the through-transmission approach. The backscatter geometry is shown in Figure 1 where the sample to be analyzed is placed at an appropriate angle with respect to the source and detector. The primary advantage of this technique is the ability to use "as is" samples with no preparation; as an example, to study the corrosion products of a mild steel specimen without any disturbance. This not only saves time but allows us to measure the depth of the products by selecting different photon energies. Also, long sampling times become shorter with the ability to use much "hotter" sources. This assumes that proper collimation and shielding procedures are used. Although ^{57}Co was used as the source material in our work, many other Mossbauer atoms are available.

MOSSBAUER EFFECT THEORY

To understand the phenomenon of the Mossbauer effect and, therefore, its use as a spectroscopic tool, consider a free atom of mass, M . The recoil energy of the atom due to the emission of a photon of energy

E_0 is $E_0^2/2Mc^2$, where c is the velocity of light. If the atom is in motion during the emission of a photon, the photon energy is modified by a term $E_0|\vec{v}/c|\cos\theta$, where $\vec{v}$ is the velocity of the atom and θ is the angle between $\vec{v}$ and the momentum vector of the photon. The energy of the photon emitted by the nucleus is

$$E_\gamma = E_0 - E_0^2/2Mc^2 + E_0|\vec{v}/c|\cos\theta, \quad (1)$$

where E_0 is the photon energy in the rest frame of the nucleus. The photon energy for resonant absorption by a similar nucleus moving with velocity $\vec{v}'$ and direction θ' is

$$E'_\gamma = E_0 + E_0^2/2Mc^2 + E_0|\vec{v}'/c|\cos\theta'. \quad (2)$$

The energy for γ -rays emitted by a group of free atoms moving with thermal velocities would be centered at

$$E_\gamma = E_0 - E_0^2/2Mc^2 \quad (3)$$

and the resonant absorption energy would be centered at

$$E'_\gamma = E_0 + E_0^2/2Mc^2. \quad (4)$$

Resonance would occur for the fraction of events represented by the overlap in energy of the emission and absorption lines which is of the order of 0.01 eV at room temperature. Mossbauer discovered that in a solid, $E_\gamma = E_0$, the recoil energy $E_0^2/2Mc^2$ becomes vanishing small because M becomes the mass of the crystal and the $E_0|\vec{v}/c|\cos\theta$ terms can be described by a quantity which relates the number of photons that have been interchanged between the lattice and the γ -ray photons. There is a nonvanishing probability that no photons will be interchanged. This is given by the Debye-Waller factor

$$f = \exp \left\{ -3/2 \frac{E_0^2}{2Mc^2} \cdot \frac{1}{K\theta} \left[1 + 2/3 \left(\frac{\pi T}{\theta} \right)^2 \right] \right\} \quad (5)$$

where harmonic forces are assumed in the solid and θ is the Debye temperature, M is the atomic mass, K is Boltzmann's constant and E_0 is the γ -ray energy; f for ^{57}Fe in metallic iron is 0.7 at room temperature.

When the above conditions prevail, the emission (or absorption) is recoil-free and the emitted (or absorbed) γ -rays match the energy levels and widths of the nuclear transition. It is this condition that characterizes the Mossbauer effect.

INSTRUMENTATION FOR BACKSCATTER MODE

The Mossbauer test configuration for the backscatter mode is shown in Figure 1. The components include:

- Source drive unit
- Source
- Collimator and shielding
- Specimen
- Detector
- Amplifying and associated electronics
- Multichannel analyzer
- Data processing equipment.

A detailed description of the above components follows.

SOURCE DRIVE UNIT

The drive unit shifts the energy of the γ -photons emitted by the source (^{57}Co was used for this study) by a precisely controlled amount. The velocity of the photon is related to this energy shift by $v = c\Delta E/E$ where c = velocity of light. The velocities required for ^{57}Co are approximately ± 1 cm/sec. Drive units are commercially available with an analog signal linearity of $\pm 0.5\%$ (this ignores the reset transient shown in Figure 2) and a reproducibility of the analog signal to $\pm 0.25\%$. The most practical system operates in the constant acceleration mode where all the energy can be scanned at once. The motion of the source produced by the drive unit is shown in Figure 2 where the position of the source as a function of time is parabolic. The position of the source is closest to the sample at zero velocity and, therefore, has an increased number of counts. This results in a parabolic shaped background count for the

FIGURE 1. BACKSCATTER MOSSBAUER EFFECT CONFIGURATION

CONSTANT ACCELERATION MODE:

MOTION OF EMITTER

FIGURE 2. MOSSBAUER SOURCE MOTION

Lorentzian resonant peaks. The curvature of the parabola can be changed by reversing the direction of the drive unit. New drive units are commercially available which minimize the parabola effect by shortening the stroke length. Although the parabola can be subtracted out by computer least squares curve fit routines, the shorter stroke length aids in the design of the collimation window and keeps the sample exposure area more nearly the same size; i.e., reduces line broadening. The design of electro-mechanical source drive units are given by Wertheim et al., and Spijkerman et al.⁽²⁾.

SOURCE

Approximately half of the elements in the periodic table are possible candidates for Mossbauer spectroscopy (see Figure 3). Over half the

		H	He																
Li	Be											B	C	N	O	F	Ne		
Na	Mg											Al	Si	P	S	Cl	Ar		
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr		
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe		
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn		
Fr	Ra	Ac																	
			Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu			
			Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr			

MOSSBAUER EFFECT OBSERVED (32 ELEMENTS)

MOSSBAUER EFFECT PREDICTED (17 ELEMENTS)

FIGURE 3. MOSSBAUER ELEMENTS

articles that have been published used ^{57}Fe . The reason for this is the large natural abundance of this element in many compounds. Also, the line width is narrow enough so that cryogenic temperatures are unnecessary thus avoiding complicated measurement procedures. The radioactive parent nuclei ^{57}Co decay by electron capture to ^{57}Fe 99.8% of the time. This is accomplished by an electron and a proton combining to produce a neutron and a γ -ray; i.e.,

The decay relationships are shown in Figure 4. The electron vacancy is filled by outer electrons which thereby produce the internal conversion: 6.5 keV X-rays.

FIGURE 4. THE DECAY OF ^{57}Co TO ^{57}Fe

By using the backscatter geometry, we are able to shield the source from the detector and thereby increase the intensity of the source. Sources up to 100 mCi are presently available from manufacturers; however, the cost becomes prohibitive with increasing intensity. The parent nuclei of the source are usually diffused onto a nonmagnetic matrix such as copper, stainless steel or palladium. Our measurements were made with a Nuclear Science 30 mCi ^{57}Co source on a palladium matrix, 0.001 in. thick and 0.5 in. in diameter.

COLLIMATOR AND SHIELDING

Because of the high intensity of the source used in the backscatter method, it is necessary to shield the source very carefully to reduce the nonresonant background scatter. Figure 1 shows the backscatter geometry in which the source is shielded from the detector. The size and shape of the exposed area on the sample can be controlled by the appropriate size and shape of the collimating window. Because the sample is set at an angle with respect to the source and the detector, the frustum of a cone in the collimating window produces an ellipse on the exposed area of the sample. The exposed area on the sample can be converted to a circle by designing the collimating window with an ellipsoidal cone where the two ellipses on each face of the collimator are offset with respect to each other.

Figures 5 and 6 indicate the effect of the size of the sample area upon the nonresonant count $[I(\infty)]$ and resonance effect magnitude $\left[\frac{I(0) - I(\infty)}{I(\infty)} \right]$ for both the 6.3 keV X-rays and 14.4 keV photons. In this case the collimator produces an ellipse on the sample. The nonresonant count without the sample in the field of view is approximately 380 counts per hour per channel for the 6.3 keV X-rays and 680 counts per hour per channel for the 14.4 keV γ -rays. (The 14.4 keV count is higher because a piece of plexiglass is placed over the collimating window when counting 6.3 keV X-rays.) The area of an ellipse from zero to 1.38 in.² (where one of the foci is placed on the centerline from the source) is the region where the entire sample area is exposed by the source; i.e., is not eclipsed by the collimating window. The penumbra region is the area between the two ellipses indicated by points a, d and b, c in Figure 1. This is the proportional area shown in Figures 5 and 6 between 1.38 and 6.46 in.². In Figures 5 and 6 only the ambient background and points A and B were measured.

We found that lead is not a good shielding material because of X-ray fluorescence. Depleted uranium is satisfactory when additional graded shielding of copper and plexiglass is provided.

FIGURE 5. PERCENT RESONANT EFFECT AS A FUNCTION OF SCATTER AREA

FIGURE 6. NONRESONANT EFFECT AS A FUNCTION OF SCATTER AREA

In conclusion, the maximum size of the collimator is a compromise between the resonance effect (increase with increasing window size) and the Doppler velocity cosine effect on the line broadening. The theoretically available line width for the mean life of the 14.4 keV transition using the uncertainty principle is

$$\Gamma = \frac{\hbar}{\tau} = \frac{6.58 \times 10^{-16} \text{ eV sec}}{1.4 \times 10^{-7} \text{ sec}} = 4.7 \times 10^{-9} \text{ eV} \quad (6)$$

where $\hbar$ = Plank's constant over 2π .

The sharpness of the resonance of the 14.4 keV γ -ray can, therefore, be

$$Q = \frac{E_{14.4 \text{ keV}}}{\Gamma} = \frac{14.4 \times 10^3 \text{ eV}}{4.7 \times 10^{-9} \text{ eV}} = 3.26 \times 10^{13} \quad (7)$$

The velocity for one line width is

$$\begin{aligned} v &= \left(\frac{\Gamma}{E} \right) c = \frac{3 \times 10^{10}}{3.26 \times 10^{13}} \text{ cm/sec} \\ &= 0.920 \times 10^{-3} \text{ cm/sec} \approx 0.1 \text{ mm/sec} \end{aligned} \quad (8)$$

so that 1 mm/sec equals about 10 line widths. The line width will increase slightly because of the decrease in Doppler velocity due to the $v \cos \theta$ effect where θ is the collimator cone angle. For an angle of 12° , $v \cos \theta = (0.920 \times 10^{-3} \text{ cm/sec}) (0.97815) = 0.8999 \times 10^{-3} \text{ cm/sec}$ or about 2% of the line width.

SPECIMEN

The specimen used for the backscatter geometry can be a steel plate, a rock with a flat face, a frozen liquid, ground-up powder placed between two pieces of transparent tape, etc. The thickness of the specimen is not restricted. Studies were performed to determine the depth of penetration of 14.4 keV γ -rays. The depth of penetration is shown in Figure 7 where an 0.4 mil thick magnetic iron foil (6 resonance peaks) was placed in front of a nonmagnetic stainless steel foil (1 peak). The single stainless steel peak can be seen between the six Zeeman peaks in the iron. The depth of penetration increases with increasing γ -ray energy, and for the 14.4 keV γ -ray is about 0.5 mil.

FIGURE 7. SAMPLE THICKNESS EFFECT

The size of the exposure area on the sample due to the γ -rays coming from the source is controlled by the collimating window. The optimum size of this area is a compromise between two considerations: 1) the incoming photons that have a velocity component off the source to sample axis are reduced in energy by $v \cos \theta$, where θ is the off axis angle. If the angle becomes too large, the result is to cause a broadening in the resonant line widths. 2) the percent resonant effect, $\frac{I(0) - I(\infty)}{I(\infty)}$, where $I(0)$ is the resonant peak height and $I(\infty)$ is the nonresonant background (as will be shown later) is increased by increasing the exposure area.

The orientation of the sample with respect to the source and detector is not critical for reasonable size exposure areas; however, the resonant signal can be maximized by optimizing these angles during the counting process.

The sampling time is a function of the number of Mossbauer nuclei and, therefore, a maximum packing density is desirable. This is most easily achieved in solids, however, samples ground into powder and enclosed in transparent tape may also be used. Frozen liquids may also be used by mounting them on the surface of a cooling reservoir containing an appropriate coolant. As an example of resolution, using the backscatter technique, we have been able to measure a 23% resonant effect with powdered basalt containing about 0.2% ^{57}Fe .

DETECTOR

The detector should be chosen to match the energy characteristics of detected photon. We have chosen a Reutor-Stokes (RSG-61M2) proportional counter with a krypton fill gas and carbon dioxide quench. Other gas components are available, however. As shown in Figure 8 and Table 1, krypton is the most efficient for the 14.4 keV γ -rays.

TABLE 1. ENERGY CHARACTERISTICS FOR DETECTOR GAS FILL COMPONENTS

Gas	Absorption Edge, keV	Upper and Lower Edge $\mu(\frac{\text{cm}^2}{\text{g}})$	Mass Absorption $\mu(\frac{\text{cm}^2}{\text{g}})$ at		Compton Scattering
			6.5 keV	14.41 keV	
Argon	3.2	$\mu_1 = 140$ $\mu_2 = 1500$	205	23	low
Krypton	14.32	$\mu_1 = 19.6$ $\mu_2 = 150$	175	149	medium
Xenon	34.58	$\mu_1 = 6.2$ $\mu_2 = 34.5$	560	70	high

FIGURE 8. MASS ABSORPTION AS A FUNCTION OF ENERGY
FOR ARGON, KRYPTON AND XENON

AMPLIFYING AND PULSE SORTING ELECTRONICS

The object of this equipment is to amplify and determine the counting rate of the photons received by the detector as a function of the photon energy. The detection system is designed to measure the counting rate as a function of the relative velocity between the source and absorber. If the transition energies of ^{57}Fe nuclei in the sample lie within the energy range of the Doppler shifted photons emitted by the source, a resonant spectrum will be observed. Refer to Figure A-1 (Appendix A) for a block diagram of the amplifying and pulse sorting electronics. A multichannel pulse height analyzer (PHA) operating as a multiscaler is used to sort out the detector pulses as a function of the velocity of the source. This is accomplished by sweeping the PHA memory synchronously with the velocity sweep while feeding the discriminated pulses into the analyzer. Note also that a single channel analyzer is used to select only those pulses with the appropriate energy (we used the 14.4 keV γ -rays in our studies). This is accomplished as explained in detail in Section VI of Appendix A by adjusting the energy window of the single channel analyzer. Refer to Appendix A for detailed operating procedures for the pulse sorting equipment.

HYPERFINE STRUCTURE

ELECTRIC MONOPOLE INTERACTION

The quantity δ in Figure 9 is called the isomer shift and is caused by the electric monopole interaction between the nucleus and its surrounding electrons. The quantity δ also includes the second order Doppler shift caused by thermal motion of the atoms; however, this effect can be minimized by holding the source and absorber at the same temperature. In iron metal the second order Doppler shift is less than the natural line width between 300 and 4 °K.

FIGURE 9. MOSSBAUER SPECTRUM FOR NONMAGNETIC STAINLESS STEEL

The isomer shift can be derived by considering the difference in energy between a nucleus with uniform charge density and a point nucleus used as a reference. The potential within a nucleus of uniform charge distribution where the charge density ρ is found by calculating

$$v(r) = v(R) - v(R-r) \quad (9)$$

where

R = radius of nucleus

r = distance from center of nucleus to some point inside nucleus

$$V(R) = - \int_R^{\infty} E dr = - \left. \frac{Ze}{r} \right|_R^{\infty} = \frac{Ze}{R} \quad \text{esu} \quad (10)$$

and

$$V(R-r) = \int_R^r E dr \quad (11)$$

The charge density is

$$Ze = \int_V \rho d^3x = \frac{4}{3} \pi R^3 \rho \quad (12)$$

Using Gauss' Law

$$\oint_S \vec{E} \cdot d\vec{a} = \int_V 4\pi \rho d^3x \quad (13)$$

gives

$$E 4\pi r^2 = 4\pi \cdot \frac{4}{3} \pi r^3 \frac{Ze}{4/3 \pi R^3} = \frac{rZe}{R^3} \quad (14)$$

Therefore,

$$V(R-r) = \int_R^r \frac{rZe}{R^3} dr = \frac{Ze}{2R^3} (r^2 - R^2) \quad (15)$$

and

$$V(r) = V(R) - V(R-r) = \frac{Ze}{R} \left(3/2 - \frac{r^2}{2R^2} \right) \quad (16)$$

The potential for a point source is

$$V_{pt} = \frac{Ze}{r} \quad (17)$$

The energy difference between the finite and point nucleus is

$$\begin{aligned}
\delta E &= \int_0^R \rho(V - V_{pt}) 4\pi r^2 dr \\
&= \frac{4\pi\rho Ze}{R} \int_0^R \left(\frac{3}{2} - \frac{r^2}{2R^2} - \frac{R}{r} \right) r^2 dr \\
&= -\frac{2\pi}{5} \rho Ze R^2 = \frac{2\pi}{5} Ze^2 |\psi(0)|^2 R^2, \quad (18)
\end{aligned}$$

where

$$\rho = -e|\psi(0)|^2. \quad (19)$$

Therefore, the true ground state energy of a spherical nucleus with radius R_g and uniform charge distribution is

$$\delta E_g = \frac{2\pi}{5} Ze^2 |\psi(0)|^2 R_g^2. \quad (20)$$

Similarly, in the excited state:

$$\delta E_{ex} = \frac{2\pi}{5} Ze^2 |\psi(0)|^2 R_{ex}^2, \quad (21)$$

and for the source nucleus,

$$\delta E_{ex} - \delta E_g = \frac{2\pi}{5} Ze^2 (R_{ex}^2 - R_g^2) |\psi(0)|_S^2. \quad (22)$$

Also, for the absorber nucleus

$$E_{ex} - \delta E_g = \frac{2\pi}{5} Ze^2 (R_{ex}^2 - R_g^2) |\psi(0)|_A^2. \quad (23)$$

Subtracting Eq. (22) from Eq. (23) gives the isomer shift

$$\begin{aligned}
\delta &= \frac{2\pi}{5} Ze^2 [R_{ex}^2 - R_g^2] [|\psi(0)|_A^2 - |\psi(0)|_S^2] \\
&= \frac{4\pi}{5} Ze^2 R^2 \left(\frac{\delta R}{R} \right) [|\psi(0)|_A^2 - |\psi(0)|_S^2], \quad (24)
\end{aligned}$$

where

$$2R\delta R = R_{ex}^2 - R_g^2, \quad (25)$$

$$\delta R = R_{ex} - R_g, \quad (26)$$

$$R \cong \frac{R_{\text{ex}} + R_g}{2} \quad (27)$$

For ^{57}Fe , $R = 4.61 \times 10^{-13} \text{ cm}$

$$\frac{\delta R}{R} = 1.8 \times 10^{-3} \quad (28)$$

When $\frac{\delta R}{R} > 0$, $\delta > 0$ corresponds to an increase in the S-electron density at the nucleus.

The total electron density at the nucleus between the source and absorber is

$$[|\psi(o)|_A^2 - |\psi(o)|_S^2]$$

The charge density is due solely to electrons in S-orbitals. The wave functions can be obtained from the Hartree-Fock calculations for different configurations.

ELECTRIC QUADRUPOLE COUPLING

The isomer shift was derived by assuming the nucleus to be spherical and the charge density to be uniform. If these terms are relaxed, other effects appear which are the higher terms in the multipole expansion of the electrostatic interaction. These terms split the nuclear levels and for the case of the quadrupole splitting, two peaks are obtained as shown in Figure 10 for sodium nitroprusside. ΔE is called the quadrupole splitting. The doublet is characteristic of the interaction between quadrupole moment of the excited state of the nucleus

$$Q_{ij} = \int_V (\vec{X}) X_i X_j d^3x = \int_V \rho(\vec{r}) (3X_i X_j - \delta_{ij} r^2) d^3r \quad (29)$$

and the gradient of the electric field produced at the nucleus by the surrounding electrons

$$\nabla E = - \frac{\partial^2 V}{\partial X_i \partial X_j} = - V_{ij} \quad (30)$$

FIGURE 10. MOSSBAUER SPECTRUM FOR $\text{Na}_2\text{Fe}(\text{CN})_5 \cdot \text{NO} \cdot 3\text{H}_2\text{O}$

The interaction between the nucleus with a quadrupole moment eQ and an electric field gradient at the nucleus is given by the Hamiltonian operator⁽³⁾

$$H_Q = \frac{eQ V_{zz}}{4I(2I-1)} \left[3I_z^2 - I(I+1) + \frac{\eta}{2} (\hat{I}_+^2 + \hat{I}_-^2) \right], \quad (31)$$

where

$$\eta = (V_{xx} - V_{yy})/V_{zz} \quad (32)$$

is the asymmetry parameter and $\hat{I}_+^2$ and $\hat{I}_-^2$ are the shift operators. The interaction energy for asymmetrical electric field gradient is

$$E_Q = \frac{eQ V_{zz}}{4I(2I-1)} [3M_I^2 - I(I-1)] \quad , \quad (33)$$

where

$$M_I = I, I-1, \dots, -I \quad .$$

For the ground state in ^{57}Fe , where $I = 1/2$, $M_I = \pm 1/2$

$$E_Q = 0 \quad .$$

For the 14.4 keV first excited state, where $I = 3/2$, $M_I = \pm 3/2, \pm 1/2$

$$E_Q = \frac{e^2 q Q}{4} \quad , \quad \frac{-e^2 q Q}{4} \quad . \quad (34)$$

The quadrupole splitting of the first excited state is

$$2|E_Q| = \Delta E = 1/2 e^2 q Q \quad . \quad (35)$$

The two transitions possible lead to the doublet absorption spectrum shown in Figure 10. For an asymmetric electric field gradient

$$\Delta E = \frac{e^2 q Q}{2} \left(1 + \frac{\eta^2}{3}\right)^{1/2} \quad . \quad (36)$$

The quadrupole moment Q of the 14.4 keV state has been estimated to be $0.29 \times 10^{-24} \text{ cm}^2$, to within a factor of about 2.

The charges surrounding the nucleus are responsible for the field gradient and, therefore, the quadrupole splitting is useful in obtaining information about the local electronic environment of the nucleus.

MAGNETIC DIPOLE INTERACTION

Interactions between the magnetic moment of the nucleus and the magnetic field produced by the electrons at the site of the nucleus produce the Zeeman six peak spectrum. The Hamiltonian is given by

$$\mathcal{H}_M = -\vec{\mu}_N \cdot \vec{H} = -\gamma \hbar \vec{I} \cdot \vec{H} \quad (37)$$

where

$\vec{\mu}_N$ = Nuclear magnetic moment

$\vec{I}$ = Spin

γ = Gyromagnetic ratio

$$\vec{H} = -2\beta \sum_i \left[\frac{\vec{L}_i}{r^3} - \frac{\vec{S}_i}{r^3} + 3\vec{r} \frac{(\vec{S}_i \cdot \vec{r})}{r^5} + \frac{8\pi}{3} S_i \delta(\vec{r}) \right]$$

The energy levels are

$$E_m = -\mu_H M_I / I = -g \mu_N H M_I \quad (38)$$

The gamma-ray transitions are observed between two nuclear levels and obey the selection rule

$$\Delta M = 0, \pm 1$$

Reference to Figure 11 shows the transitions and, therefore, indicates

FIGURE 11. MAGNETIC HYPERFINE SPLITTING OF THE GROUND AND FIRST EXCITED STATE OF ^{57}Fe

that the ground $M_I = -1/2$ to the excited state $M_I = +3/2$ and the ground $M_I = +1/2$ to excited state $M_I = -3/2$ are forbidden transitions. The six magnetic hyperfine splitting peaks are shown for a mild steel plate in Figure 12.

FIGURE 12. MOSSBAUER SPECTRUM FOR MAGNETIC HYPERFINE SPLITTING

The even spacing of the lines is changed if quadrupole interactions are also present. The $\pm 3/2$ levels are moved in one direction and the $\pm 1/2$ levels in the other direction; therefore, the sign of the quadrupole coupling can be determined. In this way we are able to detect magnetic

exchange interactions including the sign and magnitude of the magnetic hyperfine field within the material. Changing the temperature of the sample also allows for a precision determination ($\pm 1^\circ$) of the Curie point or Néel point.

APPLICATIONS FOR NONDESTRUCTIVE TESTING

METALLURGY

It is possible to identify and classify various phases of metals and alloys and quantitatively identify their specific properties. M. Ron et al.,⁽⁴⁾ have studied the structure of martensite and found that in the vicinity of the carbon atoms the chemical environment has changed, and thus, have found how much of the carbon has been trapped martensitically in a sample containing carbon iron.⁽⁴⁾ The ratio of the peak intensities in the six-line spectrum of ferromagnetic iron gives the value of the internal magnetic field. These ratios may also be used to detect elastic strain in the material produced by an external stress field. Variation of the temperature of the sample allows for the calculation of the Néel and Curie temperatures. Other applications include the determination of near neighbor configuration, order alloys, precipitation processes, lattice defects and stress corrosion products. Figure 13 is an example of stress corrosion products on a mild steel plate.

MINERALS, TERRESTRIAL AND EXTRATERRESTRIAL

It is possible to identify the iron components of minerals including the identification of Columbia Basin basalts. A number of studies have been made to analyze meteorites and presently lunar materials are being analyzed using the Mossbauer effect. The ore of (FeTiO_3) ilmenite has two peaks, whereas (Fe_3O_4) magnetite has six peaks and, therefore, these can be easily classified in a mining process.

Figures 14 and 15 are examples of Columbia Basin basalt and pyrrhotite spectra using our Mossbauer spectrometer.

FIGURE 13. MOSSBAUER SPECTRUM FOR HEAVY RUST ON MILD STEEL PLATE

FIGURE 14. MOSSBAUER SPECTRUM FOR COLUMBIA BASIN BASALT - G12

BIOLOGICAL

Metallobiochemicals are good candidates for the backscatter technique because iron enrichment may not be necessary. Iron binding has been studied in hemoglobin, myoglobin and cytochrome C which are heme protein molecules. The iron plays a central role in the function of the heme proteins. Gonser, Grant, and Kregzde have found that the red cell spectrum at 4 °K has four lines and that two of the lines disappeared when the cells were treated with N₂, deoxygenating the hemoglobin. These lines are also found in CO₂ treated red blood cells suggesting that the CO₂ is not bound as a ligand to the iron but is bound to some other part of the molecule.

FIGURE 15. MOSSBAUER SPECTRUM FOR PYRRHOTITE MINERAL

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APPENDIX A

MOSSBAUER SPECTROMETER OPERATING PROCEDURES

APPENDIX A

MOSSBAUER SPECTROMETER OPERATING PROCEDURES

This appendix contains a checklist of useful items for those concerned with the operation of the Mossbauer spectrometer. The operating procedure is written for use with a Nuclear Data 2200 Multichannel Analyzer and a Nuclear Science NS-1 Mossbauer spectrometer; however, the procedures apply to other manufactures with slight modifications.

I. Pulse Height Analyzer Mode (used to check efficiency of shielding)

A. A.D.C.

1. Conversion gain - 256
2. Lower level disc - 0.95
3. Upper level disc - 10.0
4. Linear gate - closed
5. Timer - as-desired clock or live
 - a. On master control module the timer can be set for live time (clock time plus dead time), or clock time. When placed in PHA Sec, int, present time is 1, 2, 4, 8 sec times 10, 100, 1K, 10K, or 100K. If no preset time is desired, place switch on infinity.
6. With the Tennelec 202 BLR Amplifier, the +10 delayed input is used. (A.D.C. switch to the right, disc switch to the left.)

B. Master Control

1. Acquisition program - PHA
2. Memory groups 1, 2, 3, or 4 as desired (only quarter memory required, more could be used)
3. "Group Select Compare" out of circuit when overlap switch is off
4. Live display
5. Add-off-subtract on add
6. Timer - PHA sec 10 sec to infinity

II. Multi-Channel Scaling Mode (ADC not used)

A. Master Control

1. Acquisition program
 - a. M.S. - scales every channel once
 - b. M.S. Recur. - scales every channel until stopped by mode switch, or by switching to M.S.
2. Memory groups
 - a. Address reset is wired to use 512 channels (1 and 2 or 3 and 4)
3. Live display or statis (as desired)
4. Timer
 - a. M.S. μ sec, internal, selects dwell times from 20 msec to 0.8 sec or infinity. Dwell time is the time each channel is open to count pulses.
5. With proper input to clock BNC, different dwell times can be obtained in M.S. msec, Ext. position
6. Start BNC is shorted to ground.
7. Count-input from SCA - minimum +3V, maximum +6V

III. Calibrating Oscilloscope

- A. Place master control mode switch to stop
- B. On readin/out display module switch readin/out switch to plotter
- C. Digital readout switch to ALL
- D. Memory display to 1 mega- and display gain to X1
- E. When plotter calibrate switch is at off, dot on scope should be in lower left hand corner.
- F. When switched to X, move dot to the right with horizontal gain. If using half memory (1 and 2 or 3 and 4) move dot to full scale. If using full memory (1, 2, 3, and 4) move dot half of full scale.
- G. In the Y position adjust vertical gain for full scale deflection.

IV. Teletype Readout

- A. Place teletype switch to "line"
- B. Push tape punch "on"
- C. Teletype Drive module
 - a. Place switch in readout position
 - b. If punching a tape switch Address I.D. off and Leader on

D. Switch read-in/out switch to "printer"

E. Switch mode control to "read-in/out"

All channels selected by memory groups switch will be read out

V. Teletype Read-in

A. Teletype on "line"

B. Teletype drive module switched to "read-in" and address I.D. to "off"

C. Read-in/out control on "Printer"

D. Reader control to "Start"

NOTE: Tape used to supply data for read-in must have data only.
Any other numbers will prevent proper read-in.

VI. Adjusting Energy Window of S.C.A.

A. Obtain a pulse height spectrum of the 6.3 keV and 14.4 keV peaks

B. Connect the attenuated output of the reference pulser to the preamplifier test input located on the Tennelec TC 202 BLR amplifier

C. With the detector disabled (high voltage off) vary the pulse height until it falls into the channel desired. In this manner a pulse height dial reading can be obtained for both edges of the desired peak

D. With the pulse height set for the lower edge of the peak, apply this pulse to input of S.C.A., TC 440, using ΔE mode. Connect the S.C.A. output to a count rate meter and adjust S.C.A. controls to select the desired amount of the energy peak. Repeat for the upper edge.

VII. Since a photocell in the transducer resets the sweep generator that drives the transducer and the addresser, the stroke length can be varied directly by the symmetry control. Adjust symmetry control until the transducer drifts toward the photocell end of the transducer holder. The symmetry will determine how far the transducer can travel after the resetting takes place.

FIGURE A-1. ELECTRONIC CONFIGURATION

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