CHAPTER I

INTRODUCTION

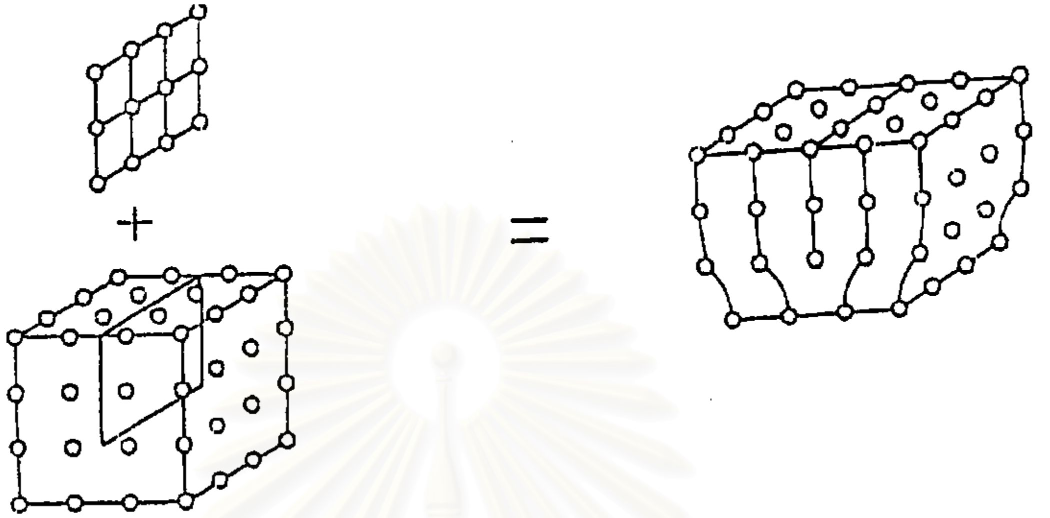
1.1 Background

Currently, there are many non-destructive testing (NDT) techniques available to detect the existence of defects in materials such as radiography, eddy current and ultrasonic testing. These techniques have been the important tools in detecting and locating the defects without affecting the service performance of the structure. Although these NDT methods provide the means to detect flaws and defects before catastrophic failure, most are not sensitive enough to reveal small cracks until they are too far advanced to be repaired. More importantly, none of the above mentioned techniques yield any information on the state of microstructures which are often responsible for materials failure. Recently, the attention has been paid to using the annihilation characteristics of positrons which are related to electron distribution in materials as a possible NDT method in studying atomic defects in solids.

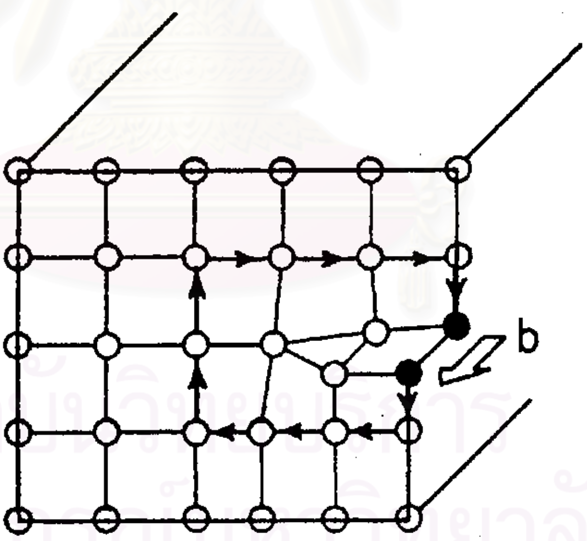
Positron annihilation (PA) can be used for evaluating the atomic defects because of the annihilation characteristics of positrons are related to electron distribution in materials. In the annihilation process, two gamma rays at approximately 180° to one another are emitted from the center of the mass of the positron/electron pairs. A very slight departure from 180° is directly proportional to the transverse component of the momentum of the pair. The momentum of the electrons involved in such collisions can thus be calculated from the geometry and intensity of the gamma rays.

For the simplest method, positron annihilation (PA) evaluation uses the Doppler-broadened positron annihilation (DBPA) spectroscopy to evaluate the microstructural condition in materials because it is easier to set up and perform than other PA techniques. The system for measuring DBPA spectroscopy consists of a positron source, Na^{22}, a semi-conductor detector, a gamma ray detection system and a computer unit to process and collect information.

The main theme of this thesis is to develop DBPA spectroscopy technique as a NDT technique to evaluate materials degradation at the microstructural level. For this thesis, the DBPA spectroscopy will be applied to evaluate dislocation density in materials. Dislocations are line defects in the crystal structure and responsible for plastic deformation in metals. Figure 1.1 shows a schematic representation of an edge and a screw dislocations. Dislocations can be directly generated as the stress is applied onto a specimen using a conventional tensile machine, and their density is known to be proportional to the square of the applied stress.



(a)



(b)

Figure 1.1 Schematic representation of (a) an edge dislocation

(b) a screw dislocation.

To ensure characteristics and performances of the developed DBPA measurement system, our results will be benchmarked against some previous research [1], Figure 1.2. Not only that dislocations are important in responsible for plasticity but also its presence, implying stress conditions in materials, is critical in certain metallurgical phenomenon such as intergranular stress corrosion cracking (IGSCC).

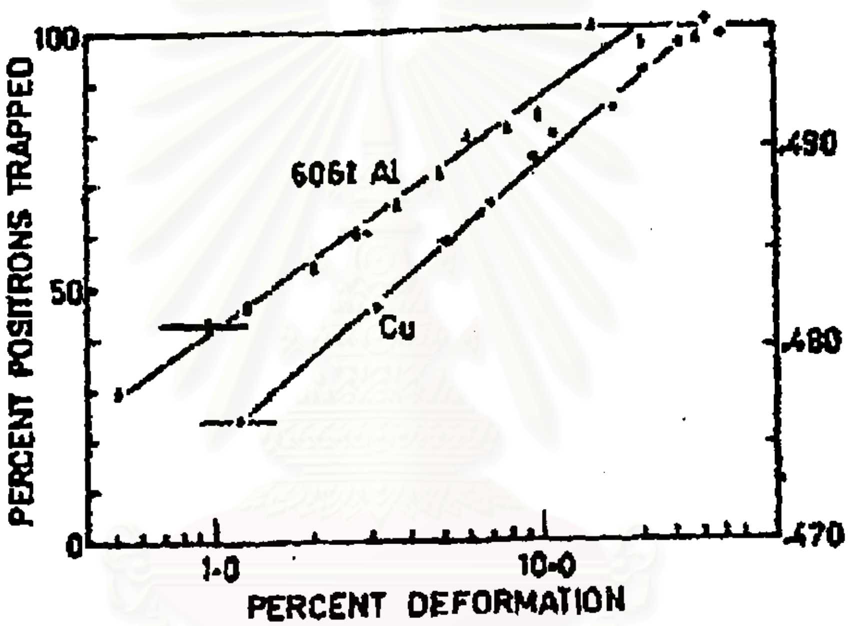


Figure 1.2 The lineshape changed with the percentage reduction in sample thickness resulting from unidirectional cold rolling. [1]

It is well known that IGSCC can occur by a manifestation of three factors, stress, aggressive environment and susceptible microstructures. The susceptibility of stainless steels to IGSCC is typically characterized by the level of chromium content at grain boundaries. Figure 1.3 shows the correlation between the level of chromium content and intergranular cracking in nickel base alloys. Several NDT techniques have now been employed to detect cracks in material, already resulted from IGSCC, but none can be used to evaluate the susceptibility of stainless steels prior to its failure by IGSCC. This research will attempt to test the possibility of employing the DBPA technique to evaluate the susceptibility of stainless steels to IGSCC.

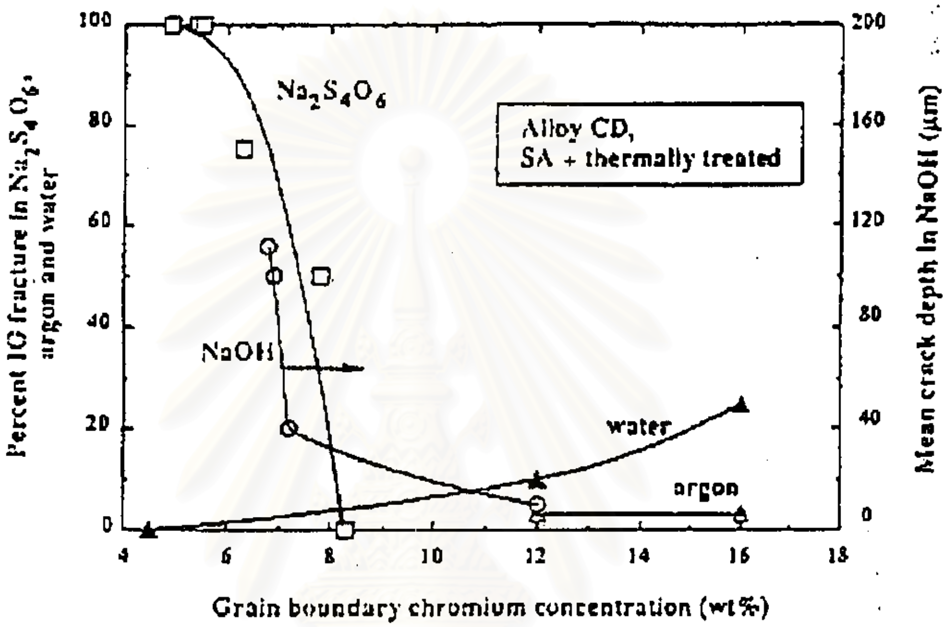


Figure 1.3 Effect of chromium concentration on intergranular cracking in argon and water of nickel-base alloys.

This is accomplished by heat treating solution annealed stainless steel specimens at 650°C for various times producing microstructures with different degrees of chromium depletion at grain boundaries, sensitization. The specimens will then be microstructurally characterized using the developed DBPA technique.

1.2 Objectives

1.2.1 To develop doppler-broadened positron annihilation (DBPA) spectroscopy technique.

1.2.2 To apply DBPA technique to evaluate dislocation density in materials.

1.3 Scope of thesis

1.3.1 To study and develop DBPA technique using Na^{22} as a positron source and a HP(Ge) detector.

1.3.2 To test and benchmark the performance of the developed DBPA system with the previous research by evaluating dislocation density in copper specimens.

1.3.3 Apply the DBPA technique to evaluate the sensitization in stainless steels with varying microstructures.

1.4 Methodology

1.4.1 To conduct literature search and review.

1.4.2 To develop DBPA technique

1.4.3 To test and benchmark the performance of the developed system with previous research.

1.4.4 To perform heat treatment on stainless steels at various times and temperatures.

1.4.5 To establish the correlation between DBPA signals and varying microstructures in stainless steels.

1.4.6 Conclude research results and write up the thesis.

1.5 Potential applications of the thesis

1.5.1 Obtain a new technique to evaluate dislocation density in copper and sensitization level in stainless steels.

1.5.2 This technique may be applied to test other materials properties; for example, airplane parts.

1.6 Relative researches

In 1969, MacKenzie of the University of Guelph, Canada, researched on the Doppler-broadened positron annihilation lineshape as an indicator of defects in metallic lattices. He found that using a high performance spectroscopy (FWHM of 1.20 keV at 514 keV) in conjunction with a very thin positron source could not only distinguish between annihilation spectra in different metals but also measure quantitatively the influence of lattice defects introduced by plastic deformation or by heating.

In 1970,MacKenzie, Eady and Campbell of the University of Guelph, Canada, researched into the interaction between positrons and dislocations in copper and in aluminum alloy. The annihilation spectrum was used to measure the percentage of positrons trapped by dislocations in pure copper and aluminum alloy. Preliminary result was found that the percentage of trapping and the sample hardness were linear functions with deformations on logarithmic scale. In addition, they proposed to use DBPA spectrum as a quantitative tool for measuring dislocation density.

In 1979, Kerr et al. of University of East Anglia, England, researched on a simple correction technique for positron annihilation lineshape measurement. They proposed to use a reference spectrum and a mathematical formula ,which will be described in chapter II, for correcting S value which is affected by changing of energy resolution during the experiment. They applied their equation to a series of the measured S values for the reference spectrum which was 478 keV Be^7 gamma spectrum and the DBPA spectrum in indium samples. Preliminary result was the standard deviation of the raw S data was diminished to 50% after correction.

In 1979, Campbell and Schulte of the University of Guelph, Canada, researched on stability problems in annihilation photon energy spectroscopy. They proposed to use a reference gamma source and a mathematical formula, which will be described in details in chapter II, to correct S value. They concluded that their method was slightly better than Kerr's method.

In 1983, Zecca, Russa and Naia of the University of Trento, Italy, researched on the counting-rate-dependence of the S parameter in Doppler-broadening positron annihilation measurement. They proposed two methods for correcting and/or compensating S values. The first method could be obtained by setting the windows of centroid area and total peak area referring to energy scale rather than to a channel number position. This method could correct the line-shift error and the line-broadening error on the S parameter. The last method used a movable gamma source. A feed-back circuit moves the gamma source so as to keep the total count rate constant. This method was found to be too difficult to implement.