Chapter V

EXPERIMENTAL RESULTS AND DISCUSSION

5.1 Introduction

This chapter provides the results of two experiments described in chapter IV together with the discussion and conclusion of the experimental results. The change in the S parameter in the deformed copper will be mainly discussed from the dislocation dynamics point of view. Both the generation and annihilation rate of dislocations will be discussed and related to the observed change in the S parameters. The correlation between the sensitized conditions of stainless steels and corresponding change in S parameter will be presented together with the result on the embrittlement in thiosulfate solution.

5.2 Experimental Results & Discussion

5.2.1 Evaluation of dislocation density in copper

The copper tensile specimens which were used to evaluate the dislocation density were strained to 3.4%, 6.6%, 9.7%, 11.9% and 19.6%deformation by using the SSRT unit and an initial strain rate of about 10^{-6}. The curve of stress and strain is shown in Figure 5.1.

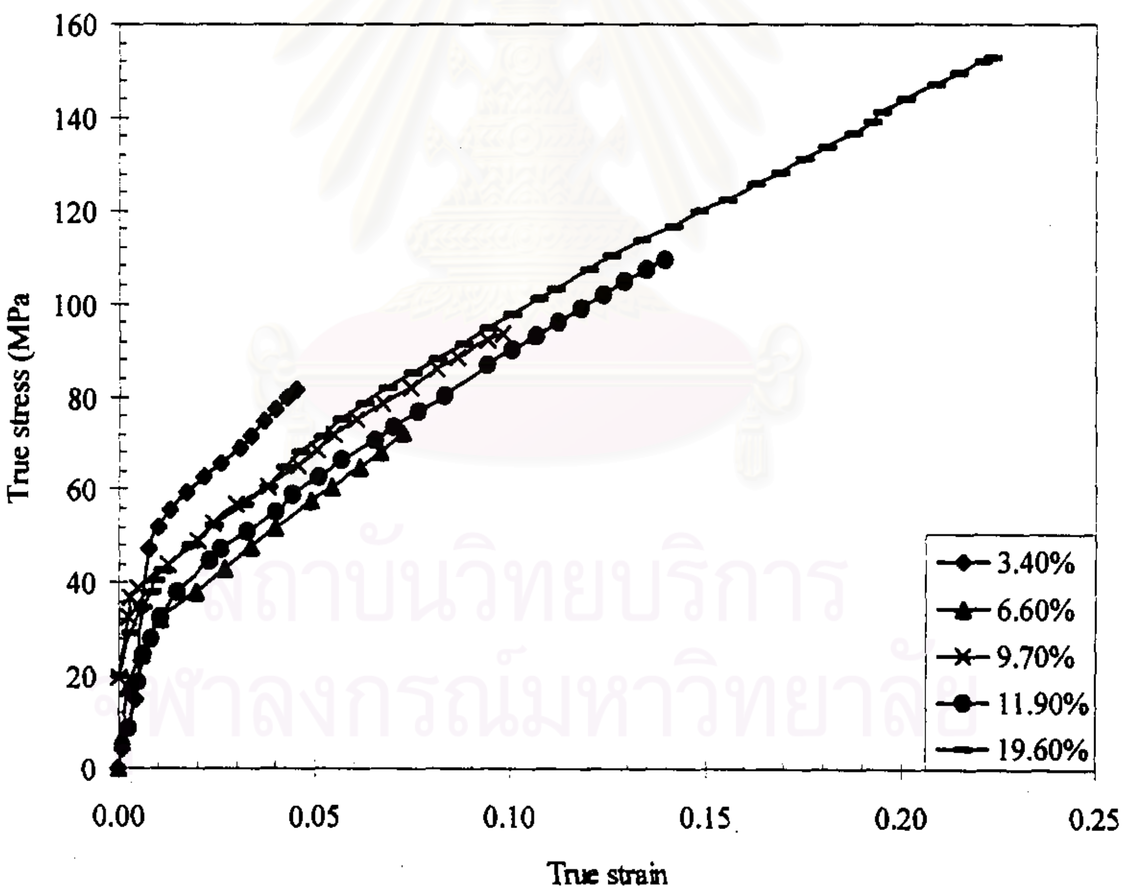

Figure 5.1 The true stress-true strain behavior for the copper specimens.

Table 5.1 (a), (b), (c) and (d) show the results of all measurements and the corresponding correlation of average S values and percent deformation were shown in Figure 5.2 (a), (b), (c) and (d), respectively.

Table 5.1(a) S values obtained from the first series of copper specimens with standard deviation (σ).

Deformation (%	Run No.1	Run No.2	Run No.3	$S_{avg.}$	σ
0.0	0.49633	0.49449	0.49330	0.49471	0.00088
3.4	0.50037	0.49359	0.49447	0.49614	0.00213
9.7	0.50994	0.51355	0.51495	0.51281	0.00149
19.6	0.49257	0.49386	0.48182	0.48942	0.00382

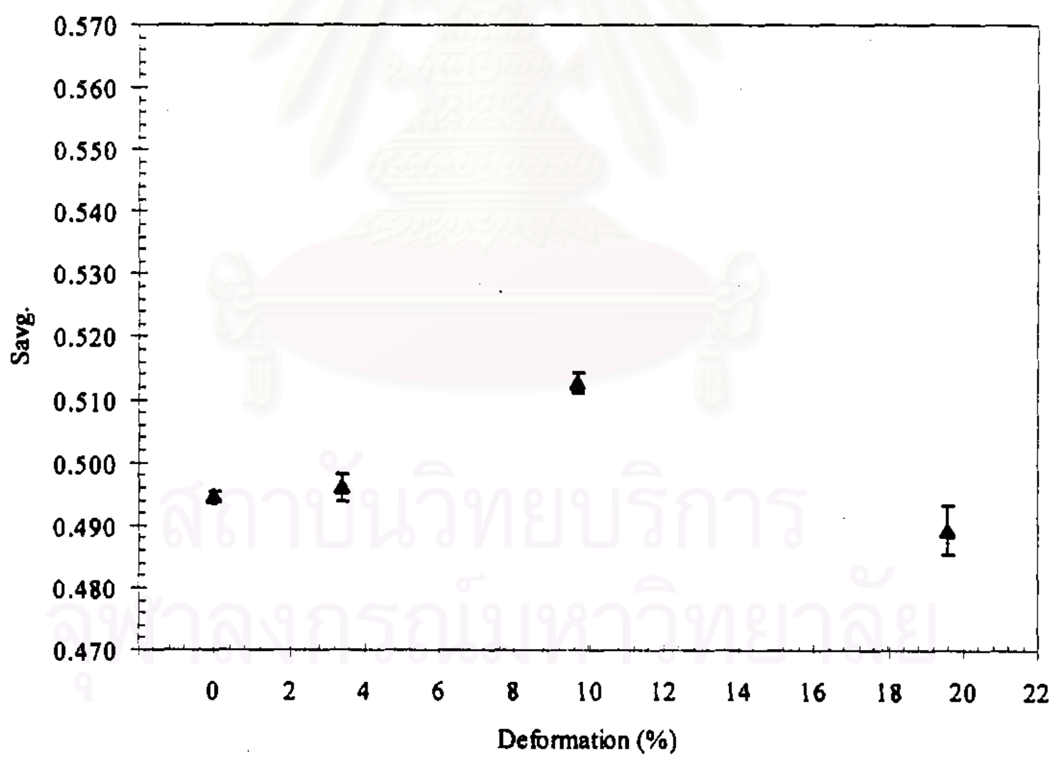

Figure 5.2 (a) The correlation between average S values as a function of percent deformation of the first series for copper specimens. Error bars represent a standard deviation.

Table 5.1(b) S values obtained from the second series of copper specimens with standard deviation (σ).

Deformation	Run No.1	Run No.2	Run No.3	Run No.4	Run No.5	$S_{avg.}$	σ
0.0%	0.49052	0.52210	0.49502	0.52042		0.50702	0.00828
6.6%	0.51539	0.51485	0.57104	0.54036		0.53571	0.01328
9.7%	0.54544	0.56115	0.51390	0.53196		0.53811	0.01004
11.9%	0.53484	0.57794	0.55440	0.54128	0.55880	0.55345	0.00750

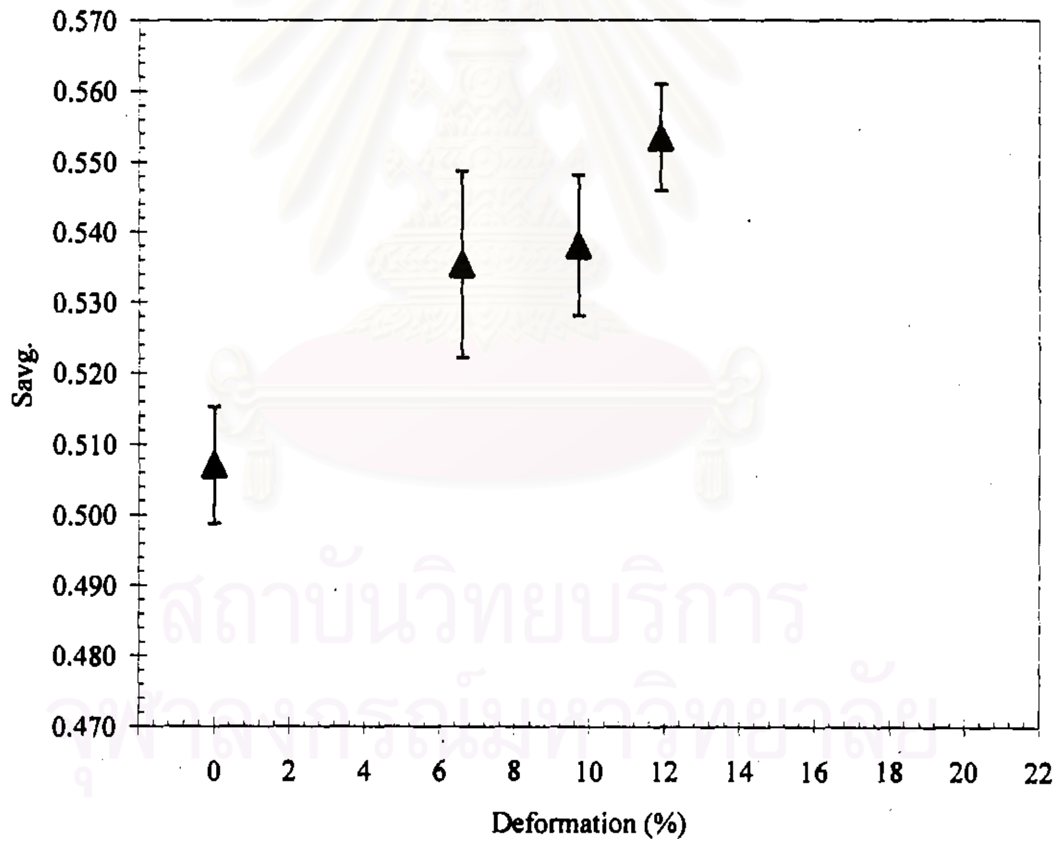

Figure 5.2 (b) The correlation between average S values as a function of percent deformation of the second series for copper specimens. Error bars represent a standard deviation.

Table 5.1(c) S values obtained from the third series of copper specimens with standard deviation (σ).

Deformation (%)	Run No.1	Run No.2	Run No.3	$S_{avg.}$	σ
0.0	0.49662	0.48451	0.50045	0.49386	0.00480
6.6	0.54762	0.49209	0.53964	0.52645	0.01733
11.9	0.49988	0.49264	0.51342	0.50198	0.00669
19.6	0.48773	0.48210	0.49924	0.49069	0.00543

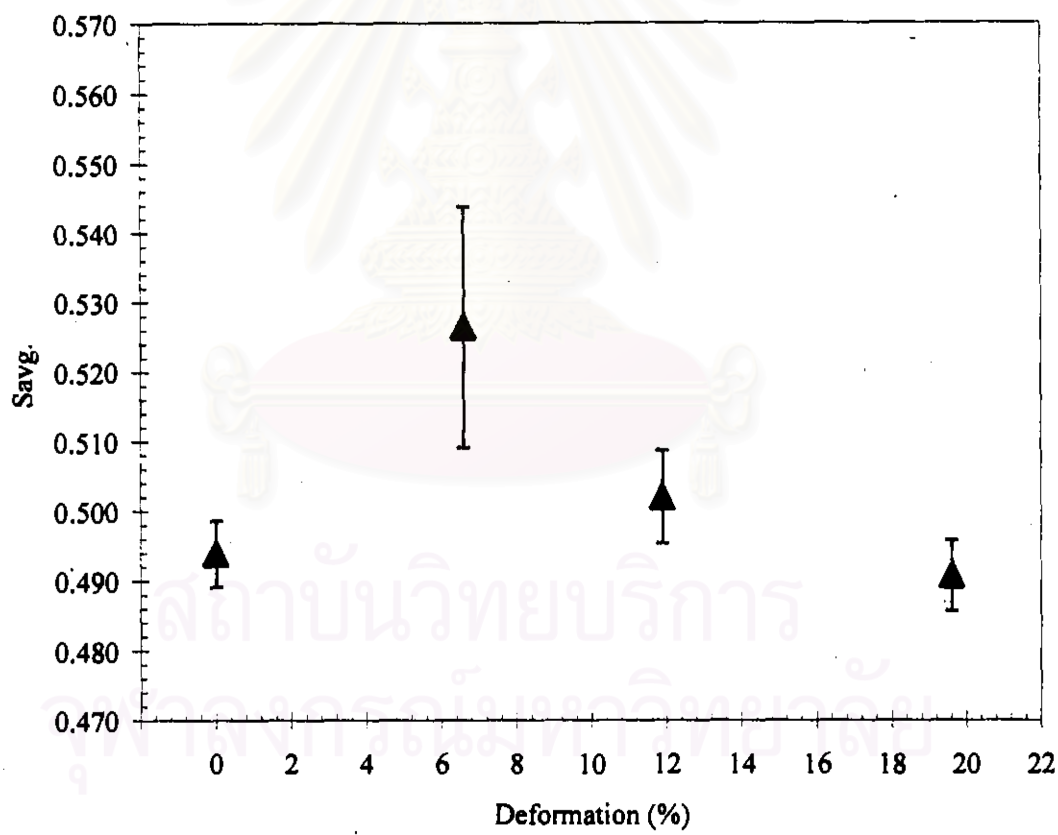

Figure 5.2 (c) The correlation between average S values as a function of percent deformation of the third series for copper specimens. Error bars represent a standard deviation.

Table 5.1(d) S values obtained from the fourth series of copper specimens with standard deviation (σ).

Deformatio	Run No.1	Run No.2	Run No.3	Run No.4	Run No.5	$S_{avg.}$	σ
0.0	0.51880	0.52571	0.51797			0.51858	0.00395
3.4	0.50647	0.56279	0.55328	0.47127	0.51320	0.52140	0.01663
11.9	0.53771	0.51937	0.53379	0.54510		0.53400	0.00541
19.6	0.46745	0.54242	0.53433	0.56140	0.50106	0.52139	0.01659

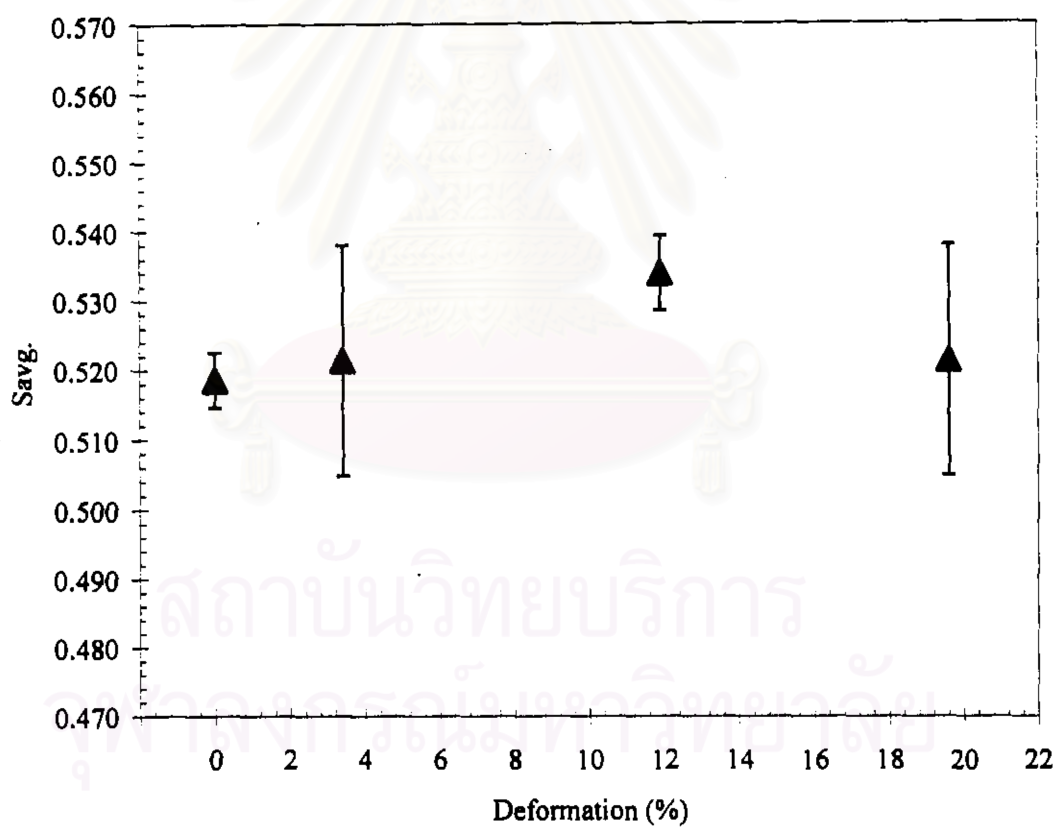

Figure 5.2 (d) The correlation between average S values as a function of percent deformation of the fourth series for copper specimens. Error bars represent a standard deviation.

Table 5.2 presents the change in S values, ΔS, of deformed specimens as compared to an undeformed specimen. Figure 5.3 shows the correlation of the change in S values of deformed specimens as compared to an undeformed specimen.

Table 5.2 The changes of S value of deformed copper specimens as compare to an undeformed specimens with standard deviation (σ) calculated by error propagation.

Deformation	the first series		the second series		the third series		the fourth series	
(%)	ΔSavg.	σ	ΔSavg.	σ	ΔSavg.	σ	ΔSavg.	σ
0.0	0.00000	0.00125	0.00000	0.00117	0.00000	0.00679	0.00000	0.00559
3.4	0.00143	0.00231					0.00282	0.00171
6.6			0.02839	0.01565	0.03259	0.01798		
9.7	0.01800	0.00173	0.03110	0.01301				
11.9			0.04644	0.01117	0.00812	0.00775	0.01542	0.00696
19.6	-0.00529	0.00392			-0.00317	0.00696	0.00281	0.01705

From Figure 5.2 (a)-(d), it is clear that S parameters extracted from the developed DBPA system are reproducible. Furthermore, their differences with deformation were statistically significant with maximum standard deviations of about 2%. When the average change in S parameter, ΔS, of deformed specimens with an undeformed were plotted, ΔS values shows a non-linear behavior. It was found that ΔS increases with deformation until about 6.6% when the ΔS begins to decrease.

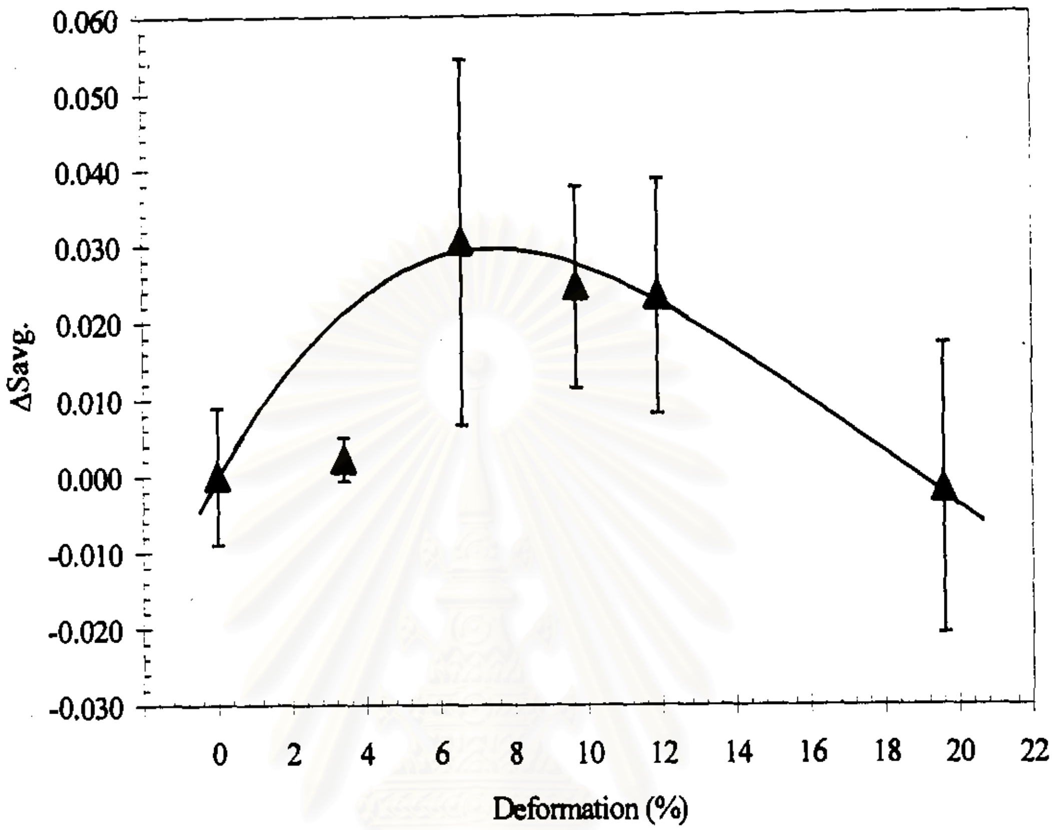

Figure 5.3 The change in S value of deformed specimens as compare to an undeformed specimens.

According to MacKenzie [1], the S parameter was found to increase with deformation which is not in agreement with our experiments. The plot of ΔS in copper as a function of deformation from MacKenzie and our experiments are shown in Figure 5.4. In order to explain the observed behavior, we must investigate into the evolution of dislocations with strain.

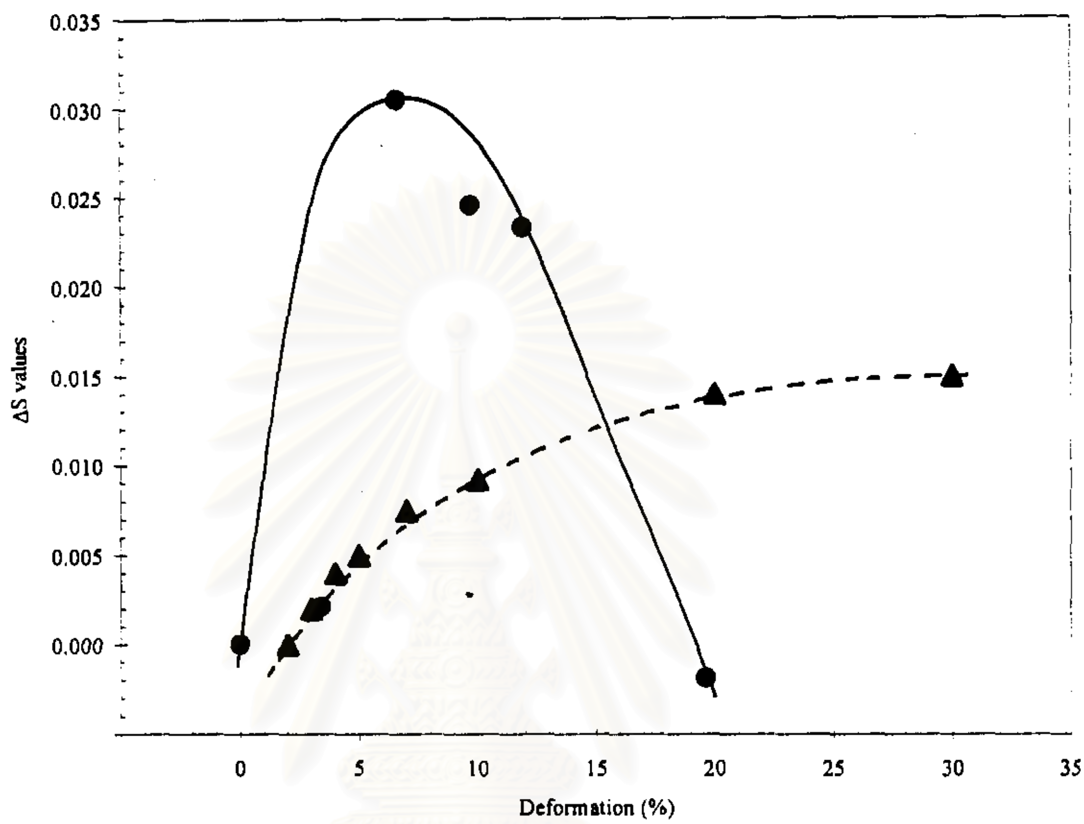

Figure 5.4 The change of ΔS values obtained from MacKenzie (▲) and our experiment (●).

The evolution of dislocation, ρ, with strain, ε, can be written as [34]

$$\frac{d\rho}{d\varepsilon} = \frac{1}{\Lambda b} - L_r N_r \frac{v_r}{\varepsilon^{\circ}} \qquad 5.1$$

where Λ is the mean free path of dislocations, b is Burgers vector, L_r is dislocation segments of length, N_r is number of dislocations per unit volume, v_r is the rearrange rate and ε° is the strain rate. The first term of this equation means the dislocation storage rate by mean free path; the latter indicates a dynamic recovery rate which is strongly influenced by the strain rate. It is thus possible that the use of slow strain rate (10^{-6}) in our experiment significantly increases the annihilation term and decreases the slope ($d\rho/d\varepsilon$) as the dislocation density increases to a certain level. In the case of MacKenzie experiment, copper specimens were deformed by cold rolling which is presumably 5-6 order of magnitude faster than our strain rate and thus the annihilation term is significantly smaller. Investigation of the evolution of dislocation with strain in copper at room temperature (293 K) by Mecking and Kocks [35] also showed that the rate increases to a certain stress level and decreases afterwards as shown in Figure 5.5. Interestingly, the change of evolution of dislocation is similar to the change of ΔS in our experiments.

Another supporting evidence for our experiments can be seen from the experiment on fatigue of SA508 alloy steels. Figure 5.6 was shown the change in S parameter in SA 508 low alloy steel as a function of fatigue life. The S parameter increases up to 1% of the fatigue life, and then decreases with increasing fatigued deformation. It was found that the reduction of S parameters beyond 10% of the fatigue life could be rationalized in term of changes in the vacancy concentration and in the dislocation density in the cell which decreased in the later stages of fatigue during fatigue process.

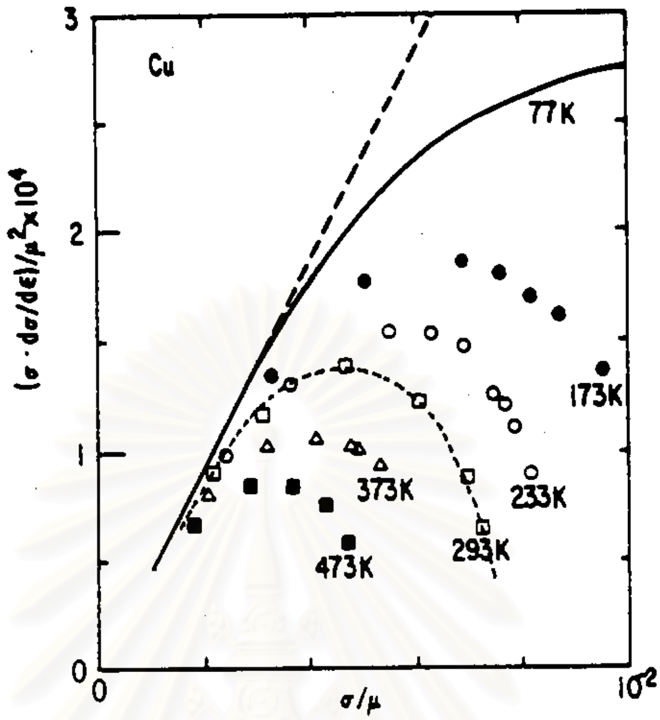

Figure 5.5 The evolution rate of the dislocation density

as a function of stress in copper.

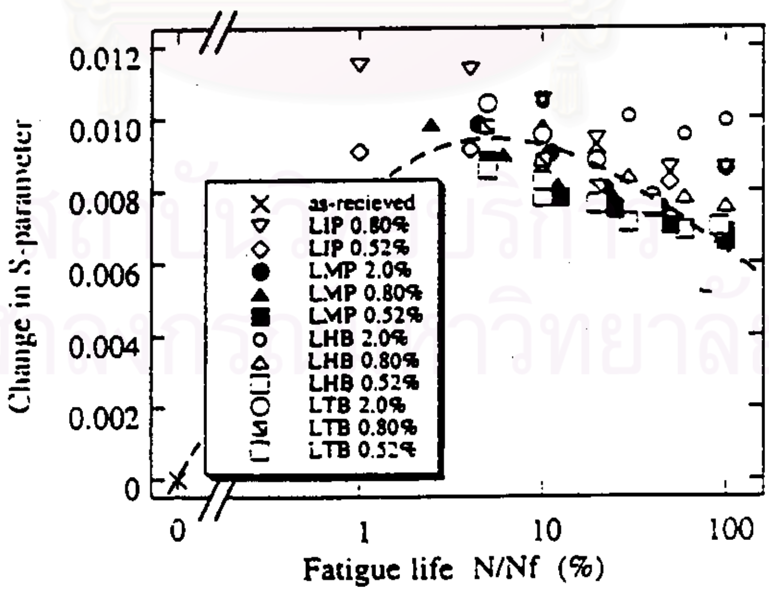

Figure 5.6 The change in S parameter in SA 508 low alloy steels.

as a function of fatigue life.

Results from experiments on dislocation density measurement of copper provide us with the confidence that the developed DBPA system is sensitive to the change in microstructures and may be satisfactorily used as a non-destructive tool yielding information on the state of microstructures.

5.2.2 Evaluation of the sensitization in stainless steels specimens

The use of DBPA system was applied to evaluate microstructures of stainless steels. The measurement of stainless steels at various sensitization time were repeated 5 times. The S values with sensitization were shown in Table 5.3.

Table 5.3 The S value of stainless steels specimens as a function of sensitization with standard deviation (σ).

Sensitization	Run No.1	Run No.2	Run No.3	Run No.4	Run No.5	$S_{avg.}$	σ
0	0.50872	0.54048	0.50313	0.49055	0.47168	0.50291	0.01134
2	0.51378	0.47902	0.51497	0.55534	0.51433	0.51549	0.01209
8	0.53087	0.57820	0.54784	0.52873	0.53827	0.54994	0.01075
16	0.50057	0.52610	0.54285	0.53416	0.47387	0.51551	0.01258

The ΔS value with the sensitization were shown in Table 5.4. The correlation between ΔS and sensitization time was shown in Figure 5.7.

Table 5.4 The change of S value as a function of sensitization with standard deviation (σ) calculated by using error propagation.

Sensitization (hr)	$\Delta S_{avg.}$	σ
0	0.00000	0.01604
2	0.01658	0.01654
8	0.04373	0.01562
16	0.01260	0.01694

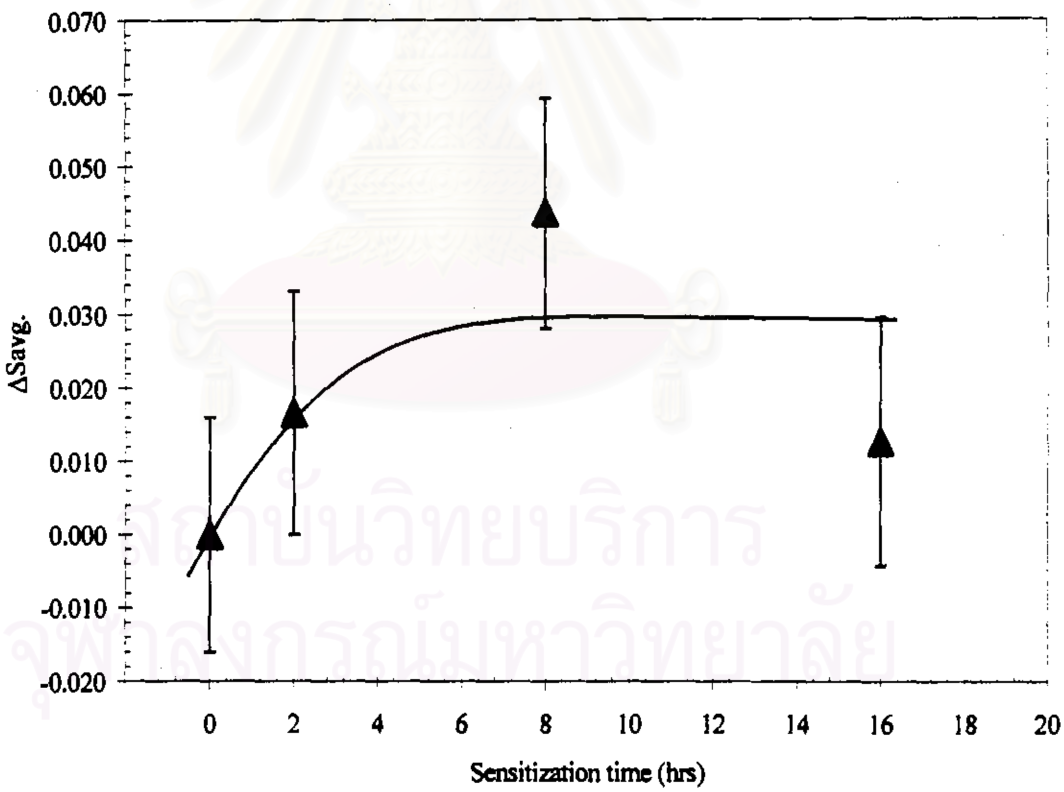

Figure 5.7 The correlation between ΔS and the sensitization time.

Generally, the ΔS value was found to increase as the sensitization time increases although the ΔS at 16 hr seens to slightly decrease after 8 hr. Following our general understanding of the S parameter, the S parameter increases as defect density increases. The increasing in the S parameter of sensitized specimen must be directly related to the precipitation of chromium carbides or chromium depletion at the grain boundaries. Figure 5.8 (a), (b) and (c) show the micrographs of stainless steels after solution annealed, 8 hr and 16 hr sensitization. It is clear that ΔS values increases with chromium carbides at grain boundaries. By mapping the grain boundary coverage and thickness of carbides, it was found that there is a correlation between the density of carbides and ΔS parameters, Figure5.9.

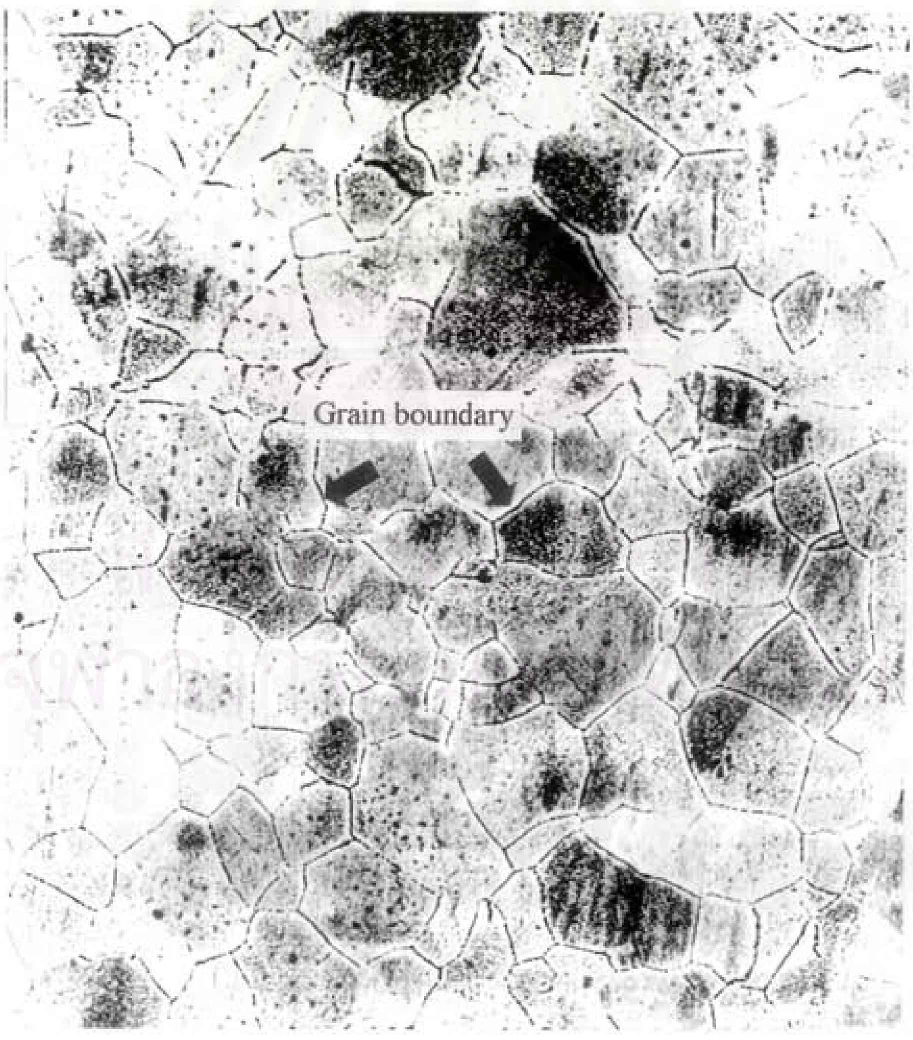

Figure 5.8 (a) Illustration of the micrograph of solution annealed stainless steels.

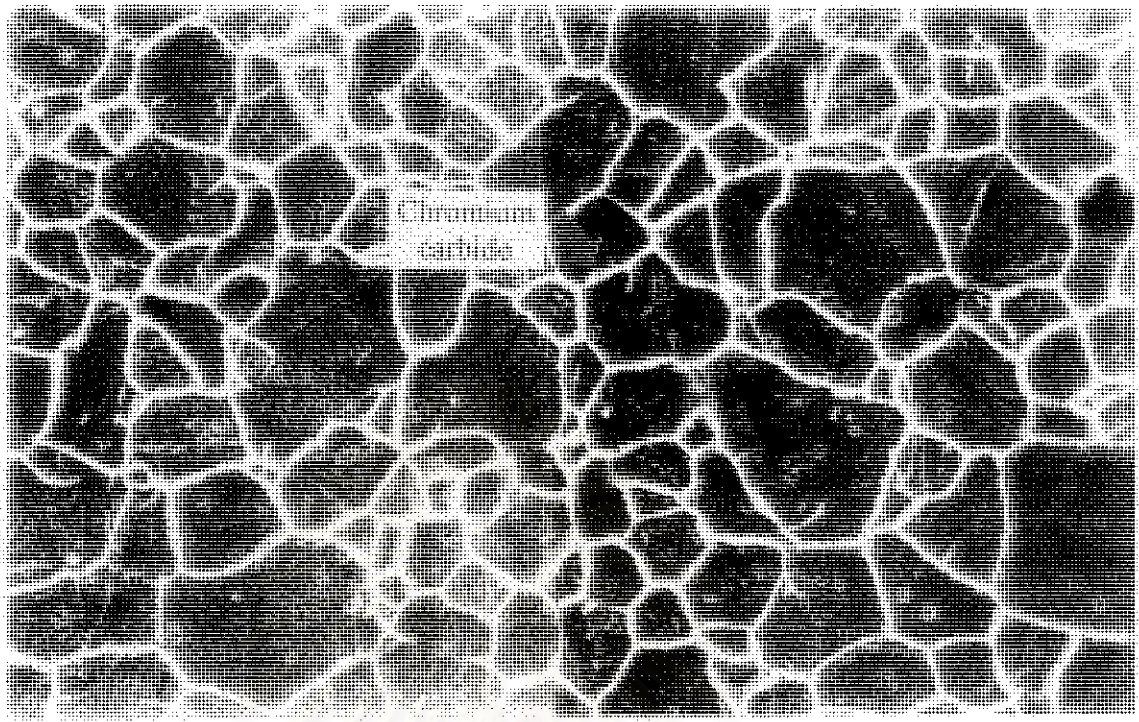

Figure 5.8 (b) Illustration of the micrograph of 8 hr sensitized stainless steels specimens.

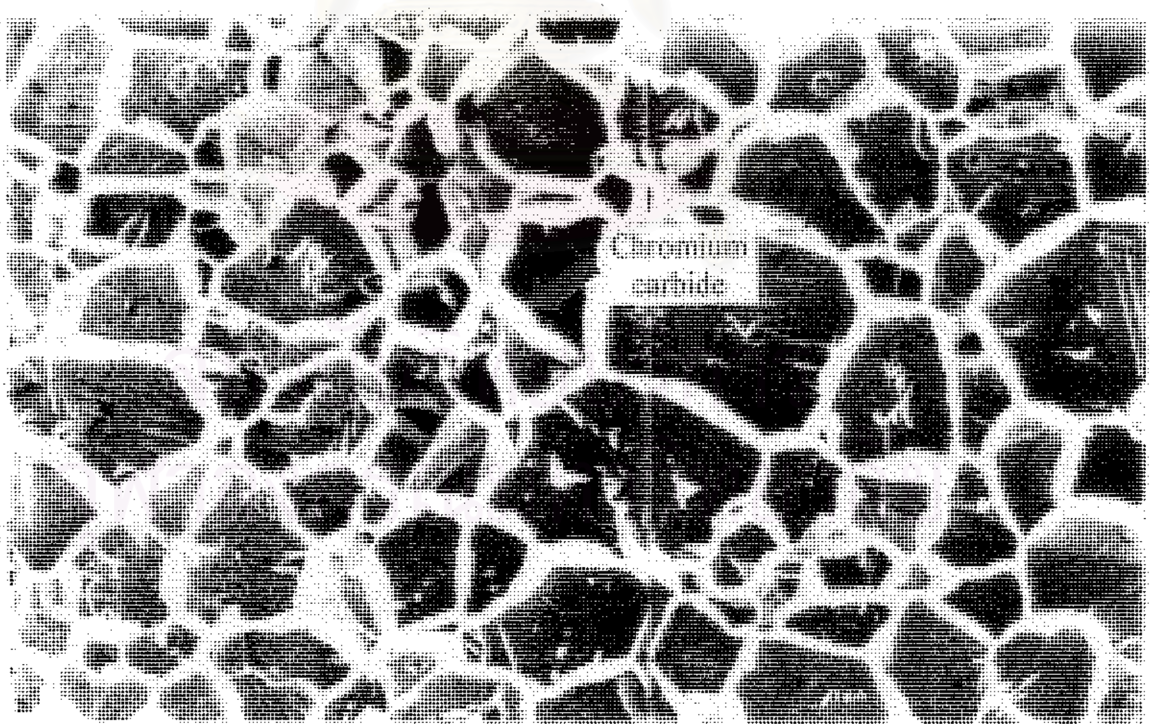

Figure 5.8 (c) Illustration of the micrograph of 16 hr sensitized stainless steels specimens.

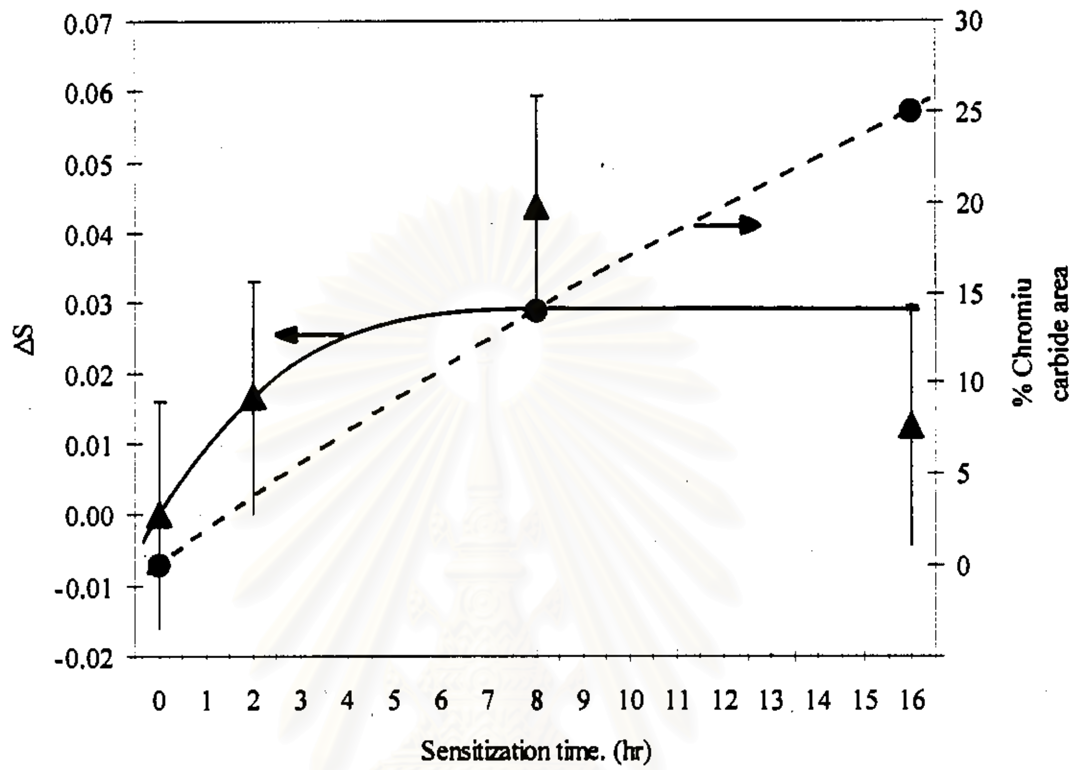

Figure 5.9 Illustration of the correlation of ΔS value (▲) and percent chromium carbide area (●) at the grain boundaries.

However, further experiments are needed to substantiate the results. It was propose of this experiment to compare the S parameter of sensitized specimens with the degree of embrittlement in order to draw a correlation between S parameters and cracking properties of stainless steels. Thus, the change in the ΔS value as a function of sensitization was plotted together with the ultimate tensile strength (UTS) tested in $Na_2S_2O_3$ solution at an initial stain rate of 10^{-6} [36], Figure 5.10.

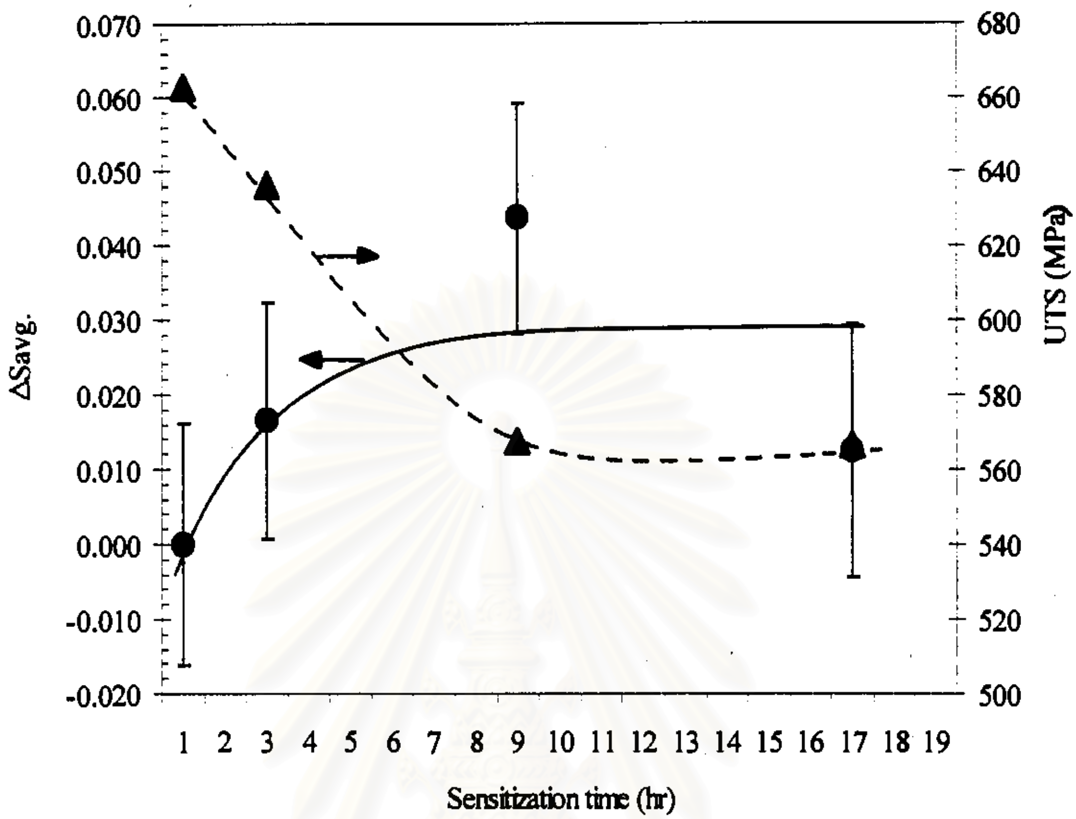

Figure 5.10 The correlation of ΔS (•) value and the

ultimate tensile strength (▲)sensitization time.

It is clear that the ΔS values increase with increasing sensitization while the UTS decreases. The ΔS parameter was then found to be inversely proportional to the degree of embrittlement. This indicates that there is an inverse correlation between the change in S parameter and the susceptibility to stress corrosion cracking (SCC) of stainless steels. Although the statistics of our S parameter still need to be improved, it was clear that the DBPA technique may be non-destructively used to evaluate the IGSCC susceptibility of stainless steels.

5.3 Conclusion

The performance of developed DBPA technique was test to evaluate the dislocation density in copper specimens. It was found that this technique can be used for evaluating the states of microstructures. The technique when applied to evaluate the sensitization in stainless steels was found to be satisfactorily in providing a general trend to predict the degree of sensitization and thus stress corrosion cracking in stainless steels. The statistics of S parameter however still need to be improved. It may be concluded that this technique may be used non-destructively to evaluate the microstructures in material.