Chapter IV

EXPERIMENTS

4.1 Introduction

This thesis was accomplished by integration of two major tasks; the first task was to test and benchmark the performance of the developed DBPA technique with the previous research by evaluating the dislocation density in copper specimens. The final task was to apply this technique to evaluate the sensitization conditions of 304 stainless steels. This chapter provides information on the experimental apparatus and procedure for both major tasks.

4.2 Experimental apparatus

The apparatus used for this work consisted of 4 principle instruments; a DBPA measurement system, a slow strain rate tensile testing (SSRT) machine, a high temperature vertical furnace and a mini-computerized milling machine. The details of these instruments are described below:

4.2.1 DBPA measurement system

The DBPA spectroscopy consists of a Na^{22}, positron source, a gamma ray detection system and a computer unit to obtain radiation signal and process information, respectively. The positron source is a 20 mm diameter 59 μCi Na^{22} (July 1998) covered with 0.005 mm Ni thin foil with an active window of 9.5 mm in diameter. The other standard radioactive sources, Na^{22}, Co^{60} and Ba^{133}, were also used to calibrate energy for evaluating the energy resolution of the detection system. The gamma ray detection system consists of a Canberra GC-1020 HP(Ge) detector powered by a Canberra 4201 high voltage unit supplying an operating voltage of 4500 volts and coupled by a Canberra 2022 Amplifier which is set up to have 1 μs shaping time with coarse gain and fine gain setting at 30 and 0.720, respectively Two Ortec 408A Biased Amplifiers and two Canberra 35 plus multichannel analyzer were set with 4096 channels active. One of MCAs and bias amplifier was set up to have a bias level of 5 volts with a coarse gain and a fine gain set at 5 and 0.53, respectively. This MCA is dedicated to record only 511 keV spectrum. The other biased amplifier was set up with a biased level of 7 volts with a coarse gain and fine gain set at 5 and 0.64, respectively. The other MCA is dedicated to record only 1274 keV spectrum and it was set up. Figure 3.9 shows the set up of the DBPA measurement system.

4.2.2 Slow strain rate tensile (SSRT) machine

The SSRT machine is a useful tool for evaluating the degree of sensitization in austenitic stainless steels. The SSRT machine is designed to perform tensile or compressive test in hazardous environment at a very low extension rate ($<5\times10^{-6}$ in/s) capable of travelling in both upward and downward direction for compressive and tensile test. The load frame of the SSRT is entirely made of carbon steels. The upper pull rod is threaded and fix to the upper load frame while the lower pull rod is attached to a lead screw driven by a reduced gear box with a ratio of 1:180,000 providing a very low extension rate capable of travelling in both upward and downward direction for compression and tension test, respectively. The gear box is powered by a ¼ HP motor capable of adjusting speed from 0-30 rpm. An OMEGA linear variable differential transformer (LVDT) type transducer, is employed for extension measurement with a resolution better than 2 μm/mV. A 1000-lb miniature load cell obtained from transducer technique is attached to the upper pull rod for measuring load on specimen. Figure 4.1 shows the SSRT unit and Figure 4.2 shows the details of the load train together with an example of failure specimen test using the SSRT machine.

4.2.3 High temperature vertical furnace

This furnace was constructed with the purpose to better controlling heat treating conditions of subjected materials. The furnace tube is made of impervious ceramics containing 60% Al_2O_3-pythagoras-with a CN9000A Omega temperature controller and a maximum working temperature of 1400°C. The furnace was powered by resistive heating of 6 silicon carbide heating elements providing at least 2 kW of power. Figure 4.3 shows the high temperature vertical furnace.

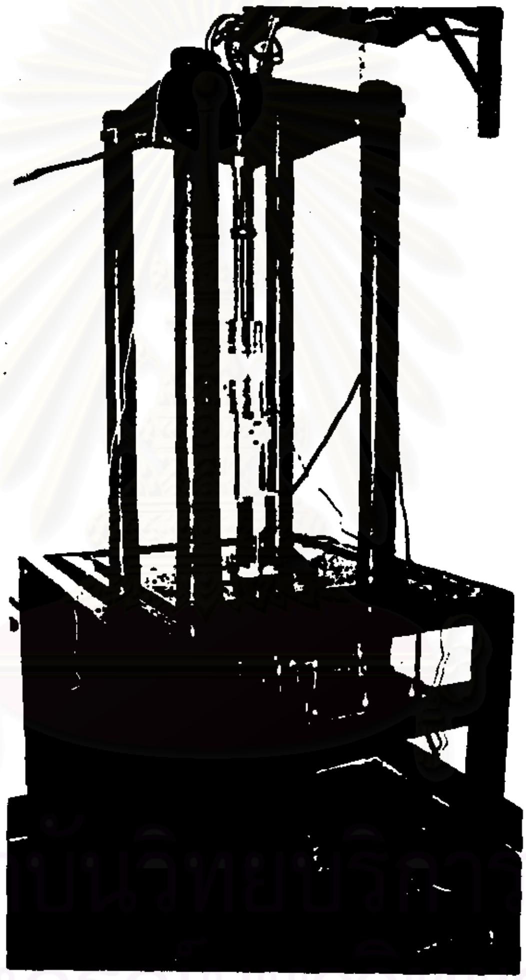

Figure 4.1 The SSRT unit

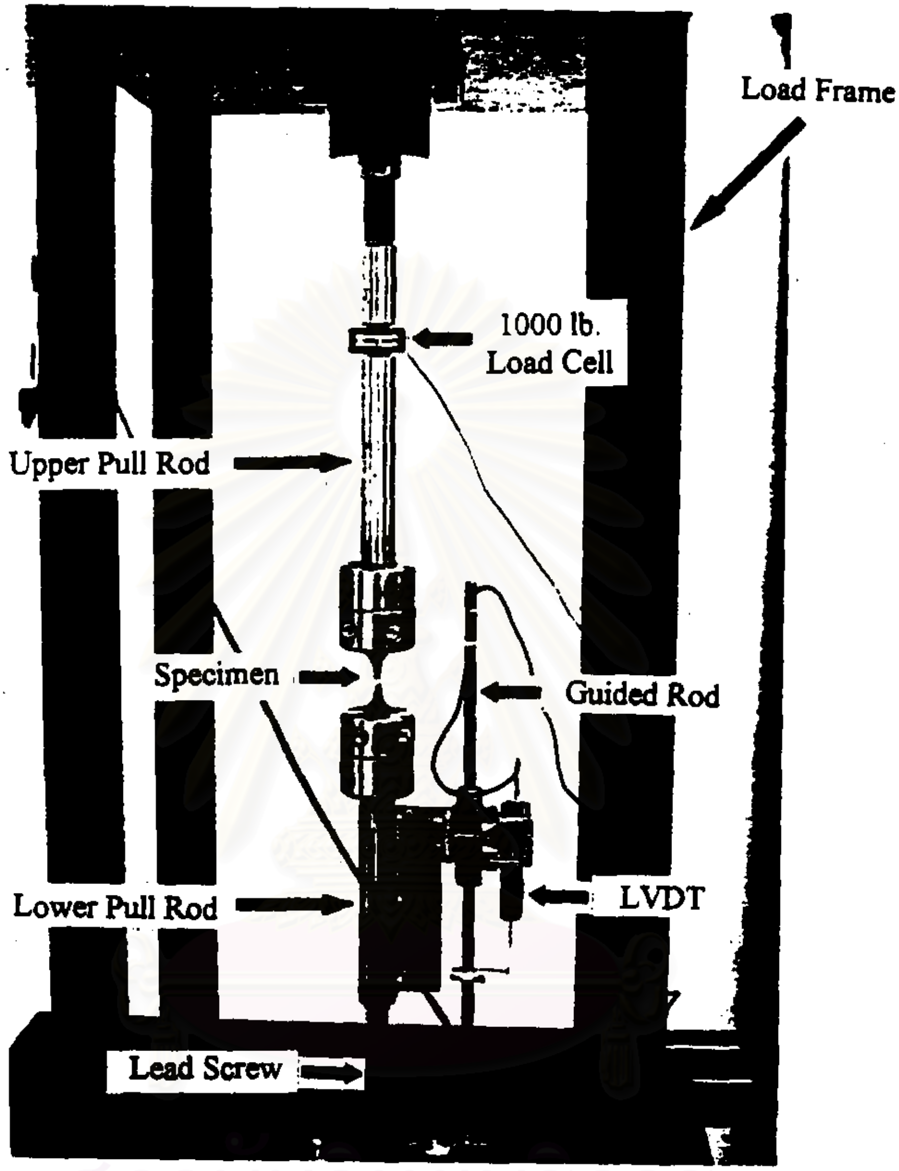

Figure 4.2 The details of load train together with

an example of failure specimen tested using the SSRT machine.

Figure 4.3 The high temperature vertical furnace.

4.2.4 Mini-computerized milling machine

The machine is used to prepare tensile specimen as designed in the Auto-Cad program. Equipped with a special high speed TiN-carbides coarse and fine cutting tools, the machine can prepare fine finish tensile specimen leaving no tool marks deep enough to act as stress concentrating notches. Figure 4.4 shows the Mini-computerized milling machine- Mimaki 300.

Figure 4.4 Mimaki-300 mini-computerized milling machine

4.3 Experimental procedures

The experiment of this thesis is separated in 2 parts; the first part is to test and benchmark the performance of the developed DBPA technique by evaluating dislocation density in copper specimens and the other part is to apply this technique to evaluate the sensitization condition of 304 stainless steels.

4.3.1 Evaluation of dislocation density in copper

As-received copper sheets with $28 \times 121 \times 1.0$ mm in dimension were cut by Mimaki-300 mini-computerized milling machine into tensile specimens as shown in Figure 4.5.

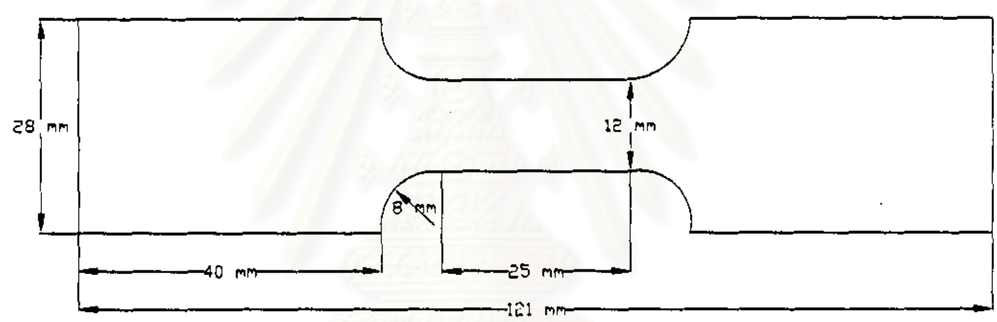

Figure 4.5 Schematic drawing of a copper tensile specimen.

Table 4.1 lists chemical composition of copper specimens from spark emission spectrometry. The specimens were wrapped in stainless steels foil and solutionized in an oven at temperature of 800°C for 20 min. After solution annealed, the specimens were polished to remove the oxide layer on the gauge length area. The samples were strained to 3.4%, 6.6%, 9.7%, 11.9% and 19.6% deformation using the SSRT an initial strain rate of about 10^{-6}.

Table 4.1 The chemical composition of copper specimen.

Alloys	Weight (%)	Alloys	Weight (%)
Cu	99.850	Fe	0.0365
Al	0.00545	Pb	0.0542
Bi	0.00448	Zn	< 0.0050
Si	0.00617	Mn	< 0.0050
Ni	0.0235	P	<0.00200

The DBPA technique was then applied to the samples of different deformation level by fixing count area of the reference spectrum equals to 420,000 within 800 channels. To minimize the electronic noises which introduced error in DBPA signals, this measurement must be performed at night when the activities in the building are low. A total of 4 series of tests were run altogether. In each series, one non-deformed specimen and 3 different strained specimens were tested and at least 3 measurements were made on each samples. The test matrix is shown in Table 4.2. The idea was that the change in S parameter must always be compared with the undeformed specimens for each series since it is possible that the S parameter may not stay the same on each series.

Table 4.2 The test matrix of copper specimens

Strain (%)	No. 1	No. 2	No.3	No. 4
0	X	X	X	X
3.4	X			X
6.6		X	X	
9.7	X	X		
11.9		X	X	X
19.6	X		X	X

4.3.2 Evaluation of sensitization condition of 304 stainless steels

A D-189 Thainox 304 stainless steel sheet was used in this study. Its chemical composition analyzed using spark emission spectrometry is listed in Table 4.3. The as-received sheet was cut into rectangular specimens with a dimension of $25 \times 76 \times 1.2$ mm. The specimens were wrapped in stainless steels foil, to reduce the oxidation during annealing, and then solutionized at 1070°C for 30 min under flowing argon. After the annealing was completed the specimens were quenched in water to room temperature. The solution annealed were then sensitized by heat treating at 650°C for 2 hr, 8 hr and 16 hr. After heat treatment, the samples were mechanically polished to remove the oxide layer down to 1 μm diamond paste. Specimens were then tested using the DBPA technique and at least 5 measurements were made on each specimens' condition.

Table 4.3 The chemical composition of D-189 Thainox 304 stainless steels.

Alloys	Weight (%)	Alloys	Weight (%)
Fe	71.91	C	0.0436
Cr	18.23	P	0.0301
Ni	8.99	N	0.0214
Mn	1.67	Nb	0.019
Si	0.462	Al	0.0116
Mo	0.173	Ti	0.00962
Cu	0.082	S	0.00539
Co	0.0453	Sn	0.00312