Chapter III

THE DEVELOPMENT OF DOPPLER-BROADENED POSITRON ANNIHILATION (DBPA) TECHNIQUE

3.1 Introduction

The object of this thesis is to develop the DBPA spectroscopy as a non-destructive testing (NDT) technique for evaluating dislocation density in stainless steels. The DBPA measurement is selected for this thesis because it is the simplest method and the apparatus can be obtained from this department. The DBPA spectroscopy which can be detected by a semi-conductor is quantifiably described using a lineshape parameter, S. The usual lineshape S parameter is used to evaluate the shape profile of DBPA spectrum reflecting the state of microstructure inherent in materials. However, S parameter is affected by several variables causing instability of the lineshape affecting the accuracy of S parameter. Thus, the development of DBPA technique is specially focused on developing electronics unit to stabilize annihilation lineshape. Furthermore, no criterion for $\Delta\varepsilon$ has ever been set for evaluating the S value making it impossible for experimental results to be directly benchmarked and compared. The compensating technique is thus proposed to correct and analyze the S values of annihilation spectra with high degree of credibility.

3.2 The DBPA measurement system

A basic system for measuring the DBPA spectroscopy consists of a positron source sandwiched between two investigated specimens, a semi-conductor detector, a gamma ray detection system and a computer unit to process and collect information. A 100 μCi Na^{22} positron source was used for this measurement because of its long half life and emission of 1274 keV gamma ray after positron decay which can be used as a reference spectrum for monitoring the changes of energy resolution during the experiments. The positron source is sandwiched between two flat specimens using the specially designed source and samples holder. It is also important to design this holder such that the samples can be easily inserted and removed without affecting the geometry of the measurement system. The positron source and sample holder was designed to be as shown in Figure 3.1. It was constructed out of three plastic plates with a dimension of $80 \times 90 \times 1.0$ mm. To house the positron source, one of the plates was first drilled to have a hole with a diameter of 20 mm which is equal to the diameter of positron source. The other two plastic plates were drilled to have an opening of 9.5 mm. The positron source is then sandwiched between these two plates acting as an active window for the positron source. The investigated specimens with a dimension larger than the active window are then positioned against the active window when testing. Figure 3.2 shows the positron source and samples holder together with the Na^{22} positron source and two investigated specimens sandwiching the source.

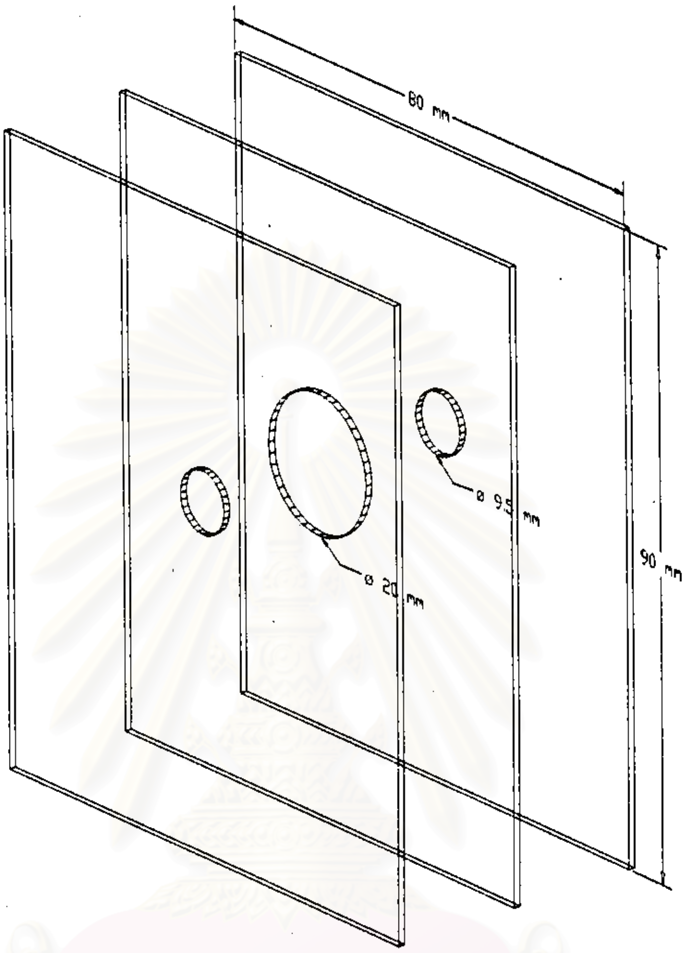

Figure 3.1 The arrangement of three plastic plates as a positron source and sample holder.

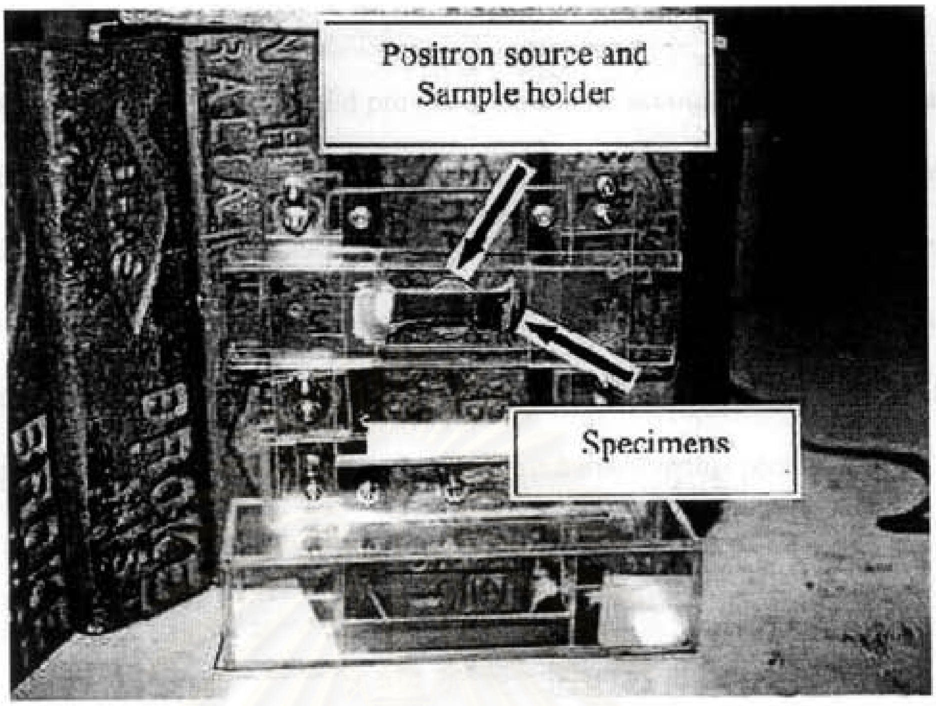

Figure 3.2 The positron source and sample holder
with positron source and two investigated specimens.

Initially, the detection system consisted of a 100 μCi Na^{22} (reference date at 15 July 1996) positron source, a Canberra GC-1020 HP(Ge) detector coupled to a Canberra 2022 Amplifier and a Canberra 35 plus multichannel analyzer (MCA) to process radiation signal and a computer unit to collect information. The HP(Ge) detector was powered by a Canberra 4201 high voltage unit supplying a voltage of 4500 volts and the amplifier was set up to have a shaping time of 1μs with coarse gain and fine gain setting at 10 and 0.665, respectively. The ADC gain in the MCA was set with all 8192 channel actives yielding a dispersion of about 0.5-0.6 keV per channel. The performance of this system was tested with an aluminum sample. After 10 runs of 60 min counting time and S parameter evaluated with $\Delta\varepsilon$ fixed at 1 keV. Figure 3.3 shows S parameter as a function of run number. It was found that the S parameters varied significantly and provided unreliable account for the S values. The fluctuation of S parameter is caused by instability of measurement system which be affected by many variables such as temperature, humidity, electronics noise, high voltage drift, liquid nitrogen level and geometry. A compensating technique to take into account of system variations in evaluating S parameter based on the use of 1274 keV gamma decaying from Na^{22} was thus proposed to solve this problem.

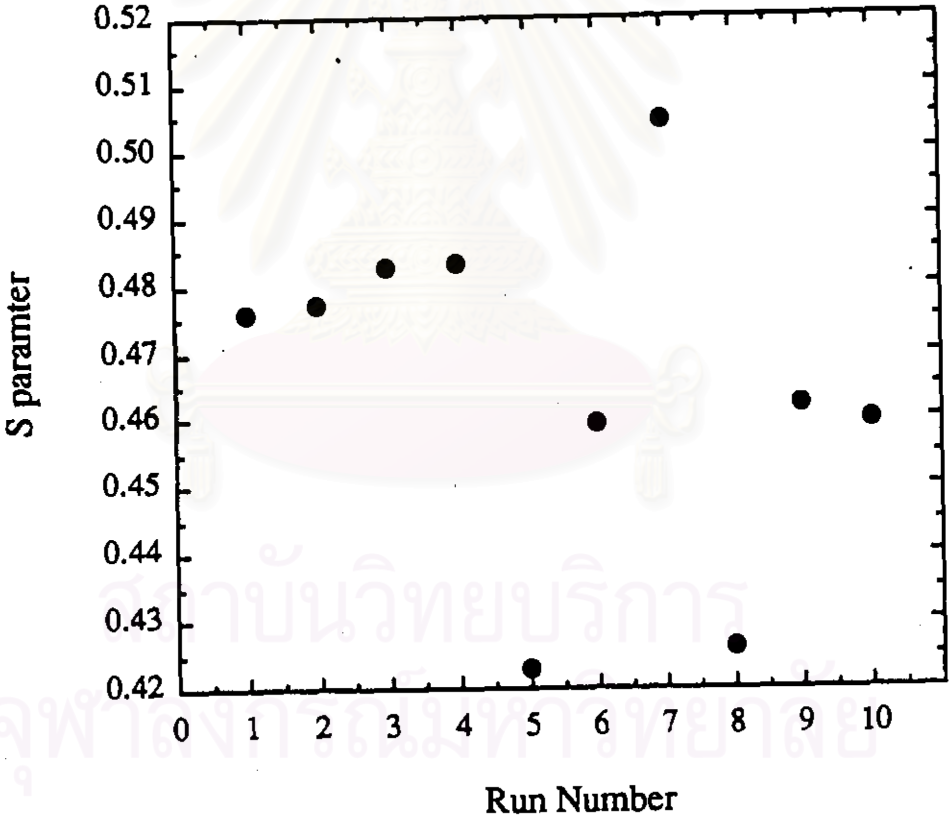

Figure 3.3 Change in S parameter with run number for

a series of experiment on a single aluminum sample.

3.3 Compensating Technique

To correct for the fluctuation in the S values, we proposed the following idea. In evaluating the state of microstructures using DBPA spectroscopy, it was the 511 keV gamma line that will be affected by defects density and distribution. The S parameter which had been described in Chapter II is normally used to evaluate the shape profile of an annihilation line reflecting the state of microstructures inherent in the materials. It should be noted that the selected value of $\Delta\varepsilon$ has not been clearly clarified in most of the literature and the value of 1 keV was cited in a few occasions [3]. The choice of $\Delta\varepsilon$ can greatly affect the values of S parameter; and thus, was found to lack of its clear physical and/or statistical meaning. Furthermore, the measurement system could be affected by many variables such as temperature, humidity, electronic noise, high voltage drift, geometry, resulting in the instability of the peak shape affecting the accuracy in S parameter.

To solve the mentioned problems, we proposed that the 1274 keV gamma rays which decay from Na^{22} be used as an indicator to experimental variations. From the decay scheme of Na^{22}, Figure 2.9, it can be clearly seen that there is a direct correlation between each positron given off from a Na^{22} source and 1274 keV gamma ray. It should be noted that for each emission of a positron, a 1274 keV gamma is also released as a result. However, the 1274 keV spectrum is insensitive to the specimen microstructures while the 511 keV, resulted from positron/electron annihilation within the specimen, is strongly influenced by the specimen microstructures. As a result, the 1274 keV spectrum carries all the information about system variations of each measurements identical to that in the 511 keV spectrum except the state of microstructure allowing us to compensate for the system variations and directly compare the S parameter of varying microstructural states. The technique may be summarized in Figure 3.4 showing the schematic of compensating technique.

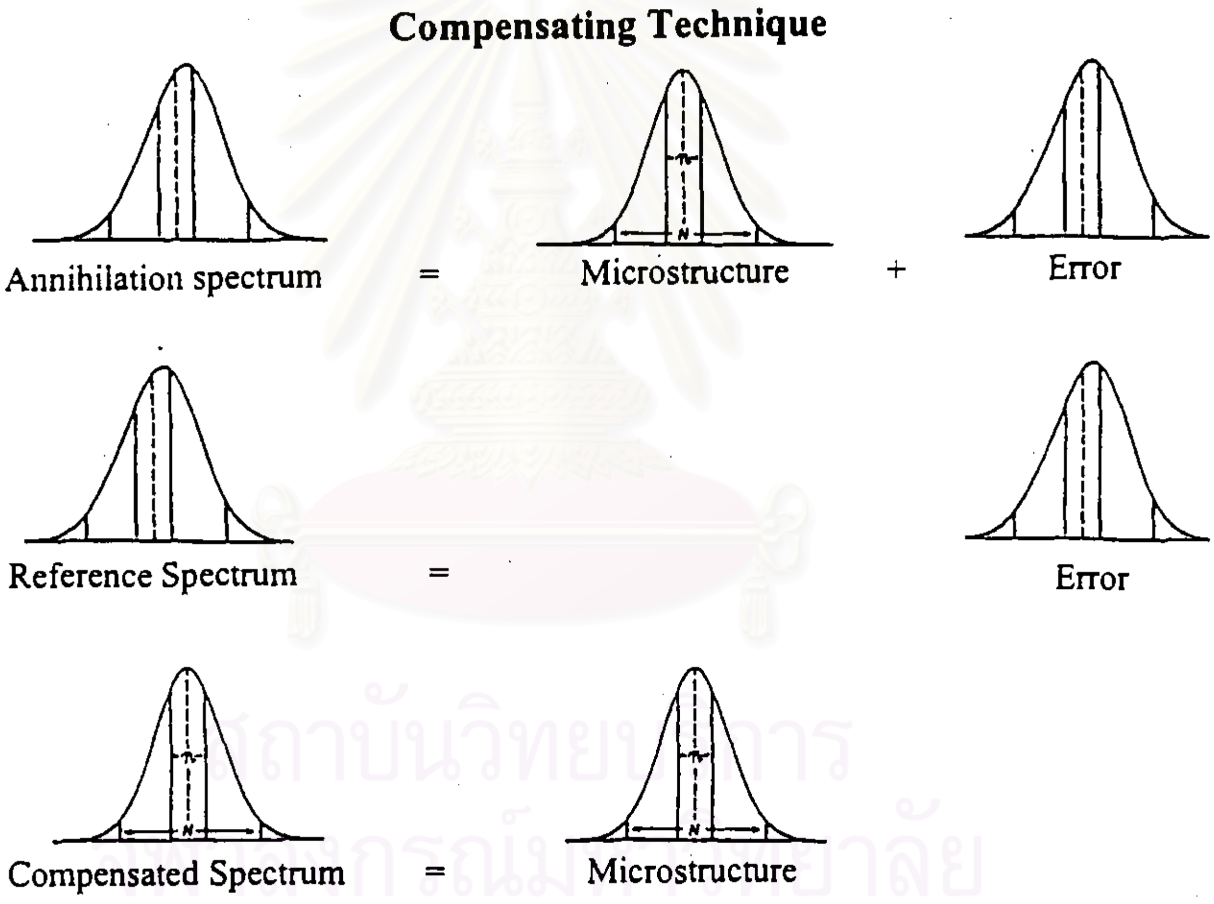

Figure 3.4 The schematic of compensating technique.

The correlation between 511 keV and 1274 keV was then substantiated by plotting the S parameter of $511 \pm 1\,\text{keV}$ and $1274 \pm 1\,\text{keV}$ peak of the aluminum specimen. After 10 runs of 60 minutes counting time, the S values were shown in

Figure 3.5. It can be clearly seen that there is a strong correlation between the 511 keV and 1274 keV peaks. To correct the annihilation spectrum, it is proposed that S value of 1274 keV is fixed to be 0.5 in order to first extract information on the width of centroid channel, Δε value. The S value of 0.5 was chosen since it gives the highest value of standard deviation, σ_S, and can be shown to be written as [18]:

$$\sigma_S = \left[\frac{n(N-n)}{N^3}\right]^{1/2} \tag{3.2}$$

Since the width of the reference spectrum is only affected by system variation and **not** microstructures; the Δε value determined from the 1274 keV spectrum can be used as a reference to evaluates the S parameter of the corresponding annihilation spectrum with system variations already compensated for.

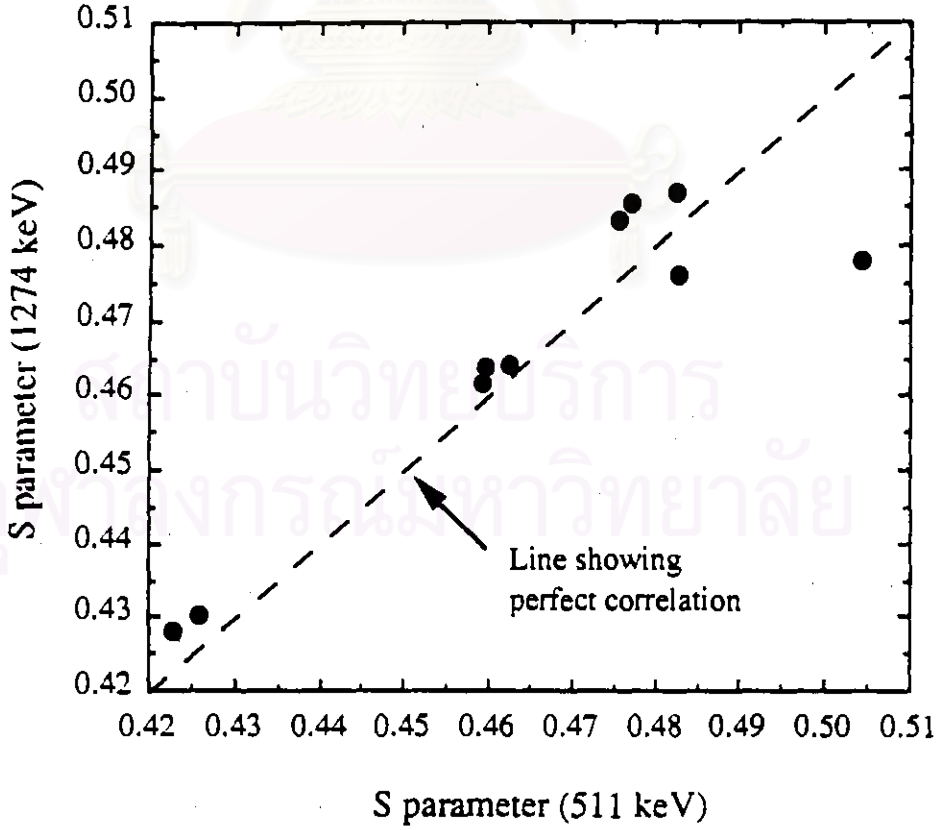

Figure 3.5 Correlation between S parameter of 511 keV and 1274 keV.

(a dash line representing a perfect correlation is also known)

The compensating technique can be applied by first calculating the total area defined as the area under the full width at tenth maximum (FWTM) area of the 1274 keV peak and the centroid area to yield the S value of 0.5. Thus, the $\Delta\varepsilon$ of the reference spectrum is obtained and will be used to calculate the centroid area in the corresponding annihilation spectrum. Figure 3.6 summarizes the procedure used in compensating.

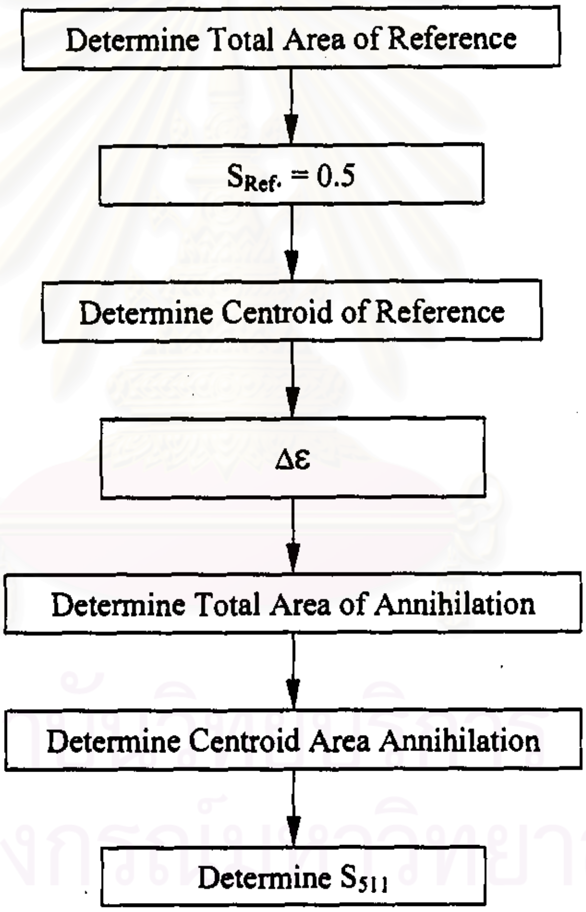

Figure 3.6 Illustration of the procedure used in compensating technique.

This technique was applied to evaluate the S values of the as-received aluminum specimen and then compare the S values previously obtained from fixing $\Delta\varepsilon$ = 1 keV.

It was found that the compensating technique could help to reduce the variation of S value from nearly 20% to less than 2%. Figure 3.7 shows the plot of the raw data was obtain from fixing Δε = 1 keV and the compensated data. Thus, this compensating technique should be useful technique to analyze S parameter However, the evaluation of S values from the reference spectrum had large errors caused by hand calculations. To help reducing time and error associated with Δε values extracted from the reference spectra, a computer program [33] was written to automatically extract the total peak area, Δε values of the reference spectra with $S = 0.5 \pm 0.00000001$ and also the corresponding S values of the annihilation spectra.

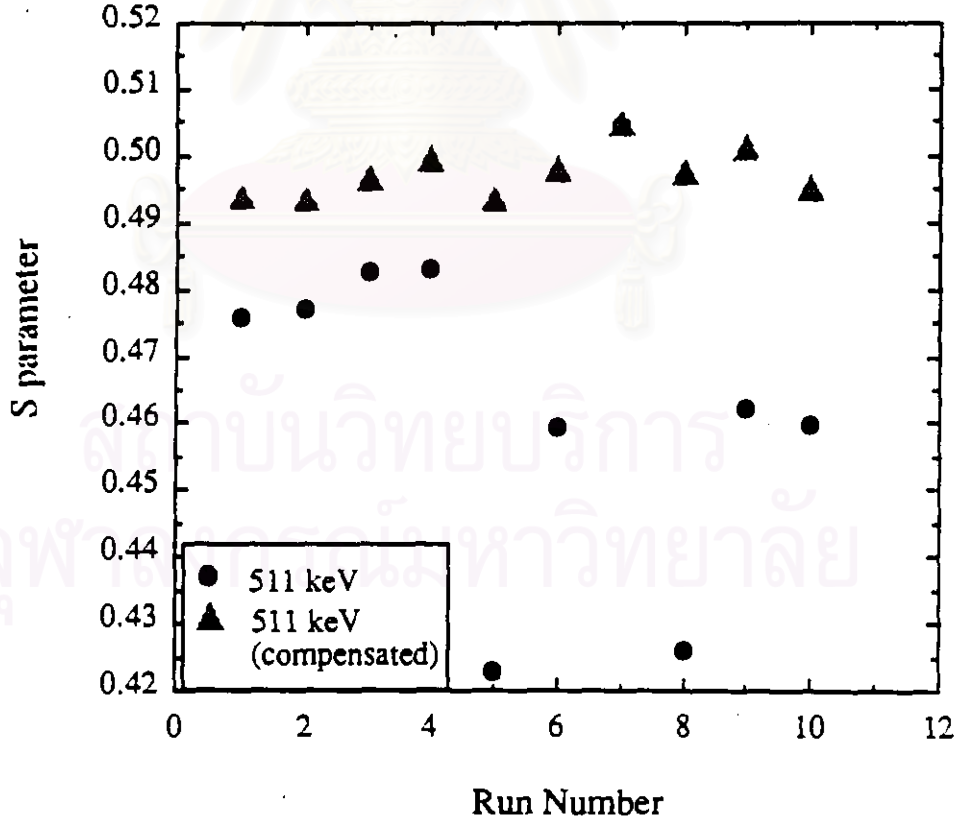

Figure 3.7 Change in S parameter and the corrected S parameter for a series of experiments on a single aluminum sample.

To further improve our system, we proposed to use two biased amplifiers to gain the signals and two MCAs for simultaneously recording both 511 and 1274 keV spectra. One of the biased amplifiers was set up to have bias level of 5 volts with coarse gain and fine gain at 5 and 0.53, respectively, and a MCA is dedicated to record only the 511 keV spectrum while the other biased amplifier was set up to have bias level of 7 volts with coarse gain and fine gain at 5 and 0.64, respectively, and the other MCA is used to record the 1274 keV gamma ray spectrum. The two MCA system enabled us to present the energy region between 358-567 keV and 1131-1337 keV using 4096 channels of memory providing a dispersion of about 48-51 eV per channel. Thus, the energy resolution of the spectrum dispersion was improved by about 10 folds compare to the one MCA system.

The DBPA measurement set up now consists of a 59 μCi Na^{22} (15 July1998), positron source, a Canberra GC-1020 HP(Ge) detector is powered by a Canberra 4201 high voltage unit supplying a voltage of 4500 volts, a Canberra 2022 amplifier which is set up to have a shaping time 1 μs, with coarse gain and fine gain setting at 30 and 0.720, respectively, two Ortec 408A biased amplifiers, two Canberra 35 plus MCAs and a computer unit to collect information. Figure 3.8 shows the schematic diagram of developed DBPA measurement with two MCAs. In addition, it is necessary to reduce the radiation background from surrounding area by shielding the detector with 70 mm thick lead bars. The lead bars were placed in the front of and around the detector not only for radiation shielding but also for collimating purposes.

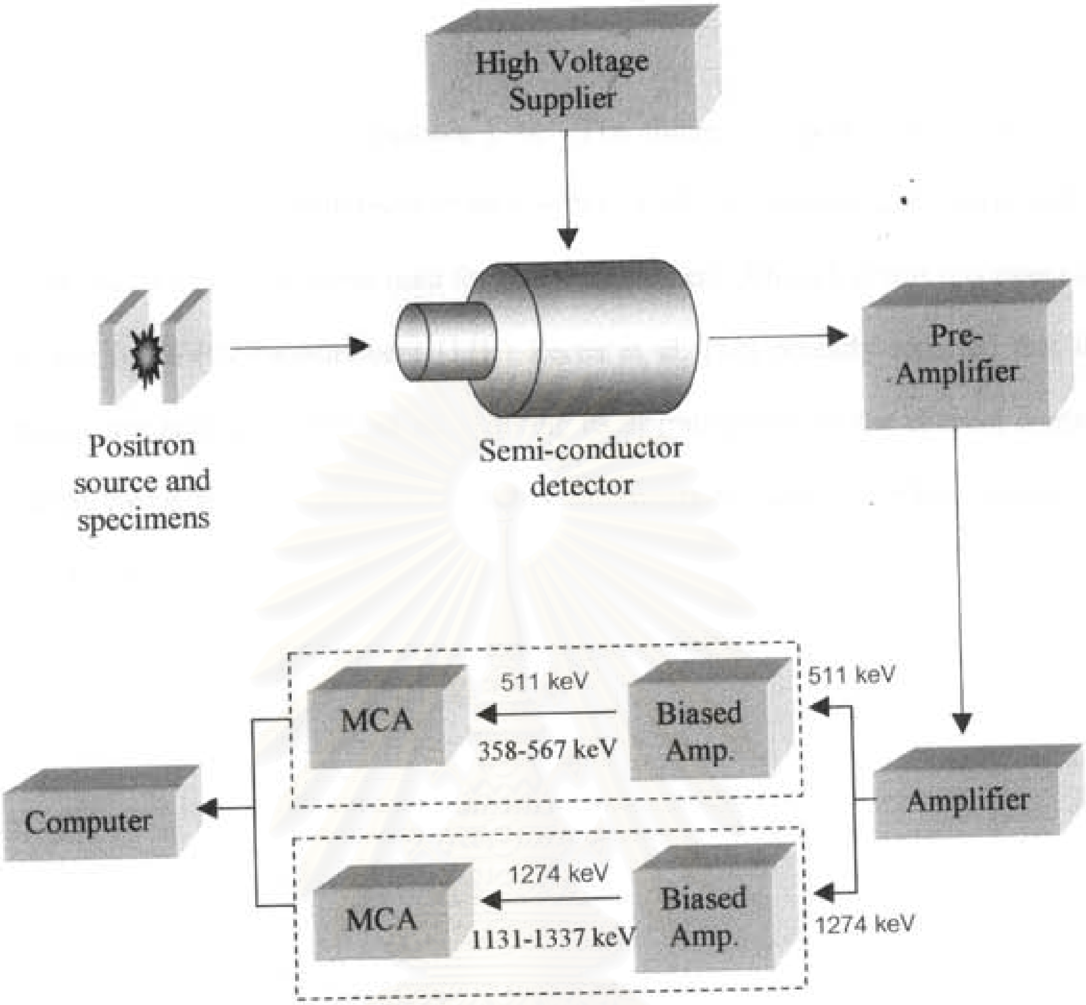

Figure 3.8 The schematic diagram of the developed DBPA measurement system.

A 7 mm hole was drilled through one of the lead shields perpendicular to the detector surface to collimate the radiation beam to reduce the system dead time resulting from high positron source activity. The high source activity introduces a high dead time, defines as the minimum time that must separate two events in order that they will be recorded as the separate pulse [34], in the electronic system. The effect of high dead time on the measurement system is a loss of the true events leading to a reduction in detector's efficiency. The 7 mm collimator was found to reduce a 50% dead time to

10% and is considered acceptable [34]. The figure 3.9 shows the set up of the developed DBPA measurement system with two MCAs. It should be noted that the peak stabilizer system is not used for this measurement although it was recommended to use in DBPA measurement [12]. Zecca et al. [16] recently reported that this instrument should be avoided since during its operation acts on the channel contents on the two sides of the spectrum and therefore interferes directly with the quantity to be measured.

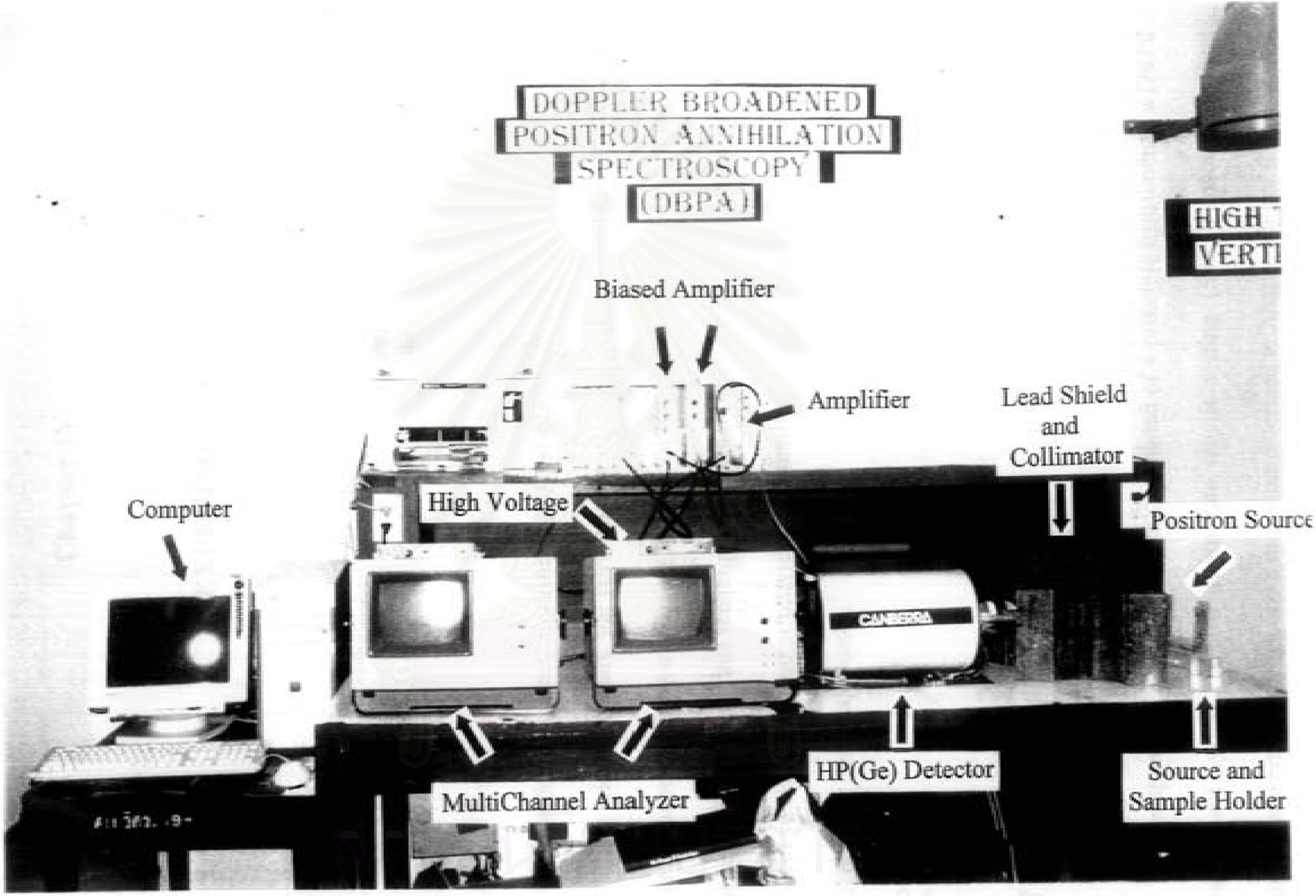

Fig 3.9 The set up of DBPA measurement system with 2 MCAs.