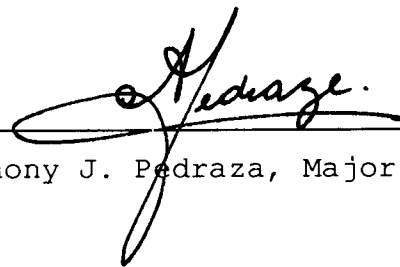


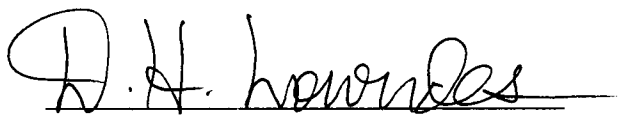
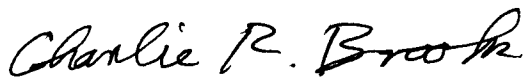
To the Graduate Council: .

I am submitting herewith a thesis written by Jaewon Park entitled " Residual Stresses in Stainless Steel Produced by Cold Rolling and Laser Processing ". I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Metallurgical Engineering.



Anthony J. Pedraza, Major Professor

We have read this thesis  
and recommend its acceptance:



Accepted for the Council:



Associate Vice Chancellor  
and Dean of the Graduate School

**RESIDUAL STRESSES IN STAINLESS STEEL**  
**PRODUCED BY**  
**COLD ROLLING AND LASER PROCESSING**

A Thesis  
Presented for the  
Master of Science  
Degree  
The University of Tennessee, Knoxville

JAEWON PARK

August, 1992

## **DEDICATION**

TO MY PARENTS, MY WIFE AND MY DAUGHTER

## **ACKNOWLEDGMENTS**

For his guidance, support and encouragement, I am indebted to my major professor, Dr. A.J. Pedraza.

Gratitude is also extended to the other committee members, Dr. D.H. Lowndes and Dr. C.R. Brooks for being on my thesis committee, and to Dr. M.G. Godbole and Mr. M.G.Desilva for their help on sample preparation of laser processing.

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## ABSTRACT

Stainless steel specimens were analyzed by X-ray diffraction (XRD) in the as-received conditions and cold rolled condition and after excimer laser treatments.

The  $\sin^2\psi$  method was employed to measure the macro-residual stresses. The contribution to line broadening from mosaic structure and defects present in the stainless steel specimens were obtained using the Fourier analysis method developed by Warren and Averbach.

Stainless steel films were sputter-deposited on stainless steel substrates. Diffraction patterns showed that when the passive oxides layer present in the substrate was not removed the film had a body-centered cubic crystal structure. If the oxides layer is removed, then the films grow from the substrate, and have a face-centered cubic structure (FCC). Thermodynamic calculations suggest that the stable phase in austenitic stainless steel should be BCC rather than the usual FCC. It is reasonable to assume that if no oxide layer is present the films should grow in a homo-epitaxial manner reproducing the FCC crystal structure. If an oxide layer is present, then nucleation of the stable BCC can take place as observed.

Tensile residual stresses were produced in the surface of 20% cold rolled specimen. In the 40% cold rolled and 60%

cold rolled specimens the residual stresses were compressive in nature. In the rolling operation the metal sheet is subjected to the compressive stress by squeezing action of the rolls and the shear stress in the surface due to frictional force between rolls and metal sheet. That is, the surface is deformed more than the center, producing the surface compressive residual stresses. However, the tensile residual stress produced in 20% cold rolled specimen indicates that the center was elongated more than the surface.

Stainless steel specimens in the annealed and with 20%, 40% and 60% cold rolling were irradiated with a XeCl excimer laser at various energy densities.

The X-ray lines of annealed specimens were broadened after laser irradiation. On the other hand the line broadening of the X-ray lines for all the cold rolled specimens was reduced. The laser processing in metals produced two effect; first, annealing owing to the heating process, and second, plastic deformation due to the extremely steep thermal gradient.

Fourier analysis of the line broadening in annealed specimens showed that at lower energy densities the broadening was mostly due to the generation of single dislocations (lattice strain broadening) and for higher energy density the mosaic structure (size effect broadening) was the major contribution to the line broadening of annealed specimen. The mosaic boundaries, also called cell boundaries,

originate from dislocation aggregation and interactions after laser irradiation.

In the cold rolled specimens the size effect on the line broadening decreased after laser irradiation. The single dislocation density did not change during the laser processing of the cold rolled specimens.

Residual macro-stresses are always tensile, independently of the initial state of the specimen (viz. annealed or cold rolled). Moreover, the magnitude of the stresses measured is almost the same for annealed or cold rolled specimens irradiated with an energy density of  $4\text{J/cm}^2$ .

The line width of silicon single crystals did not change after laser processing. This result indicates that very little or no plastic deformation takes place during laser processing of the silicon. In agreement with that result no macro residual stresses were detected in the irradiated silicon.

## TABLE OF CONTENTS

| CHAPTER                                                                       | PAGE |
|-------------------------------------------------------------------------------|------|
| 1. Introduction -----                                                         | 1    |
| 2. Literature review -----                                                    | 5    |
| 2.1 Basic concepts in X-ray diffraction-----                                  | 5    |
| 2.1.1 Scattering of X-rays -----                                              | 5    |
| 2.1.2 Absorption of X-rays -----                                              | 6    |
| 2.1.3 The structure factor of a unit cell -----                               | 7    |
| 2.1.4 Intensity of diffracted lines for<br>polycrystalline materials -----    | 9    |
| 2.1.5 X-ray penetration depth -----                                           | 12   |
| 2.2 Fundamental concepts of stress and strain<br>fields -----                 | 13   |
| 2.2.1 Description of stresses -----                                           | 13   |
| 2.2.2 Transformation of axes and tensor<br>notation -----                     | 14   |
| 2.2.3 Elastic stress - strain relationship in an<br>isotropic materials ----- | 15   |
| 2.2.4 Anisotropy of elasticity behavior -----                                 | 17   |
| 2.3 Residual stress analysis using X-ray -<br>diffraction method -----        | 19   |
| 2.3.1 Concept of the residual stress -----                                    | 19   |
| 2.3.2 The measurement of the residual<br>stress -----                         | 21   |

|                                                   |    |
|---------------------------------------------------|----|
| 2.3.2.1 Destructive method -----                  | 21 |
| 2.3.2.2 Nondestructive methods -----              | 21 |
| 2.3.3 Macro-stress analysis using X-ray           |    |
| diffraction method -----                          | 22 |
| 2.3.3.1 Basic concepts -----                      | 22 |
| 2.3.3.2 Basic equations and calculation           |    |
| procedure to determine stresses from              |    |
| X-ray data -----                                  | 24 |
| 2.3.3.3 Interpretation of stresses from           |    |
| oscillatory data -----                            | 30 |
| 2.3.4 Diffractometric and analytic techniques for |    |
| measuring Macro residual stress -----             | 33 |
| 2.3.4.1 Alignment of diffractometer -----         | 33 |
| 2.3.4.2 Determination of peak position ---        | 34 |
| 2.3.4.3 The Rachinger correction -----            | 35 |
| 2.3.5 Effect of small coherent domain and strains |    |
| -- Line broadening analysis -----                 | 36 |
| 2.3.5.1 The size and strain effects on line       |    |
| broadening -----                                  | 36 |
| 2.3.5.2 Separation of the pure broadening         |    |
| from the total X-ray peak -----                   | 38 |
| 2.3.5.3 Separation of crystal size and            |    |
| lattice strains from pure line                    |    |
| broadening -----                                  | 42 |
| 2.3.6 Determination of dislocation density ----   | 50 |

|                                                                                |           |
|--------------------------------------------------------------------------------|-----------|
| 2.3.7 Centroid and variance method for the line<br>broadening -----            | 50        |
| 2.4 Fundamentals of cold rolling -----                                         | 51        |
| 2.5 Laser processing -----                                                     | 53        |
| 2.5.1 Fundamentals of laser -----                                              | 53        |
| 2.5.2 Excimer laser -----                                                      | 54        |
| 2.5.3 Thermal analysis of the process -----                                    | 57        |
| 2.6 Phase transformation in austenitic<br>stainless steel -----                | 58        |
| 2.6.1 Phase transformation in pure iron -----                                  | 58        |
| 2.6.2 Chromium addition effect -----                                           | 58        |
| 2.6.3 Nickel addition effect -----                                             | 59        |
| 2.6.4 The Phase of the austenitic stainless steel<br>at room temperature ----- | 60        |
| <b>3. Specimen preparation and experimental<br/>procedure -----</b>            | <b>62</b> |
| 3.1 Specimen preparation -----                                                 | 62        |
| 3.1.1 For residual stress analysis -----                                       | 62        |
| 3.1.2 For melting depth measurement -----                                      | 63        |
| 3.1.3 For dislocation observation -----                                        | 64        |
| 3.1.4 For phase formation study of the<br>austenitic stainless steel -----     | 64        |
| 3.2 X-ray diffraction condition -----                                          | 65        |
| 3.2.1 Diffractometer geometry and operation<br>principles -----                | 65        |
| 3.2.2 Diffraction condition -----                                              | 68        |

|                                                                                                    |           |
|----------------------------------------------------------------------------------------------------|-----------|
| 3.3 Analytical procedure for residual stresses ----                                                | 70        |
| 3.3.1 Background subtraction from intensity<br>data -----                                          | 70        |
| 3.3.2 Analytical procedures for macro-residual<br>stress -----                                     | 70        |
| 3.3.2.1 Least squares parabolic fitting -                                                          | 70        |
| 3.3.2.2 Residual stress calculation -----                                                          | 71        |
| 3.3.3 Analytical procedure for micro-residual<br>stress -----                                      | 73        |
| 3.3.3.1 Line broadening determination ---                                                          | 73        |
| 3.3.3.2 Separation of the pure line<br>broadening -- the method of<br>Stoke's correction -----     | 73        |
| 3.3.3.3 Separation of the particle size<br>and the micro-strain using pure<br>broadened peak ----- | 77        |
| 3.4 Calculation of melting depth and laser affected<br>zone -----                                  | 82        |
| <b>4. Results -----</b>                                                                            | <b>85</b> |
| 4.1 Calibration method -----                                                                       | 85        |
| 4.2 Macro-residual stress analysis -----                                                           | 85        |
| 4.2.1 Peak scans -----                                                                             | 85        |
| 4.2.2 Parabola fitting curves -----                                                                | 85        |
| 4.2.3 d versus $\sin^2\psi$ plot -----                                                             | 91        |
| 4.3 Micro-residual stress analysis -----                                                           | 96        |
| 4.3.1 Peak scans -----                                                                             | 96        |

|                                                                                                           |            |
|-----------------------------------------------------------------------------------------------------------|------------|
| 4.3.2 Line broadening measurement -----                                                                   | 101        |
| 4.3.3 Separation of the crystallite size and RMS<br>strain -----                                          | 101        |
| 4.3.4 Dislocation density analysis -----                                                                  | 115        |
| 4.4 Microstructural view -----                                                                            | 118        |
| 4.4.1 Melting depth profile -----                                                                         | 118        |
| 4.4.2 Dislocation observation -----                                                                       | 122        |
| 4.5 Result of the computer simulation for the<br>melting depth and thermal gradient -----                 | 122        |
| 4.6 Crystal structure of stainless steel deposited on<br>austenitic stainless steel -----                 | 129        |
| <b>5. Discussion -----</b>                                                                                | <b>132</b> |
| 5.1 Macro-stress analysis -----                                                                           | 132        |
| 5.1.1 Residual stress during cold rolling<br>operation -----                                              | 132        |
| 5.1.2 The effects of laser irradiation on the<br>surface normal component of the residual<br>stress ----- | 132        |
| 5.1.3 Residual stresses during laser treatment-----                                                       | 137        |
| 5.2 Line broadening analysis -----                                                                        | 143        |
| 5.2.1 Variance and dislocation density -----                                                              | 143        |
| 5.2.2 Cell structure effect -----                                                                         | 145        |
| 5.2.3 Prior cold rolling effect on dislocation<br>density change during laser processing -                | 146        |
| 5.2.4 Dislocations from the TEM -----                                                                     | 146        |

|                                                                                                             |     |
|-------------------------------------------------------------------------------------------------------------|-----|
| 5.3 The crystal structure of stainless steel thin<br>films deposited on austenitic stainless<br>steel ----- | 147 |
| <b>6. Conclusions</b> -----                                                                                 | 147 |
| <b>BIBIOGRAPHY</b> -----                                                                                    | 151 |
| <b>VITA</b> -----                                                                                           | 158 |

## LIST OF TABLES

| TABLE                                                                                                                                     | PAGE |
|-------------------------------------------------------------------------------------------------------------------------------------------|------|
| 3.1 Operation conditions used in X-ray diffraction -----                                                                                  | 69   |
| 4.1 Macro-residual stresses determined using $\sin^2\psi$<br>method -----                                                                 | 95   |
| 4.2 Variance comparison before and after laser treatment<br>treatment -----                                                               | 102  |
| 4.3 Effective crystallitic sizes and RMS strains for the<br>specimens laser-treated on annealed substrates ---                            | 111  |
| 4.4 Effective crystallite sizes and RMS strains for the<br>specimens cold-rolled and laser-treated on the cold-<br>rolled specimens ----- | 112  |
| 4.5 Dislocation density (from RMS strain and small crystal<br>size) variation before and after laser treatment --                         | 116  |
| 4.6 Average dislocation density variation before and<br>after laser treatment -----                                                       | 119  |

## LIST OF FIGURES

| FIGURE |                                                                                                                                                                                                     | PAGE |
|--------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|
| 2.1    | a) Variation of intensity with diffraction angle for perfect crystals b) Line broadening caused by the instrument and imperfections in the crystal structure.-----                                  | 9    |
| 2.2    | The geometry of the flat specimen and X-ray beam to calculate the absorption factor in a diffractometer. -----                                                                                      | 12   |
| 2.3    | a) Two arbitrary unit cubes A and b in a uniformly stressed body, b) The new and old coordinate systems $L(x_i')$ and $S(x_i)$ and the definition of the direction cosines between them.-----       | 16   |
| 2.4    | The near surface is longer than the interior causing the tensile residual stress on the surface. -----                                                                                              | 20   |
| 2.5    | Schematic description of the neutron scattering method to measure the residual stress. -----                                                                                                        | 23   |
| 2.6    | Stress-strain phenomena and back-reflection of x-rays in a surface crystal of a metal bar. -----                                                                                                    | 24   |
| 2.7    | Definition of the laboratory coordinate system $L_i$ , sample coordinate system $S_i$ and the angles $\phi$ and $\psi$ ---                                                                          | 26   |
| 2.8    | Types of "d" vs. $\sin^2\psi$ plots. Plots (a) and (b) denote regular behavior, The plot(c) exhibits oscillatory behavior implying the presence of inhomogeneous stress and strain gradients. ----- | 29   |

|      |                                                                                                                                                                                                                                                               |    |
|------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| 2.9  | The range of incident x-rays is the angle $d\alpha$ .<br>The angles $d\beta$ and $d\gamma$ define the position of a<br>diffracted beam on the detecting plane. The<br>three angles $d\alpha, d\beta, d\gamma$ define a volume in reciprocal<br>lattice. ----- | 45 |
| 2.10 | Forces acting during rolling. -----                                                                                                                                                                                                                           | 52 |
| 2.11 | Potential-energy curves for the KrF excimer laser. -                                                                                                                                                                                                          | 55 |
| 2.12 | Partial iron chromium phase diagram. -----                                                                                                                                                                                                                    | 59 |
| 2.13 | Effect of nickel content on the stability of<br>austenite in an iron alloy containing 18%<br>chromium. -----                                                                                                                                                  | 60 |
| 2.14 | Compositions of iron-chromium-nickel alloys for<br>which austenite persists in room temperature. Point<br>P indicates the position of an alloy containing 18%<br>chromium and 8% nickel.-----                                                                 | 61 |
| 3.1  | The geometry of the diffractometer with monochromator-                                                                                                                                                                                                        | 67 |
| 3.2  | An example of the least squares parabolic fitting<br>method for top 15% of max. intensity; a) a view from<br>a full scan, b) a view from top 15% intensity data.--                                                                                            | 72 |
| 3.3  | Examples of the Rachinger correction.-----                                                                                                                                                                                                                    | 74 |
| 3.4  | Examples of intensity peak profiles reconstructed<br>using Fourier coefficients. -----                                                                                                                                                                        | 76 |
| 3.5  | Comparison of experimental peak with instrumental peak,<br>reconstructed by Fourier coefficient. -----                                                                                                                                                        | 78 |
| 3.6  | Examples of the pure broadened line profile<br>constructed by corrected Fourier coefficient after                                                                                                                                                             |    |

|                                                                                                                                                                                               |    |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| stoke's corrections.-----                                                                                                                                                                     | 79 |
| 3.7 An example of logarithmic plot to separate the<br>crystallite size and RMS strain using Fourier cosine<br>coefficient. From the slope the RMS strain is<br>obtained. -----                | 81 |
| 3.8 Plots of cosine size coefficient( $AL^3$ ) vs. real column<br>length(L) (examples of the plot to get the effective<br>crystallite size). -----                                            | 84 |
| 4.1 Peak scans of 420 reflection at various $\psi$ angles for<br>a fully annealed specimen after calibration. -----                                                                           | 86 |
| 4.2 Least squares parabola fitting curves for the peaks<br>from Fig.4.1.-----                                                                                                                 | 86 |
| 4.3 Peak scans of 420 reflections for the<br>specimens used in the experiment.-----                                                                                                           | 87 |
| 4.4 Least squares parabola fitting curves for the peak<br>scans shown in the Fig.4.3. -----                                                                                                   | 89 |
| 4.5 Plots of d vs. $\sin^2\psi$ for annealed and cold rolled<br>specimens. -----                                                                                                              | 92 |
| 4.6 Plots of d vs. $\sin^2\psi$ for annealed and cold rolled<br>specimens after laser irradiation with $4J/cm^2$ energy<br>density(above;interpolation, below;least square<br>fitting). ----- | 93 |
| 4.7 Plots of d vs. $\sin^2\psi$ for annealed specimens after<br>laser treatment with various energy densities. -----                                                                          | 94 |
| 4.8 Plot of the residual stress versus laser energy<br>density for annealed specimen. -----                                                                                                   | 97 |

|      |                                                                                                                                                                                            |     |
|------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| 4.9  | Plot of the residual stress versus cold rolling rate<br>before and after laser treatment.-----                                                                                             | 98  |
| 4.10 | Intensity peak profiles of 111 and 222<br>reflections used in determining the micro-residual<br>stresses. -----                                                                            | 99  |
| 4.11 | Plot of variance vs. cold rolling -----                                                                                                                                                    | 103 |
| 4.12 | Variance change vs.laser energy density for the<br>annealed substrate after laser irradiation with<br>various energy densities -----                                                       | 104 |
| 4.13 | Plot of the variance change vs. percent of cold work<br>for cold rolled specimens after laser irradiation at<br>4J/cm <sup>2</sup> energy density. -----                                   | 104 |
| 4.14 | Plot of the variance change vs. different energy<br>densities for 60% cold rolled specimen -----                                                                                           | 105 |
| 4.15 | Pure broadened K $\alpha$ 1 line profiles after<br>Stoke's correction.(The integrated intensity should<br>be consistent with the variance values determined<br>from the total peaks.)----- | 107 |
| 4.16 | Plots used to separate the crystallite size and the<br>lattice strain.-----                                                                                                                | 109 |
| 4.17 | Plot of the effective crystallite size versus laser<br>energy density for the annealed specimen -----                                                                                      | 113 |
| 4.18 | Plot of the RMS strain versus laser energy density<br>for the annealed specimen -----                                                                                                      | 113 |
| 4.19 | Variation of the RMS strain versus cold rolling rate<br>before and after laser irradiation -----                                                                                           | 114 |

|       |                                                                                                                                                                                             |     |
|-------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| 4.20  | Variation of the effective crystallite size versus cold rolling rate before and after laser irradiation ----                                                                                | 114 |
| 4.21  | Plot of the $D_s/D_d$ versus laser energy density<br>( $D_s$ ; the dislocation density from the crystallite size, $D_d$ ; the dislocation density from the lattice strain). -----           | 117 |
| 4.22  | Plot of the dislocation density ratio (between dislocation densities before and after laser treatment) versus cold rolling rate in terms of RMS strain and effective crystallite size.----- | 117 |
| 4.23  | Plot of the dislocation density versus cold rolling rate before and after laser irradiation with $4\text{J}/\text{cm}^2$ energy density.-----                                               | 120 |
| 4.24  | Plot of dislocation density change versus cold rolling rate due to laser treatment with $4\text{J}/\text{cm}^2$ energy density. -----                                                       | 120 |
| 4.25  | Plot of dislocation density versus laser energy density for 60% cold rolled specimen laser irradiated. -----                                                                                | 121 |
| 4.26  | Plot of the the dislocation density vs. laser energy density for the annealed substrate. -----                                                                                              | 121 |
| 4.27. | (a), (b) Cross sectional view of the solidified surface layer during laser treatment with $4\text{J}/\text{cm}^2$ energy density from SEM. -----                                            | 123 |
| 4.28  | Dislocations from the as-received specimen from TEM.-----                                                                                                                                   | 125 |

|      |                                                                                                                                          |     |
|------|------------------------------------------------------------------------------------------------------------------------------------------|-----|
| 4.29 | Dislocations in the laser affected zone from TEM<br>after laser irradiation with 4J/cm <sup>2</sup> energy density                       | 126 |
| 4.30 | Plot of the melted depth and vaporized depth versus<br>laser energy density. -----                                                       | 127 |
| 4.31 | Plot of the temperature versus laser affected zone<br>depth obtained from the computer simulation. -----                                 | 128 |
| 4.32 | Diffraction pattern for an annealed substrate for<br>the sputter deposition. -----                                                       | 130 |
| 4.33 | Diffraction pattern for a deposited film on a<br>annealed substrate. -----                                                               | 130 |
| 4.34 | Diffraction pattern for a ground substrate after<br>annealing. -----                                                                     | 131 |
| 4.35 | Diffraction pattern for a deposited film on a ground<br>substrate. -----                                                                 | 131 |
| 5.1  | Plot of the fraction of the total integrated<br>intensity(Gx) versus X-ray penetration depth for 316L<br>stainless steel. -----          | 135 |
| 5.2  | X-ray diffracting area for 420 reflection. -----                                                                                         | 136 |
| 5.3  | Schematic explanation of the process to induce the<br>residual stress during laser processing. -----                                     | 140 |
| 5.4  | Schematic explanation of the stress deviations due to<br>the different conditions of non-affected zone in the<br>X-ray measurement.----- | 142 |

# **CHAPTER 1**

## **INTRODUCTION**

Residual stresses are the self-equilibrating internal stresses existing in a free body when no external forces or moments are applied. These stresses arise as a consequence of temperature gradients or mechanical loadings causing non-uniform plastic deformations.

The residual stress can be measured by mechanical and by non-destructive methods. The mechanical methods involve removing part of the stressed material. Thus they are relatively expensive and restricted to parts with special geometric forms. Among several non-destructive methods in measuring the residual stress, the X-ray diffraction method is mainly used [1]- [4]. However, this method can measure the residual stress only near the surface because of its shallow penetration depth.

The residual stress is developed in each metal working process. In a rolling operation, plastic flow occurs near the surface of the metal sheet. The surface grains in the sheet tend to elongate, while the grains in the center of the sheet are relatively unaffected. Consequently, it is expected that a residual stress pattern in the rolled sheet will consist of a high compressive stress at the surface and a tensile residual stress at the center of the sheet [5]. However, since the amount of the residual stress and the stress state

in the material are a function of the roll diameter, the specimen thickness and the degree of reduction, they can be determined by actual measurement.

An excimer laser can deliver an intense and localized energy to the work piece. It can result in energy densities more than  $10\text{J}/\text{cm}^2$  by controlling an appropriate system of lenses. The pulse duration of the excimer laser is in the range of tens of nanoseconds and the power density can reach hundreds of megawatts. The energy of a nearly coherent laser beam incident on the material is partly absorbed by the electron photon interactions and this energy is transferred as heat to the lattice in a very short time ( $10^{-11}$ – $10^{-12}\text{s}$ ). Thus, the laser can be used as a surface heating source. It is being applied to the semiconductor industry in terms of the fabrication of premium solar cells[6], doping[7], etching [8], photolithography[9] and laser-assisted chemical vapor deposition[10]. The laser is also being used as an important tool for deposition of high  $T_c$  superconductor film [11].

Laser beam processing can result in the non-uniform plastic deformation near the surface of the work piece due to steep thermal gradient and rapid solidification. The residual stress can thus be formed on the laser irradiated surface.

Since plastic deformation occurs near the surface due to the cold rolling and laser irradiation, X-ray diffractometry is an appropriate tool to measure the residual stress for this type of processing. In this research, well annealed and

cold rolled specimens are used for laser processing. In each process, the residual stresses are measured and compared with each other. Dislocation density can be calculated from line broadening. TEM work is being performed to check the validity of the calculation qualitatively. Since the X-ray methods measure residual stresses near the surface, the comparison between 'X-ray penetration depth' and 'laser melting plus affected zone depth' will be very important to insure that the measurement covers the sufficient depth. SEM work is being done to check this point in this experiment and the result will be compared with the computer simulated result.

The X-ray method for the measurement of macrostress is based on the line shift, while the microstress due to bent or distorted grains is determined by line broadening. Analysis of the residual stress by the X-ray method requires complex computation; therefore most past studies were primarily empirical or limited to analyses of simple cases. With the advancement of modern computers and techniques such as the finite element method, effort has been recently renewed to study these stresses

The phase formation study of the austenitic stainless steel is another experiment that is conducted in this research.

The stable structure of the austenitic stainless steel at room temperature is BCC. However, due to the sluggish transformation, the FCC austenite is persistent even at room

temperature. If the atoms of the 18Cr-8Ni stainless steel are stacked in the free constraining condition, we will get the BCC structure that is thermodynamically stable at room temperature. The sputter deposition has been known as a good process to get the atomic level stacking [12]. In this research, these phenomena are being investigated by comparing the X-ray diffraction patterns before and after sputter deposition for various conditions of the substrates.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Basic concepts in X-ray diffraction

##### 2.1.1 Scattering of X-rays

The photons of an X-rays beam impinging on a specimen strongly interact with the electrons of all the materials. If the electron in an inner orbitals is very tightly bound to the nucleus and the collision is elastic, the scattered photon will then have the same energy and wavelength as before the collision took place (Coherent Scattering). If the electron, on the other hand, collides with a weakly bound electron in the outer orbital of an atom, there will be some momentum transferred from the photon to the scattered electron. Thus, the scattered photon will have a longer wavelength than the incident photon (Compton modified scattering). This type of scattering is incoherent because there is no definite relationship between the incident and scattered wavelength. The total coherent scattered intensity at a position is given by Thomson's equation. In cgs units;

$$I = I_0 \cdot \frac{e^4}{r^2 m^2 c} \left( \frac{1 + \cos^2 2\theta}{2} \right) \quad \text{Eq.2.1.1}$$

Here  $m$  is the electron mass,  $c$  is the speed of light,  $e$  is the electron charge and  $r$  is the length of the position vector. The term  $(1 + \cos^2 2\theta)$  is called the polarization factor.

Due to this term the scattered intensity in the forward( $2\theta=0^\circ$ ) and backward( $2\theta=180^\circ$ ) directions will be stronger than directions at  $90^\circ$  with the incident beam. In general the photons coherently scattered will be out of phase by various amounts and destructive interference will take place, resulting a reduction in the intensity scattered. Compton modified scattering, due to its random wavelength distribution and arbitrary phase relationships, can not take part in constructive or destructive interference and contributes only to the overall background. The term used to describe the total coherent scattering from an atom to a point(electron) is "f", the atomic scattering factor, which is defined as

$$f = \frac{\text{amplitude of wave scattered by an atom}}{\text{amplitude of wave scattered by an electron}} .$$

Since the waves scattered by individual electrons become more and more out of phase as  $\theta$  increases, f decreases with  $\theta$  increases. At a fixed value of  $\theta$ , however, f will be smaller the shorter the wavelength, since the path differences will be larger relative to the wavelength, resulting in greater interference between the scattered beams. In the actual calculation, f decreases as the quantity  $(\sin\theta)/\lambda$  increases.

### 2.1.2 Absorption of X-rays

X-rays are attenuated as they pass through matter, so that the transmitted beam is weaker than the incident beam.

The contributions to this decrease may include coherent scattering, incoherent scattering, activation of fluorescent X-rays, production of heat or excitation of photo-electrons, etc.. The total loss in intensity due to the sum of all these processes is termed absorption. The total attenuation is related to the thickness  $dz$  of an infinitesimal slab by the relation

$$\frac{dI}{I} = -\mu dz. \quad \text{Eq.2.1.2}$$

$I$  is the transmitted beam intensity, and  $\mu$  is the linear absorption coefficient. This coefficient is proportional to the density( $\rho$ ) of the absorber. The term  $(\mu/\rho)$  is a function of the chemical species and is independent of the state of aggregation(solid, liquid or gas). For homogeneous slab of infinite thickness  $z$  Eq.2.1.2 can be integrated to yield

$$I_z = I_0 \cdot \exp(-\mu/\rho) \rho z \quad \text{Eq.2.1.3}$$

where  $I_0$  is the incident beam intensity. The mass absorption coefficient of a mixture of  $n$  atomic species, each with weight fraction  $w_i$ , can be calculated from

$$(\mu/\rho)_t = \sum_{i=1}^n w_i \cdot (\mu/\rho)_i. \quad \text{Eq.2.1.4}$$

### 2.1.3 The structure factor of a unit cell

The total intensity diffracted by a unit cell is determined from the summation of waves reflected from the

individual atoms. If the scattering amplitude from the  $j$ th atom, with fractional dimensionless coordinates  $u_j, v_j, w_j$ , is  $f_j$ , the resulted wave scattered by all the atoms in the unit cell for a given reflection  $hkl$  is proportional to

$$F_{hkl} = \sum_N^{j=1} f_j \cdot e^{2\pi i (hu_j + kv_j + lw_j)} , \quad \text{Eq.2.1.5}$$

where the summation is over all the atoms present in the unit cell. The term  $F_{hkl}$  is called the structure factor and represents the sum of the amplitude and phase difference of the waves scattered by the individual atoms. The magnitude of the total scattered intensity is proportional to  $|F|^2$ . It can be seen that, when a perfectly monochromatic, parallel beam of X-rays impinges on an infinite single crystal that is perfect throughout its volume, diffraction can occur if and only if:

(a) Bragg's law  $n\lambda = 2d \sin \theta$  is satisfied for one or more sets of crystallographic planes ( $hkl$ ),

(b) The structure factors  $F$  for the  $hkl$  reflections are non-zero for the unit cell of the particular crystal. Under these ideal conditions the variation of diffracted intensity  $I$  with incidence angle  $2\theta$  can be described by a step function. (Fig.2.1.a) However, the ideal conditions are never satisfied. The beams produced by X-ray tubes are polychromatic even if a filtered characteristic line itself has a range of wavelengths (about  $0.001\text{\AA}$ ). Furthermore, the

beam contains parallel, divergent and convergent rays. That will produce an instrumental broadening. The crystal itself is rarely "perfect", but contains mosaic structure and other defects such as dislocations that contributes to the broadening of the diffracted beam.

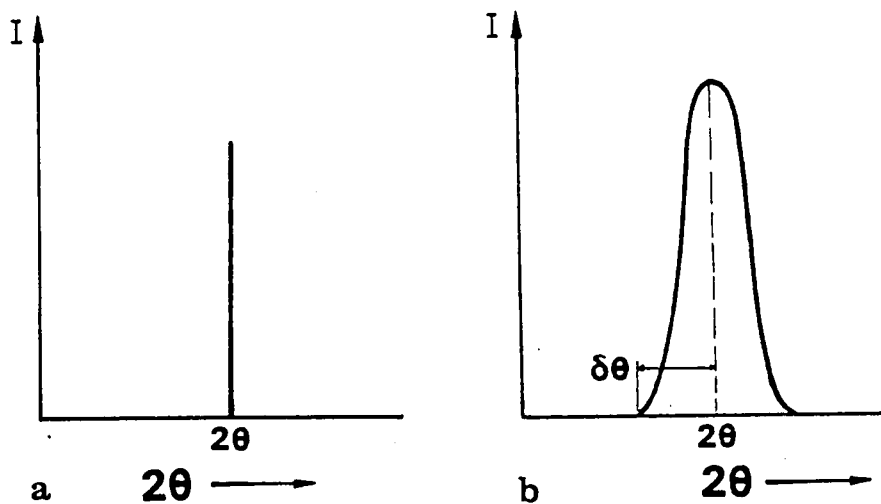


**Fig.2.1.** a) Variation of intensity with diffraction angle for perfect crystals b) Line broadening caused by the instrument and imperfections in the crystal structure

#### 2.1.4 Intensity of diffracted lines for polycrystalline materials

The relative diffracted intensities for each reflection are dependent on the following parameters:

(a) The structure factor was already discussed in the section 2.1.3

(b) The polarization factor was already analyzed in the section 2.1.1

(c) The multiplicity factor that describes the number of equivalent planes that can diffract at a given Bragg angle, i.e., the members of an {hkl} family of planes is a function of the unit cell of the crystal.

(d) The Lorentz term that describes the dependence of the diffracted intensity on the diffraction angle, is given by

$$L \cdot F = K \cdot (1/\sin^2\theta), \quad \text{Eq.2.1.6}$$

where K is constant. The Lorentz factor and the polarization factor are usually combined together to form the L-P (Lorentz-Polarization) factor;

$$(L.P.) = \frac{1+\cos^2 2\theta}{\sin^2 \theta}. \quad \text{Eq.2.1.7}$$

It may be noted that this equation is valid only for an nonpolarized incident beam.

(e) Absorption also affects the diffracted intensities. Absorption is directly related to the path length the incident and diffracted beams traverse in the specimen for a given geometry.

The total diffracted intensity outside the specimen is given by

$$dI_D = \alpha b I_0 \cdot e^{-\mu(AB+CD)} dx, \quad \text{Eq.2.1.8}$$

where  $a$  is the volume fraction of the crystallite that can diffract at this angle,  $d$  is the fraction of incident energy diffracted by unit volume and  $AB$  and  $CD$  are the path length of the incident and diffracted beam. From Fig.2.2 it can be seen that

$$l=1/\sin\alpha; AB=x/\sin\alpha; BC=x/\sin\beta,$$

thus

$$dI_D = (I_0 \cdot ab/\sin\alpha) \exp\{-\mu x(1/\sin\alpha + 1/\sin\beta)\} dx. \quad \text{Eq.2.1.9}$$

Thus, substituting  $\alpha=\theta+\psi$  and  $\beta=\theta-\psi$ ,

$$dI_D = \{I_0 \cdot ab/\sin(\theta+\psi)\} \exp\{-\mu x[1/\sin(\theta+\psi) + 1/\sin(\theta-\psi)]\} dx. \quad \text{Eq.2.1.10}$$

The total diffracted intensity for an infinitely thick specimen is;

$$dI_D = \int_{x=0}^{\infty} dI_D = \frac{I_0 \cdot ab}{2\mu} (1 - \tan\psi \cot\theta). \quad \text{Eq.2.1.11}$$

The angular dependence of absorption for this particular geometry is thus given by the term  $(1 - \tan\psi \cot\theta)$  which is called the absorption factor.

(f) The temperature causes a change in the  $d$  spacing [13]. The change of atomic spacing increases with increasing the temperature. As the temperature increases the intensity may decrease because the atomic planes can no longer be treated. Thus, at a sufficiently high temperature the intensity may become zero. Therefore, when the temperature effect is not negligible, the atomic scattering factor must be corrected. Due to the temperature diffuse scattering,

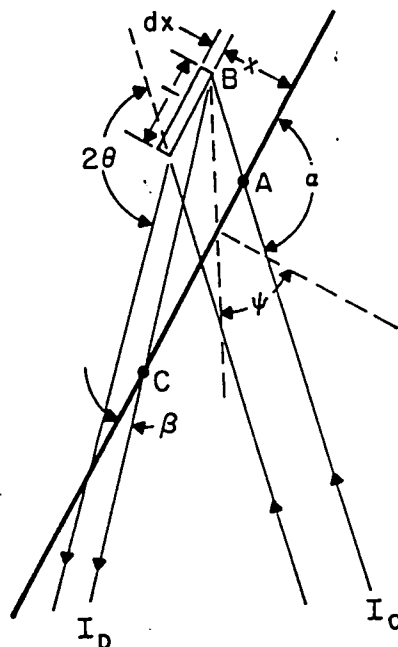


**Fig.2.2.** The geometry of the flat specimen and X-ray beam to calculate the absorption factor in a diffractometer

that is , general coherent scattering in all direction, background intensity increases progressively and merges finally with the decreased intensity peak as the temperature is raised up to the melting temperature of the materials.

#### 2.1.5 X-ray penetration depth

Most metals strongly absorb X-rays, and the intensity of the incident beam is reduced almost to zero at a few micrometers below the surface. The diffracted beams therefore originated mainly from a thin surface layer. As introduced

previously the integrated intensity diffracted by an infinitesimally thin layer located at a depth  $x$  below the surface is

$$dI_D = \frac{I_0 ab}{\sin \gamma} e^{-\mu x (1/\sin \alpha + \sin \beta)} \quad \text{Eq.2.1.12}$$

Let  $G_x$  a fraction of the total integrated intensity diffracted by a specimen of infinite thickness.

$$G_x = \frac{\int_{x=0}^{x=\infty} dI_d}{\int_{x=0}^{x=x} dI_d} = 1 - e^{-\mu x (1/\sin \alpha + 1/\sin \beta)} \quad \text{Eq.2.1.13}$$

In the case of the parafofocusing of diffractometer,  $\alpha = \beta = \theta$ , and Eq.2.1.13 reduces to

$$G_x = 1 - e^{-2\mu x (1/\sin \theta)} \quad \text{Eq.2.1.14}$$

## **2.2 Fundamental concepts of stress and strain fields**

### **2.2.1 Description of stresses**

It is often convenient to resolve the stresses into normal and shear. Normal stress is the component of the stress field acting in a direction normal to the plane, while shear stresses are the components of the stress field acting in the plane. Many types of notation are used to index stresses. One of them, most commonly used, is,

$$\sigma_{ij} = \begin{pmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{pmatrix}. \quad \text{Eq.2.2.1}$$

In all notations given above normal stresses are indicated by the repeated index (i.e.,  $\sigma_{11}, \sigma_{22}, \sigma_{33}$ ) while stresses with mixed indices (i.e.,  $\sigma_{12}, \sigma_{23}, \sigma_{31}$ ) are the shear components of the stress field. The first subscript indicates the plane in which the force is applied, while the second subscript gives the direction of the force.

### 2.2.2 Transformation of axes and tensor notation

When a polycrystalline specimen is loaded, being each grain differently oriented they will deform differently. Therefore, the applied stress may exceed the yield point in certain grains, whereas the rest of the grains may still be in the elastic region, causing inhomogeneous plastic deformation.

Let's assume two arbitrary unit cubes A and B in a homogeneously stressed body. (Fig.2.3(a)) If the forces acting on the surfaces of cube A are known, the forces acting on the surfaces of cube B can be determined by applying the transformation rules for tensors: (fig.2.3(b)) Since stress is a second-rank tensor, the transformation of the tensor  $\sigma_{ij}$  from the old coordinating system  $(x_1, x_2, x_3)$  to the new coordinating  $(x_1', x_2', x_3')$  is given by

$$\sigma_{kl} = a_{ki} a_{lj} \sigma_{ij}, \quad \text{Eq.2.2.2}$$

where  $a_{ij}$  is the direction cosine between  $x_i'$  and  $x_j$ . The repeated subindices (ij) in the right hand side implies summation for all the values that the subindex can have. The strain tensor is also second-rank tensor. Therefore, it has all of the properties described for stress. Thus, the strain tensor transformation from one set of coordinate axes to new coordinate axes can be expressed by

$$\epsilon_{kl} = a_{ki} a_{lj} \epsilon_{ij}. \quad \text{Eq.2.2.3}$$

For an isotropic body the direction of principal strains coincide with principal stress direction.[14]

### 2.2.3 Elastic stress-strain relations in an isotropic materials

The normal component of the stress tensor is related to the normal component of the strain tensor by Hookes' law,

$$\sigma_{11} = E \cdot \epsilon_{11}, \quad \text{Eq.2.2.4}$$

where E is the elastic modulus in tension or compression. A tensile force in the  $x_1$  direction produces an extension along that axis and a contraction in the transverse  $x_2$  and  $x_3$  directions. The transverse strain is a constant fraction of the strain in the longitudinal direction,

$$\epsilon_{11} = \epsilon_{22} = -\nu \epsilon_{33} = -\frac{\nu \sigma_{11}}{E}, \quad \text{Eq.2.2.5}$$

where  $\nu$  is the Poisson's ratio. For most metals the values of  $\nu$  are close to 0.33 [15]. Since the coordinates are arbitrary selected, the Eq.2.2.5 may be generalized for any stress  $\sigma_{ii}$ :

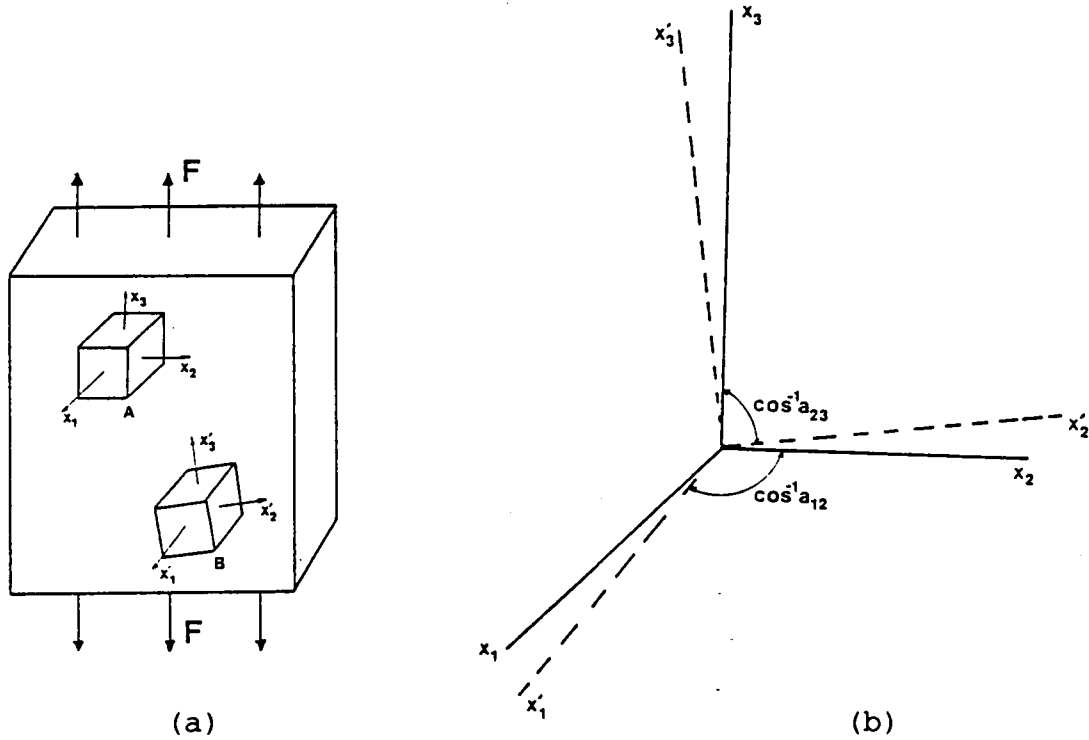


**Fig.2.3.** a) Two arbitrary unit cubes A and b in a uniformly stressed body, b) The new and old coordinate systems  $L(x_i')$  and  $S(x_i)$  and the definition of the direction cosines between them

$$\epsilon_{ii} = 1/E \sigma_{ii}, \quad \sigma_{jj} = -\frac{\nu \sigma_{ii}}{E}, \quad i \neq j. \quad \text{Eq.2.2.6}$$

From Eq.2.2.6 it is seen that for isotropic materials normal stresses  $\sigma_{ii}$  cause only dilatation. Pure distortion(shape change) occurs when a shear stress  $\sigma_{ij}$ ,  $i \neq j$ , is applied to the materials. The shear strain in this case is given by

$$2\epsilon_{ij} = \gamma = 1/\mu \cdot \sigma_{ij}, \quad i \neq j, \quad \text{Eq.2.2.7}$$

where  $\gamma$  is the engineering shear strain and the proportionality constants  $\mu$  is the shear modulus. For

isotropic materials  $\mu$  is related to  $E$  and  $\nu$  by  $2\mu = \frac{E}{(1+\nu)}$ .

Eq.2.2.6 and Eq.2.2.7 can be expressed compactly in the suffix notation as

$$\epsilon_{ij} = \frac{1+\nu}{E} \sigma_{ij} - \delta_{ij} \frac{\nu}{E} \sigma_{kk} \quad \text{Eq.2.2.8}$$

where  $k$  is dummy suffix and implies summation for all  $k$ , and  $\sigma_{ij}$  is Kroenecker's delta, which is defined as;

$$\delta_{ij} = \left\{ \begin{array}{ll} 1 & ; i=j \\ 0 & ; i \neq j \end{array} \right\} \quad \text{Eq.2.2.9}$$

Since single crystals, or grains in a polycrystalline materials, are macroscopically homogeneous but anisotropic, the simple equations between stress and strain for the isotropic behavior given in this section are no longer applicable. More general treatment in terms of the crystalline structure is therefore required.

#### 2.2.4 Anisotropy of elasticity behavior

When a force is applied to crystalline solid, it either pulls the atoms apart or pushes them together. The applied force is resisted by the forces of attraction or repulsion between the atoms. When an applied force is zero, the atoms are separated by a distance ' $a_0$ '. For a small applied forces, the atoms will find a new equilibrium spacing ' $a$ ' at which the external and internal forces are balanced. Along various directions in the point lattices the spacings between the atoms are different. Since the binding forces will be strongly affected by distance between atoms, the elastic

constants will vary with directions in the crystal lattice. In the generalized case [16 - 17] Hooke's law may be expressed as

$$\epsilon_{ij} = S_{ijkl}\sigma_{kl}, \quad \text{Eq.2.2.10}$$

and

$$\sigma_{ij} = C_{ijkl}\epsilon_{kl}, \quad \text{Eq.2.2.11}$$

where  $S_{ijkl}$  is the compliance tensor and  $C_{ijkl}$  is the elastic stiffness (often called elastic coefficient). Both  $S_{ijkl}$  and  $C_{ijkl}$  are fourth-rank tensor quantities. Expanding Eq.2.2.10 and Eq.2.2.11, we would get nine equations, each with nine terms and 81 constants. However, since both  $\epsilon_{ij}$  and  $\sigma_{ij}$  are symmetric tensors, that is,  $\sigma_{ij} = \sigma_{ji}$ , the number of constants can be considerably reduced [18].

The compliance tensor in any other coordinate system can be calculated from the transformation rule for fourth rank tensors;

$$S'_{ijkl} = a_{im}a_{jn}a_{ko}a_{lp} S_{mnop}, \quad \text{Eq.2.2.12}$$

where  $S'_{ijkl}$  is the elastic compliance in the new coordinate system,  $a_{ij}$  are the direction cosine between this system and the crystal axes, and  $S_{mnop}$  is the compliances defined in the crystal axes (old coordinate system). With Eq.2.2.12 Young's modulus in any arbitrary direction can be determined. Assume that a uniaxial stress  $\sigma$  is applied to a cubic single crystal along the direction  $[hkl]$ . Then strain along  $[hkl]$  is linearly related to the stress through Hooke's law:

$$\epsilon = \frac{1}{(E)_{hkl}} \cdot \sigma. \quad \text{Eq.2.2.13}$$

However, since the materials are anisotropic, Eq.2.2.13 can also be written through the general form of Hooke's law. Arbitrarily defining [hkl] as the xi' coordinate system,

$$\epsilon_{ij} = S'_{ijkl} \cdot \sigma_{kl}, \quad \text{Eq.2.2.14}$$

where  $S'_{ijkl}$  are referred to the xi' coordinate system.

Young's modulus along any oblique [hkl] can be written in terms of elastic compliance  $S_{mnop}$  defined in the crystal axes (xi coordinate system):

$$\frac{1}{(E)_{hkl}} = S'_{ijkl} = a_{im} a_{jn} a_{ko} a_{lp} S_{mnop}. \quad \text{Eq.2.2.15}$$

## 2.3 Residual stress analysis using X-ray diffraction method

### 2.3.1 Concept of the residual stress

Mura [19] defines residual stresses as the "Self-equilibrating internal stresses existing in a free body which has no external forces or constraints acting on its boundary". At equilibrium the integral of this stress in the volume of the body must be zero, and, as well, the integral over any plane through the specimen.

In Fig.2.4[20], two parts of a body, "bulk" and "near surface" regions, are shown separated. Suppose that for some reason the near-surface is elongated plastically and then joined it to the bulk with compressing elastically. The bulk puts this region under compression, while the near surface exerts tension on the bulk. Bending will result depending on

the thickness of layers. Residual stress arises in this manner, and stress type of near surface layer is compressive and that of the bulk is tensile. These stresses are called uniform macrostresses. Shot-peening, grinding, rolling and machining are examples of processes that cause internal stresses.

Almost no macrostresses will be developed in a multi-phase body pulled in tension, even if the phases have

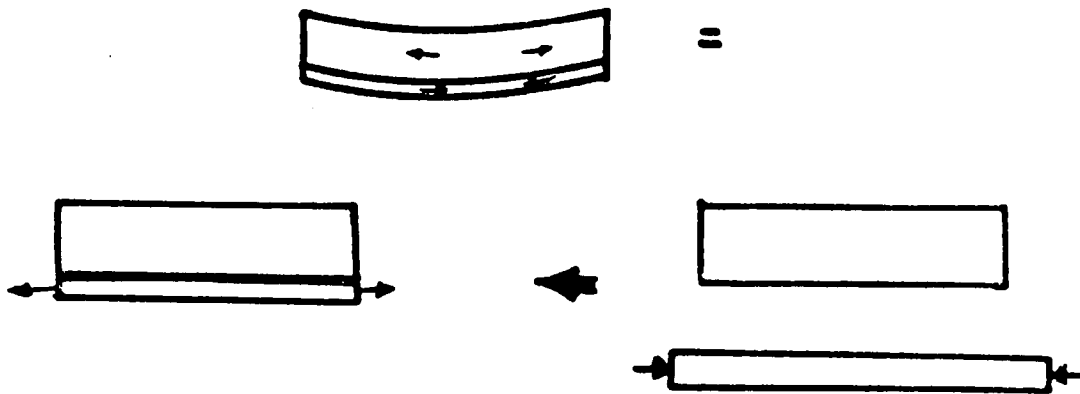


**Fig.2.4.** The near surface is longer than the interior causing the tensile residual stress on the surface.

different yield points, because the plastic deformation is uniform at all depths. However, the inhomogeneous partitioning of plastic strains between the phases due to the inhomogeneous distributions of yield points in the materials volume gives rise to residual stress field. Residual stresses of this type are called micro-stresses. These stresses can be

induced in a single phase polycrystalline body due to inhomogeneous distribution of plastic deformation between grains.

### 2.3.2 The measurement of the residual stresses

#### 2.3.2.1 Destructive method

One popular procedure for measuring residual stresses is by hole drilling or dissection [21]. Stress relief around a drilled hole produces a distortion around the cavity that can be measured with strain gauges. The operation should be carefully performed to avoid producing large stress. Another common method is to remove material by boring or electro-polishing and to determine the stresses by measuring the strains on the surface opposite to the one where material is removed [22]. In applying such methods, problems can also arise. For example, if a soft materials has a heavily deformed layer, removing materials may cause the interior to deform plastically.

#### 2.3.2.2 Non-destructive methods

##### a. Acoustics[23]

The basic idea of this method is that most solids are anharmonic; when a stress is applied there is a change in the

elastic constants. And, the velocity of wave propagation is altered.

b. Magnetic methods[24]

This technique is promising for field measurements on steels. The magnetization curve of a ferromagnetic materials is not as smooth as it is commonly drawn. The discrete motion of magnetic domains cause small sharp changes in magnetization as the field is applied. The existence of stress, local variation of dislocation density and micro structure can alter the signal.

c. Neutron scattering methods

Neutron produced at nuclear reactor during fission process can be used to determine the bulk residual stress at different depths from the surface.(Fig.2.5) The resolution in this case is the order of 1-2mm, depending on the neutron intensity(reactor power) [25].

2.3.3. Macro-stress analysis using X-ray diffraction method

2.3.3.1. Basic concepts

Stress-strain phenomena are conveniently illustrated by the dimensional changes of a uniform metal bar when tension is applied along its axis. Under such stress the bar is elongated, and its area of cross section is decreased in

direct proportion to the applied stress, provided the bar's elastic limit has not been exceeded. The resulting effect on each individual crystallite in the bar is an extension in direction parallel to the bar axis and a compression in the directions normal thereto. Crystal planes perpendicular to these extensive and compressive forces have their interplanar spacings changed by amount  $\pm\Delta d$ , and measurement of this change yields a measure of the elastic strain and thereby of

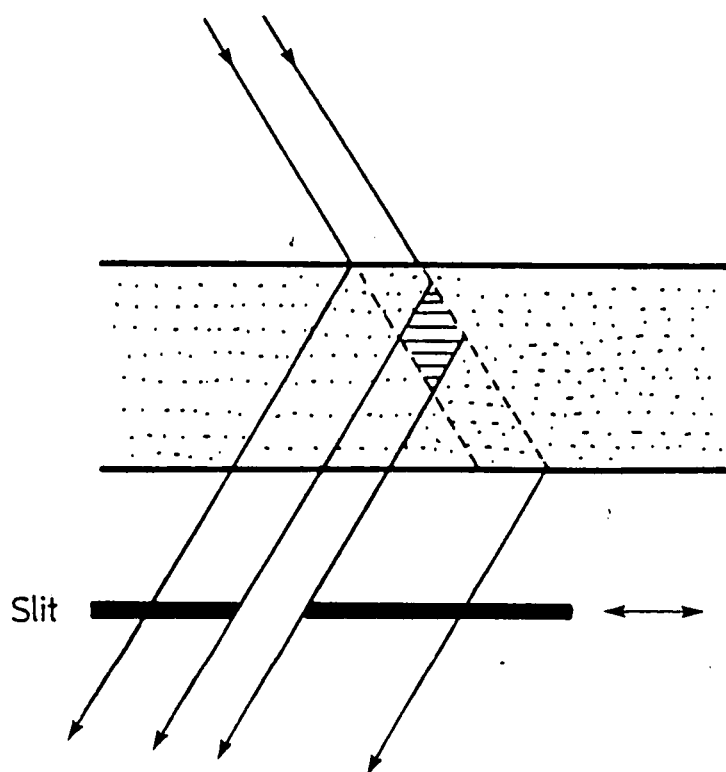


**Fig.2.5.** Schematic description of the neutron scattering method to measure the residual stress.

the stress as well. For crystal lying in the surface of the bar, such measurement can be made with high precision by back-reflection method as shown in Fig.2.6 [26].

#### 2.3.3.2 Basic equations and calculation procedure to determine stresses from X-ray data

Once the plane spacing is measured with diffraction, it is converted into strain:

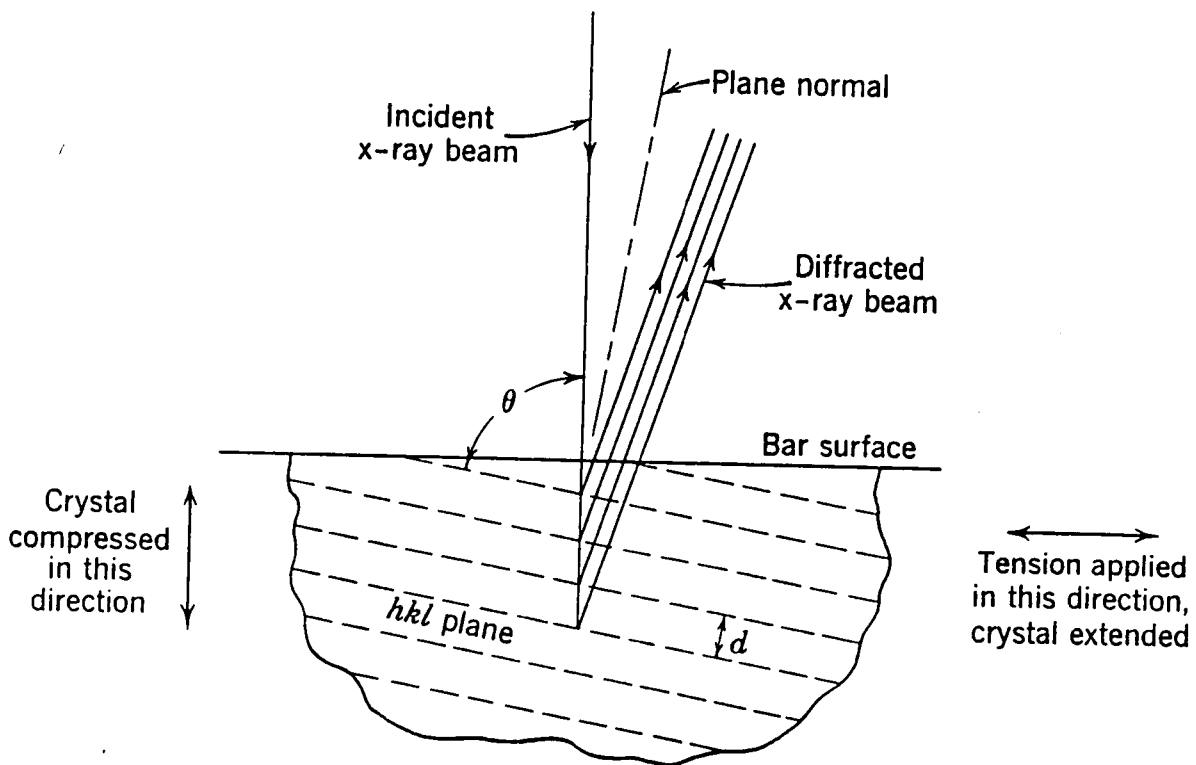


**Fig.2.6.** Stress-strain phenomena and back-reflection of x-rays in a surface crystal of a metal bar.

$$\langle \epsilon'_{33} \rangle = \frac{d_{\phi\psi} - d_0}{d_0}, \quad \text{Eq.2.3.1}$$

where  $d_0$  is the unstressed plane spacing and "< >" implies an X-ray average over the diffracting crystallites. The strain  $\epsilon'_{33}$  is along the  $\mathbf{L}_3$  direction and makes an angle  $\phi$  and  $\psi$  with  $\mathbf{S}_1$  and  $\mathbf{S}_3$  respectively as shown in Fig.2.7. In Fig 2.7, the  $\mathbf{S}$  coordinate system defines the specimen axes (old coordinate system) and the  $\mathbf{L}$  coordinate system defines the laboratory axes (new coordinate system) in which diffraction occurs. The strains in the  $\mathbf{L}$  and  $\mathbf{S}$  axes are related through the second rank tensor transformation:

$$\langle \epsilon'_{33} \rangle = a_{3k} a_{3l} \epsilon_{kl}, \quad \text{Eq.2.3.2}$$

where the primed and unprimed strain components are referred to the  $\mathbf{L}$  and  $\mathbf{S}$  axes respectively and  $a_{3k}$ ,  $a_{3l}$  are the direction cosines between these sets of axes. Eq.2.3.2 defines a set of linear equations in the unknown strains  $\epsilon_{kl}$  and can be solved for these strains if a sufficient number of  $\langle \epsilon'_{33} \rangle$  are measured at various independent  $\phi$  and  $\psi$  combinations. Once the strains  $\epsilon_{kl}$  are thus determined, the residual stress tensor can be calculated from Hooke's Law:

$$\sigma_{ij} = C_{ijkl} \epsilon_{kl}, \quad \text{Eq.2.3.3}$$

where  $C_{ijkl}$  are the stiffness coefficients of the materials in question. This procedure implicitly assumes that a homogeneous stress/strain distribution exists within the x-ray irradiated volume from which the lattice parameter data is obtained, since the components obtained from the above

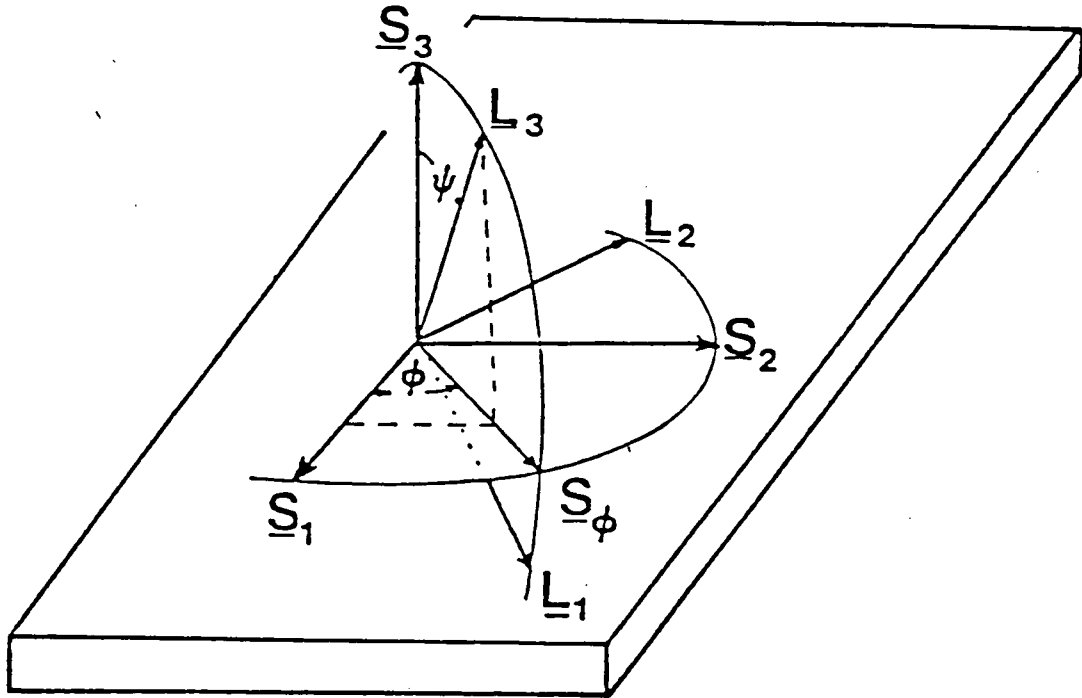


**Fig.2.7.** Definition of the laboratory coordinate system  $L_i$ , sample coordinate system  $S_i$  and the angles  $\phi$  and  $\psi$ .

treatments do not exhibit dependency on position within the irradiated volume.

Returning to the Eq.2.3.2, the direction cosine matrix for this case is

$$a_{ik} = \begin{pmatrix} \cos\phi\cos\psi & \sin\phi\cos\psi & -\sin\psi \\ -\sin\phi & \cos\phi & 0 \\ \cos\phi\sin\psi & \sin\phi\sin\psi & \cos\psi \end{pmatrix}. \quad \text{Eq.2.3.4}$$

Substituting for Eq.2.3.4 in Eq.2.3.1,

$$(\epsilon'_{33})_{\phi\psi} = \frac{d_{\phi\psi} - d_0}{d_0} = \epsilon_{11} \cos^2 \phi \sin^2 \psi + \epsilon_{12} \sin 2\phi \sin^2 \psi + \epsilon_{22} \sin^2 \phi \sin^2 \psi + \epsilon_{33} \cos^2 \psi + \epsilon_{13} \cos \phi \sin 2\psi + \epsilon_{23} \sin \phi \sin 2\psi. \quad \text{Eq.2.3.5}$$

This is a fundamental equation of X-ray strain determination. Assuming an homogeneous stress/strain distribution, Eq.2.3.5 predicts a linear dependency of  $d_{\phi\psi}$  in the  $\sin^2 \psi$  if the shear strain  $\epsilon_{13}, \epsilon_{23}$ , or corresponding shear strain, are zero.

(Fig.2.8(a)) In this case, the slope of the  $d_{\phi\psi}$  vs.  $\sin^2 \psi$  line is proportional to the product of the elastic constants of the material and residual stress in the  $\phi$  direction. The determination of the slope of this line is the classical method to determine residual stresses.[27] The strains in the sample coordinate system can be expressed in terms of stresses by the inverse of Eq.2.3.3:

$$\epsilon_{ij} = s_{ijkl} \sigma_{kl} \quad \text{Eq.2.3.6}$$

However, the elastic compliances are also referred to the  $S_i$  system of axes, and must be obtained from the elastic constants referred to the unit cell axes, by the tensor transformation rule for fourth rank tensors (Eq.2.2.15). For  $\epsilon_{ij}$ ,

$$\epsilon_{ij} = a_{im} a_{jn} a_{ko} a_{lp} s_{mnop} \sigma_{kl}. \quad \text{Eq.2.3.7}$$

Here  $a_{ko}$  are direction cosines between the crystal axes and the surface coordinate system and  $s_{mnop}$  is defined in the crystal axes. If the material is isotropic, Eq.2.3.7 reduces to Eq.2.2.8,

$$\epsilon_{ij} = \frac{1+\nu}{E} \sigma_{ij} - \delta_{ij} \frac{\nu}{E} \sigma_{kk}$$

Substituting above equation into Eq.2.3.5 yields

$$\begin{aligned} \frac{d_{\psi}-d_0}{d_0} &= \frac{1+\nu}{E} \{ \sigma_{11} \cos^2 \phi + \sigma_{12} \sin 2\phi + \sigma_{22} \sin^2 \phi - \sigma_{33} \} \sin^2 \psi + \\ &\frac{1+\nu}{E} \sigma_{33} \cos^2 \psi - \frac{\nu}{E} (\sigma_{11} + \sigma_{22} + \sigma_{33}) + \frac{1+\nu}{E} \{ \sigma_{13} \cos \phi + \sigma_{23} \sin \phi \} \sin 2\psi. \end{aligned} \quad \text{Eq.2.3.8}$$

Therefore, if the  $d$  vs.  $\sin^2 \psi$  plot obtained from the surface layer is linear, the shear stress  $\sigma_{13}$ ,  $\sigma_{23}$  will be zero. And, an oscillation in the  $d$  vs.  $\sin^2 \psi$  plot (Fig.2.8(c)) indicates the presence of the shear stresses.

If the stress tensor existing in the X-ray irradiated layers is biaxial,  $\sigma_{13}$ ,  $\sigma_{23}$  and  $\sigma_{33}$  terms are zero in Eq.2.3.8.

Therefore, in biaxial stress condition Eq.2.3.8 becomes

$$\frac{d_{\psi}-d_0}{d_0} = \frac{1+\nu}{E} \sigma_{\phi} \sin^2 \psi - \frac{\nu}{E} (\sigma_{11} + \sigma_{22}), \quad \text{Eq.2.3.9}$$

where  $\sigma_{\phi}$ , the stress component along the  $S_{\phi}$  direction, is given by

$$\sigma_{\phi} = \sigma_{11} \cos^2 \phi + \sigma_{22} \sin^2 \phi$$

or

$$\sigma_{\phi} = \sigma_{11} \cos^2 \phi + \sigma_{12} \sin 2\phi + \sigma_{22} \sin^2 \phi. \quad \text{Eq.2.3.10}$$

Eq.2.3.9 is a form of the traditional X-ray residual stress equation, and has been used for over 60 years [28]. It predicts a linear variation of  $d$  vs.  $\sin^2 \psi$ . The stress may be obtained directly from the slope of a least square line fitted to experimental data, measured at various  $\psi$ , if the elastic constants  $E$ ,  $\nu$  and the unstressed plane spacing,  $d_0$ ,

are known. This procedure is known as the  $\sin^2\psi$  technique [29] since it utilizes multiple  $\psi$  tilts.

If the shear stresses exist in the diffracted volume, a " $\psi$ -split" variation is predicted with opposite curvature for the branches measured at positive and negative  $\psi$  (Fig.2.8(b)). On the other hand, oscillatory variation of  $d_{\phi\psi}$  with  $\sin^2\psi$  (Fig2.8(c)) indicates the presence of inhomogeneous

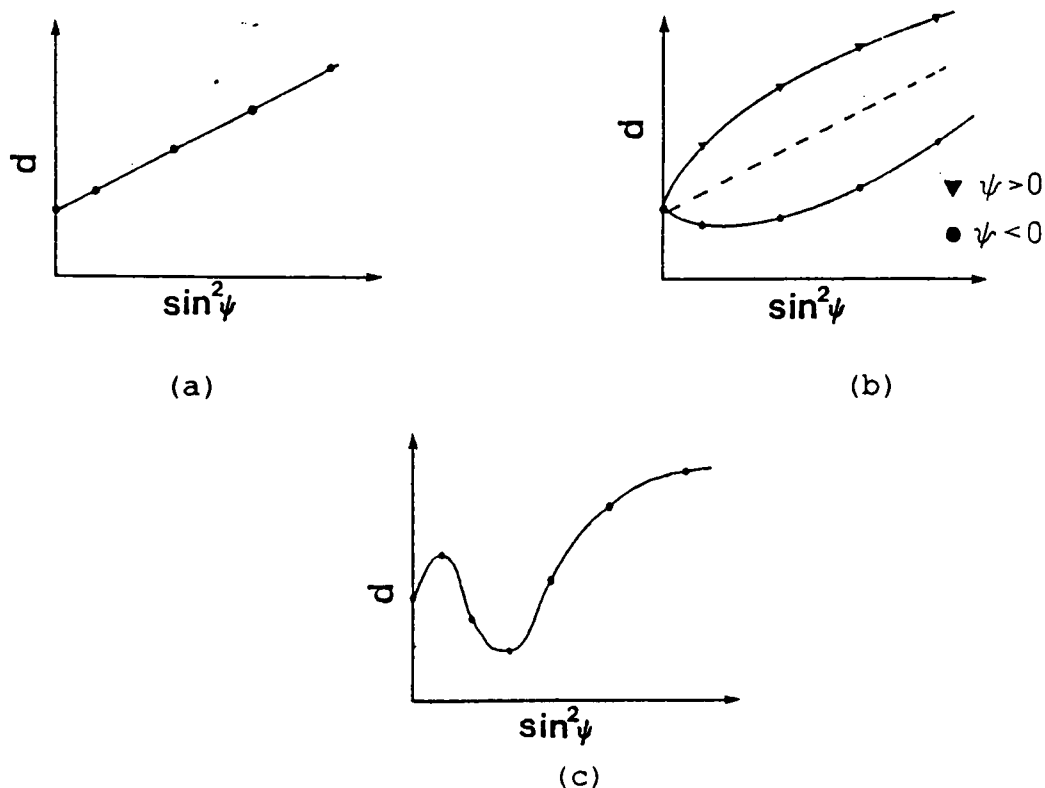


**Fig.2.8.** Types of " $d$ " vs.  $\sin^2\psi$  plots. Plots (a) and (b) denote regular behavior. The plot(c) exhibits oscillatory behavior implying the presence of inhomogeneous stress and strain gradients.

stress/strain gradients within the X-ray irradiated volume, where the strain at a point is expressed as

$$\epsilon_{ij}^t = \epsilon_{ij}^o + \epsilon_{ij}^{in} + \epsilon_{ij}^r. \quad \text{Eq. 2.3.11}$$

$\epsilon_{ij}^o$  is the elastic strain that would be observed if the materials was homogeneous.  $\epsilon_{ij}^{in}$  is the interaction strain term due to inhomogeneous distribution of the elastic properties and  $\epsilon_{ij}^r$  is a residual strain term that is caused by inhomogeneous partitioning of plastic deformation.

### 2.3.3.3 Interpretation of stresses from oscillatory data [30]

As already shown in the previous section, oscillatory  $d$  vs.  $\sin^2\psi$  data indicates the presence of an inhomogeneous strain/stress distribution within the irradiated volume for the particular lattice plane  $hkl$  used in the measurement. Analysis of such data with the regular procedures may cause large errors in the results. In this case, the total strain,  $\epsilon_{ij}^t$ , measured by X-rays may contain  $\epsilon_{ij}^o$ ,  $\langle \epsilon_{ij}^{in} \rangle$ , and  $\langle \epsilon_{ij}^r \rangle$ . Thus, if a homogeneous stress  $\sigma_{11}^o$  in the  $S_1$  direction is applied at the boundary of the materials, the component of the strain tensor at any point A in the materials may be expressed as

$$\epsilon_{11}(x, y, z) = \sigma_{11}^o \cdot \{1/E + k_1'(x, y, z)\}$$

$$\epsilon_{22}(x, y, z) = \sigma_{11}^o \cdot \{-\nu/E + k_2'(x, y, z)\}$$

$$\epsilon_{33}(x, y, z) = \sigma_{11}^o \cdot \{-\nu/E + k_3'(x, y, z)\}$$

$$\epsilon_{13}(x, y, z) = \sigma_{11}^o \cdot k_4'(x, y, z)$$

$$\epsilon_{23}(x,y,z) = \sigma_{11}^0 \cdot k5'(x,y,z)$$

$$\epsilon_{12}(x,y,z) = \sigma_{11}^0 \cdot k6'(x,y,z) \quad . \quad \text{Eq.2.3.12}$$

Here  $x$ ,  $y$  and  $z$  are the coordinates of the point A with respect to a coordinate system describing the surface, and  $k_i'(x,y,z)$  are the proportionality constants at A between the applied load and the resulting reaction strains. The constants  $k4'$ ,  $k5'$ ,  $k6'$  may be have non-zero values depending on the symmetry of the constitutive grains of the materials or the symmetry of texture in the surface layers. Also, in certain cases the local symmetry changes as one approaches a grain boundary. In the following discussion it is assumed, for simplicity, that  $k4'=k5'=k6'=0$ . (If these terms are different from zero, the discussion following along the same lines but the equations must be modified to take into account the shear strains  $\epsilon_{13}, \epsilon_{12}, \epsilon_{23}$ .) From Eq.2.3.5 and Eq.2.3.12, one can obtain for  $\phi=0$ ,

$$\langle \epsilon'_{33} \rangle_{\psi} = \frac{\langle d \rangle_{\psi} - d_0}{d_0} = (\langle \epsilon^t_{11} \rangle_{\psi} - \langle \epsilon^t_{33} \rangle_{\psi}) \cdot \sin^2 \psi + \langle \epsilon^t_{33} \rangle_{\psi}.$$

Eq.2.3.13

By substituting relevant equations in Eq.2.3.11 and Eq.2.3.12 into Eq.2.3.13, the relationship between the applied stress and the measured strains is obtained;

$$\begin{aligned} \langle \epsilon'_{33} \rangle_{\psi} = \frac{\langle d \rangle_{\psi} - d_0}{d_0} = & [\sigma_{11}^0 \left\{ \frac{1+\nu}{E} + K1(\psi) + K3(\psi) \right\} + \langle \epsilon^r_{11} \rangle_{\psi} - \\ & \langle \epsilon^r_{33} \rangle_{\psi}] \sin^2 \psi + \sigma_{11}^0 \left\{ -\frac{\nu}{E} + K3(\psi) \right\} + \langle \epsilon^r_{33} \rangle_{\psi}. \end{aligned} \quad \text{Eq.2.3.14}$$

The first term in brackets in Eq.2.3.14 is the homogeneous strain that causes a linear variation of  $d$  with  $\sin^2\psi$ . The second and third terms are inhomogeneous strain terms which cause the non-linearity in  $d$  vs.  $\sin^2\psi$ . In Eq.2.3.14, the stress/strain terms are the X-ray averages of the local variations over the diffracting volume. Since the diffracting volume itself is a function of  $\psi$ , these terms are expressed here as variables in  $\psi$ . It can be seen from Eq.2.3.14 that  $d_{\phi\psi}$  is linear in  $\sin^2\psi$  if  $\langle\epsilon^{rij}\rangle_\psi$  and  $K_i(\psi)$  are independent of  $\psi$ . If these terms are both zero, the materials is equivalent to an isotropic homogeneous substance, and the X-ray stress analysis using Eq.2.3.14 will yield  $\sigma_{11}^0$ . If these terms are finite, but independent of  $\psi$ , the  $d_{\phi\psi}$  is still linear in  $\sin^2\psi$  but the analysis will yield a stress value with an average term that is proportional to the inhomogeneity effects added to  $\sigma_{11}^0$ . If, on the other hand,  $K_i(\psi)$  or  $\langle\epsilon^{rij}\rangle_\psi$  are non-linear in  $\psi$ , oscillations will occur in the  $d_{\phi\psi}$  versus  $\sin^2\psi$  curve. Determination of the macro stress,  $\sigma_{11}^0$ , and the average microstrain,  $\langle\epsilon^{rij}\rangle_\psi$ , from Eq.2.3.14 is not easy. If the interaction constants  $K_i(\psi)$  at each tilt, as well as the functional dependence of  $\langle\epsilon^{rij}\rangle_\psi$  on  $\psi$  is known,  $d$  vs.  $\sin^2\psi$  plots may be analyzed by curve fitting, or non-linear least squares analysis. However, in the general case, the equations describing  $K_i(\psi)$  and  $\langle\epsilon^{rij}\rangle_\psi$  are not simple functions of  $\psi$  and require the definition of a large number of parameters which can cause large errors in

the results because the interaction constants  $K_i(\psi)$  and the inhomogeneous residual strain distribution depend on the texture function and the distribution of grain shapes in the given diffraction volume.

#### 2.3.4 Diffractometric and analytic techniques for measuring macro residual stresses

##### 2.3.4.1 Alignment of diffractometer

Actually, in the stress analysis the quantity measured in the diffractometer method is  $\Delta 2\theta$ , the shift in diffraction line position due to stress as the angle  $\psi$  is changed.

Therefore, the alignment of the diffractometer is critical in the stress measurement. Instrument misalignment can introduce small errors which change in  $2\theta$  even for a stress free specimen. When  $\psi$  is changed from  $0^\circ$  to  $45^\circ$ , it is therefore necessary to determine this change experimentally and apply it as a correction. If  $(\Delta 2\theta)_0$  is the line shift for a stress free specimen and  $(\Delta 2\theta)_m$  the measured shift for a stressed specimen, then the line shift due to stress is

$$\Delta 2\theta = (\Delta 2\theta)_m - (\Delta 2\theta)_0. \quad \text{Eq. 2.3.15}$$

The correction to minimize  $(\Delta 2\theta)_0$  is best determined on a specimen which is necessarily free of stress. For this purpose a fully annealed specimen or stress free fine powder specimen can be used. In the case of stress analysis a value

of  $(\Delta 2\theta)_0$  greater than about  $0.03^\circ$  needs the better instrument alignment.

#### 2.3.4.2 Determination of peak position

In the stress measurement, the analysis is based on the strains  $\epsilon_{\phi\psi}$ , given by  $\epsilon_{\phi\psi} = \frac{d_{\phi\psi} - d_0}{d_0}$ . Since the strains  $\epsilon_{ij}$  are less than 0.01% for elastic behavior in most materials, the absolute value of the peak position, i.e., the true peak maximum, is required for the accurate calculation of  $d_{\phi\psi}$  from Bragg's law and an appropriate method to determine peak location must be used.

Half value method and centroid methods [31] can be used after the appropriate angular corrections of the raw intensities. However, one of the most popular methods today is the parabola fitting method. This method was first used by Ogilivie [32]. In this method, it is assumed that a region in the vicinity of the peak can be represented by a parabola. Therefore, the center of a diffraction line is determined by fitting a parabola to the top of the line and taking the axis of parabola whose axis is parallel to the intensity ordinate can be given

$$I(2\theta_i) = I_0 + c(2\theta_i - 2\theta_0)^2 \quad \text{Eq.2.3.16}$$

by neglecting the third or higher order terms of the Cauchy or the Gaussian functions [33]. Here,  $c$  is a constant. In this method, two critical factors are the region of parabola fit and the number of points used in fitting the parabola. An

empirical rule proposed by Koistinen and Marburger[34] is using the 85% of the maximum intensity in fitting the parabola. This so called "top 15%" rule has been accepted widely in practice. In this method, we substitute several pairs of  $2\theta$ ,  $I(2\theta)$  values into the equation and solve for  $2\theta_p$  by the method of least squares. Various software packages are available for such a fit.

#### 2.3.4.3 The Rachinger correction [35]

In most of the procedures above a symmetric peak was assumed when no resolution of the  $\alpha_1 - \alpha_2$  doublet is observed. This is mostly the case for severely deformed materials. When the doublet is totally or partially resolved, however, most of the procedures discussed above must be modified to take into account the peak asymmetry. In this case, the method of Rachinger is effective and more easily applied. It is assumed that  $\alpha_1$  and  $\alpha_2$  line profile are identical in shape and not necessarily symmetrical, and that the  $\alpha_2$  profile of the one half the intensity of the  $\alpha_1$  profile and shifted from it toward large angles by

$$\Delta 2\theta = 2 \tan \theta \left( \frac{\Delta \lambda}{\lambda} \right), \quad \text{Eq. 2.3.17}$$

$\Delta \lambda$  being the dispersion separating  $\lambda(\alpha_2) - \lambda(\alpha_1)$  in Å. Now let  $I_i$  be the intensity of the experimental profile at point  $i$  in the foregoing series of  $n$  intervals, and let  $I_i(\alpha_1)$  be that part of  $I_i$  due to  $\alpha_1$  alone. then

$$I_i(\alpha_1) = I_i - \frac{1}{2}I_{i-m}(\alpha_2). \quad \text{Eq.2.3.18}$$

Here,  $m$  is the number of interval between  $K\alpha_1$  intensity peak and  $K\alpha_2$  intensity peak position, and then  $\frac{1}{2}I_{i-m}(\alpha_2)$  is the  $K\alpha_2$  intensity data. Thus,  $K\alpha_1$  intensity peak can be obtained.

### 2.3.5 Effect of small coherent domains and strains - Line broadening analysis

#### 2.3.5.1 The size and strain effects on line broadening

Suppose that the crystal has a thickness  $t$  measured in a direction perpendicular to a particular set of reflection planes. Let there be  $(n+1)$  planes in this set. We call  $\theta_B$  the angle which exactly satisfies the Bragg law for the particular values  $\lambda$  and  $d$  involved, or

$$\lambda = 2d\sin\theta_B.$$

At the diffraction angle  $2\theta_B$ , reflecting rays from each planes are completely in phase and add to form a diffracted beam of maximum amplitude, hence the beam of maximum intensity (as intensity is proportional to the square of the amplitude). When a ray from the surface makes slightly larger angle  $\theta_1$ , a ray from the  $n^{\text{th}}$  plane below the surface is  $(n+1)$  wavelength out of phase with the ray from the surface. This means that midway in the crystal there is a plane scattering a ray which is an integer plus one-half wave length out of the phase with the ray from the surface plane. These rays cancel one another, and similarly for the other rays from

other pairs of planes throughout the crystal, so that rays scattered by the top half of the crystal cancel those scattered by the bottom half. The intensity of the beam diffracted at  $2\theta_1$  will be zero. Similarly, a ray from the surface that makes slightly smaller angle  $\theta_2$  is  $(n-1)$  wavelength out of phase with the ray from the  $n^{\text{th}}$  plane below the surface. So, the intensity of the beam diffracted at  $2\theta_2$  will decrease to zero. As decreasing the number of reflecting planes,  $\Delta\theta$  ( $=\theta_1-\theta_B$  or  $\theta_B-\theta_2$ ) will be increased. This means that the smaller the thickness  $t$ , the wider is the range  $2\theta_1-2\theta_2$ . The relationship is given by

$$t = \frac{\lambda}{B \cos \theta_B} \cos \theta_B, \quad [36]$$

$$\text{more exactly, } t = \frac{0.9\lambda}{B \cos \theta_B} \cos \theta_B \quad \text{Eq.2.3.19}$$

, where  $B$  is  $(\theta_1-\theta_2)$ .

We may imagine a grain to be composed of a number of small regions in each of which the plane spacing is substantially constant but different from the spacing in adjoining regions. This regions cause the various sharp lines, each slightly displaced from the other, is the broadened diffraction line shown by the full curve and, of cause, the broadened line is the only one experimentally observable. The relationship between the broadening produced and the non-uniformity of the strain is given by differentiating the Bragg's law.

$$\Delta 2\theta^\circ = -\frac{2\Delta d}{d} \tan \theta. \quad \text{Eq.2.3.20}$$

Actually, peak broadening is related to the standard deviation,  $\langle (\frac{\Delta d}{d})^2 \rangle^{1/2}$ . Therefore, the higher the strain, the wider is the range  $2\theta_1 - 2\theta_2$ .

From Eq.2.3.19 and Eq.2.3.20, one can obtain

$$\Delta 2\theta^0 \cos\theta = \frac{180}{\pi} \left[ \frac{\lambda}{D} - 2 \langle (\frac{\Delta d}{d})^2 \rangle^{1/2} \cdot \sin\theta \right]. \quad \text{Eq.2.3.21}$$

Thus, from a plot of  $\Delta 2\theta^0 \cos\theta$  vs  $\sin\theta$ ,  $D$  is obtained from the intercept and the RMS microstrain from the slope. In this case, multiple order of an hkl peak should be employed, not just any peaks, as most materials are elastically anisotropic. The size is actually an average over the volume of the X-ray beam. Actually there is a better way using Fourier series with several assumptions in analyzing the X-ray line broadening. Thus, the method using Fourier method will be used in this experiment.

#### 2.3.5.2 Separation of the pure broadening from the total X-ray peak

The geometrical properties of the diffractometer introduce aberrations into the pure diffraction profile which cause it to be more or less asymmetrical, broadened, and displaced from its theoretical angle  $2\theta$  on the goniometer scale. The breadth and shape of a diffraction line are important in the measurement of crystallite size and lattice distortions, while the displacement of the line is significant in the determination of accurate interplanar

spacings and lattice constants as discussed previously. According to the superposition theorem[37], the profile of the observed maximum  $h(\epsilon)$  is the convolution or fold, of the pure diffraction profile  $f(\epsilon)$  and the weight function of the instrumental  $g(\epsilon)$ . The geometrical weight function  $g(\epsilon)$  of a well aligned parafocusing diffractometer may reasonably be regarded as consisting of six specific instrumental functions, among which are X-ray source function, flat specimen surface function, axial divergence function, specimen transparency function, receiving slit function and misalignment function [38]. The pure profile  $f(\epsilon)$  are determined both by the mean crystallite size, or distribution of sizes, characterizing the specimen, and by the particular imperfections prevailing in the crystalline lattice. Hence in principle at least, an appropriate analysis of the line profile  $f(\epsilon)$  should yield such information as the mean crystallite dimension or size of the coherent domains, and the distribution of imperfections. In order to get such imperfections we need to extract the pure diffraction profile  $f(\epsilon)$ . The convolution relationship among  $h(\epsilon)$ ,  $g(\epsilon)$ , and  $f(\epsilon)$  is given below:

$$h(\epsilon) = \int_{-\infty}^{\infty} g(\eta) f(\epsilon - \eta) d\eta. \quad \text{Eq.2.3.22}$$

Thus  $f(\epsilon)$  can be obtained from  $h(\epsilon)$  by the process of deconvolution(unfolding). Let the Fourier transforms  $h(\epsilon)$ ,  $g(\epsilon)$  and  $f(\epsilon)$  be  $H(\zeta)$ ,  $G(\zeta)$ , and  $F(\zeta)$ , respectively. By

application Fourier's integral theorem [39], it can be shown that

$$F(\zeta) = \frac{H(\zeta)}{G(\zeta)}, \quad \text{Eq.2.3.23}$$

and furthermore [40]

$$\begin{aligned} f(\epsilon) &= \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\infty} F(\zeta) \exp(-2\pi i \epsilon \eta) d\zeta \\ &= \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\infty} \frac{H(\zeta)}{G(\zeta)} \exp(-2\pi i \epsilon \eta) d\zeta. \end{aligned} \quad \text{Eq.2.3.24}$$

According to Stoke's method [41], the integrals are replaced by the corresponding summations, and the limits of  $\epsilon$  are changed from  $\pm\infty$  to  $\pm\epsilon_m$ , the maximum values of beyond which the intensity can be considered to have fallen to its background value. Eq.2.3.24 may be written as

$$f(\epsilon) = \frac{1}{\sqrt{2\pi}} \sum_{\zeta} \frac{H(\zeta)}{\epsilon_m G(\zeta)} \exp\left(\frac{-2\pi i \epsilon \eta}{\epsilon_m}\right) \Delta\zeta. \quad \text{Eq.2.3.25}$$

By eliminating the constant factors  $1/\sqrt{2\pi}$ ,  $1/\epsilon_m$ , and  $\Delta\zeta$  from the computations because they affect only the height of a line profile and not its shape,

$$f(\epsilon) = \sum_{\zeta} \frac{H(\zeta)}{G(\zeta)} \exp\left(\frac{-2\pi i \epsilon \eta}{\epsilon_m}\right), \quad \text{Eq.2.3.26}$$

in which

$$H(\zeta) = \sum_{\epsilon} h(\epsilon) \exp\left(\frac{2\pi i \epsilon \eta}{\epsilon_m}\right), \quad \text{Eq.2.3.27}$$

$$G(\zeta) = \sum_{\epsilon} g(\epsilon) \exp\left(\frac{2\pi i \epsilon \eta}{\epsilon_m}\right). \quad \text{Eq.2.3.28}$$

Now, since

$$e^{iz} = \cos z + i \sin z, \quad \text{Eq.2.3.29}$$

it is evident that each of the series above consists of real and imaginary parts and they may be represented by

$$G(\zeta) = G_r(\zeta) + iG_i(\zeta), \quad \text{Eq.2.3.30}$$

$$\text{and } H(\zeta) = H_r(\zeta) + iH_i(\zeta). \quad \text{Eq.2.3.31}$$

Therefore

$$F(\zeta) = \frac{H(\zeta)}{G(\zeta)} = \frac{H_r + iH_i}{G_r + iG_i} = \frac{(H_r + iH_i)(G_r - iG_i)}{G_r^2 - G_i^2}. \quad \text{Eq.2.3.32}$$

F has the real( $F_r$ ) and imaginary parts( $F_i$ ). Thus,  $F_r$  and  $F_i$  can be calculated from the Fourier series  $G_r$ ,  $G_i$ ,  $H_r$  and  $H_i$ , which can be evaluated from experimental data;

$$F_r = \frac{H_r G_r + H_i G_i}{G_r^2 - G_i^2} \quad \text{Eq.2.3.33}$$

$$\text{and } F_i = \frac{H_i G_r - H_r G_i}{G_r^2 + G_i^2}. \quad \text{Eq.2.3.34}$$

Returning to Eq.2.3.26, the desired pure diffraction profile  $f(\epsilon)$  can be computed as follows;

$$\begin{aligned} f(\epsilon) = & \sum_{\zeta} F_r(\zeta) \cos\left(\frac{2\pi\epsilon\zeta}{\epsilon_m}\right) + \sum_{\zeta} F_i(\zeta) \sin\left(\frac{2\pi\epsilon\zeta}{\epsilon_m}\right) \\ & - i \sum_{\zeta} F_r(\zeta) \sin\left(\frac{2\pi\epsilon\zeta}{\epsilon_m}\right) + i \sum_{\zeta} F_i(\zeta) \cos\left(\frac{2\pi\epsilon\zeta}{\epsilon_m}\right). \end{aligned} \quad \text{Eq.2.3.35}$$

Because  $F(-\zeta)$  is the complex conjugate of  $F(\zeta)$ ,  $F_r(-\zeta) = -F_r(\zeta)$  and  $F_i(-\zeta) = -F_i(\zeta)$ . Terms  $-i \sum_{\zeta} F_r(\zeta) \sin\left(\frac{2\pi\epsilon\zeta}{\epsilon_m}\right)$

and  $i \sum_{\zeta} F_i(\zeta) \sin\left(\frac{2\pi\epsilon\zeta}{\epsilon_m}\right)$  in Eq.2.3.35 reduce to zero, and  $f(\epsilon)$  can

be calculated from equation

$$f(\epsilon) = \sum_{\zeta} F_r(\zeta) \cos\left(\frac{2\pi\epsilon\zeta}{\epsilon_m}\right) + \sum_{\zeta} F_i(\zeta) \sin\left(\frac{2\pi\epsilon\zeta}{\epsilon_m}\right). \quad \text{Eq.2.3.36}$$

Eq.2.3.36 expresses the pure line profile.

The computation would be greatly simplified by elimination of the 'sine' terms if the functions  $f$ ,  $h$  and  $g$  were symmetrical. In many experimental line profiles, due to  $K\alpha$  doublet, line profiles are asymmetrical. Rachinger, Papoulis[42], and Keating[43] have developed general methods to eliminate the  $k\alpha_2$  contribution from the combined  $k\alpha_1$  and  $k\alpha_2$  functions  $h(\epsilon)$  and  $g(\epsilon)$ . In this experiment the method of Rachinger correction is used in separating the symmetrical  $k\alpha_1$  peak from the combined peak.

#### 2.3.5.3 Separation of crystal size and lattice strains from pure line broadening[44]

There have been several suggestions advanced in explaining the nature of the imperfections:

(a) The material is broken up into crystallites, or coherently diffracting domains in the case of metallic grains, so small(100 to 1000Å) that diffraction broadening occurs.

(b) The materials is broken up into large crystallite size about  $1\mu\text{m}(10,000\text{\AA})$ , with lattice spacing differing from one to another.

(c) The crystallites composing the materials remain fairly large(about  $1\mu$  in size), but they are individually elastically distorted, suffer deformation faulting, or undergo both effects.

Case (a) and (b) is favored. A reasonable general interpretation [45] is that cold working produces dislocation arrays and small crystallites. These domains are visualized as being sufficiently disoriented from one to the next that they scatter incoherently with respect to one another. The dislocations that cause the subdivision of the original larger grains into domains also produce tensile and compressive strains within the domains. The formation of both small crystallites and the elastic strains introduced by dislocations results in broadening of the line profile.

#### A. Derivation of the Fourier series expression

Fourier series method of Warren and Averbach has been supported by a lot of experiences since it was developed for investigating of cold-working effect in metals. Assuming that the Bragg's law is satisfied, we can express the intensity of the beam diffracted by a crystal as a function of atom position. Since the crystal is merely a repetition of the fundamental unit cell, the arrangement of atoms within single unit cell affects the diffracted intensity. The wave vectors scattered by unit cell have different wave functions and will produce the resultant wave vector by summing up all the wave vectors from the atoms in a unit cell. If a unit cell contains atoms,  $1, 2, 3, \dots, N$ , with fractional coordinates  $u_1v_1w_1, u_2v_2w_2, u_3v_3w_3, \dots, u_Nv_Nw_N$  and

atomic scattering factors  $f_1, f_2, f_3, \dots, f_N$ , the structural factor is then given by

$$F(hkl) = \sum_N f_N \exp 2\pi i (hu_N + kv_N + lw_N). \quad \text{Eq.2.3.37}$$

The intensity in electron units is given by

$$I_{eu} = F^2 \sum_{mm'} \exp \left[ \frac{2\pi i}{\lambda} (\mathbf{s} - \mathbf{s}_0) (\mathbf{R}_m' - \mathbf{R}_m) \right]. \quad \text{Eq.2.3.38}$$

where  $\mathbf{s}_0$  and  $\mathbf{s}$  are unit vectors giving directions of the primary and diffracted beams, and  $\mathbf{R}_m$  and  $\mathbf{R}_m'$  are the vector locations of the origins of particular unit cells  $m_1 m_2 m_3$  and  $m_1' m_2' m_3'$  respectively. Representing the diffraction vector by its terminal point in reciprocal space,

$$(\mathbf{s} - \mathbf{s}_0) / \lambda = h_1 \mathbf{b}_1 + h_2 \mathbf{b}_2 + h_3 \mathbf{b}_3, \quad \text{Eq.2.3.39}$$

where  $\mathbf{b}_1, \mathbf{b}_2, \mathbf{b}_3$  are reciprocal vectors, and  $h_1, h_2, h_3$  are continuous variables. The intensity per crystal can thus be written

$$I(h_1 h_2 h_3) = F^2 \sum_{m'm'} \exp \left[ 2\pi i (h_1 (m_1' - m_1) + h_2 (m_2' - m_2) + h_3 (m_3' - m_3) + \frac{\mathbf{s} - \mathbf{s}_0}{\lambda} (\delta m' - \delta m)) \right]. \quad \text{Eq.2.3.40}$$

The diffracted power theorem for powder pattern is stated by

$$P = \frac{MR^2 \lambda^3}{4v_a} \int \int \int \frac{I(h_1 h_2 h_3)}{\sin \theta} dh_1 dh_2 dh_3, \quad \text{Eq.2.3.41}$$

where  $M$  is the number of crystals in the powder sample,  $R$  is the distance between sample and receiver,  $v_a$  is the volume of the unit cell and  $I(h_1 h_2 h_3)$  is the intensity per crystal (the variables  $d\alpha d\beta d\gamma$  in the Fig.2.9 can be replaced by  $dh_1 dh_2 dh_3$

in the reciprocal space). (Fig.2.9) The distribution of power angle is expressed by  $P_{2\theta}$ , where

$$P = \int P_{2\theta} d(2\theta) \quad . \quad \text{Eq 2.3.41}$$

Integrating about the point 001, it is assumed that spreading out of the intensity is small as compared to the distance of

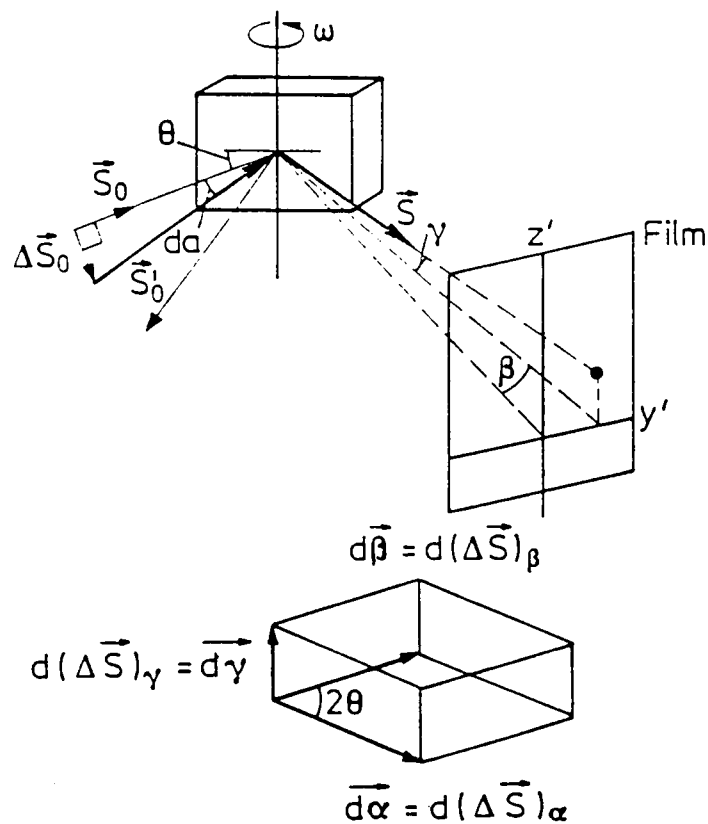


**Fig.2.9.** The range of incident x-rays is the angle  $d\alpha$ . The angles  $d\beta$  and  $d\gamma$  define the position of a diffracted beam on the detecting plane. The three angles  $d\alpha, d\beta, d\gamma$  define a volume in reciprocal lattice.

the point from the origin so that we can approximate a spherical layer as a tangential layer. Therefore,

$$\left| \frac{\mathbf{s}-\mathbf{s}_0}{\lambda} \right| = \frac{2\sin\theta}{\lambda} = |h_1\mathbf{b}_1 + h_2\mathbf{b}_2 + h_3\mathbf{b}_3| \rightarrow h_3|\mathbf{b}_3|$$

$$dh_3 = \frac{\cos\theta d(2\theta)}{\lambda(\mathbf{b}_3)} \quad \text{Eq.2.3.42}$$

Actually we measure the distribution of power per unit length of diffraction line  $P'(2\theta)$ , where

$$P'(2\theta) = \frac{P_{2\theta}}{2\pi R \sin 2\theta} \quad \text{Eq.2.3.43}$$

$$\text{Let } K(\theta) = \frac{MR\lambda^2 F^2}{16\pi v a |\mathbf{b}_3| \sin^2\theta} \quad \text{Eq.2.3.44}$$

The observable distribution of power is then:

$$P'(2\theta) = k(\theta) \int \int \sum_{m'm'} \exp[2\pi i \{ h_1(m_1' - m_1) + h_2(m_2' - m_2) + h_3(m_3' - m_3) + \frac{\mathbf{s}-\mathbf{s}_0}{\lambda}(\delta m' - \delta m) \}] dh_1 dh_2 \quad \text{Eq.2.3.45}$$

Representing the displacement  $\delta m$  by its components,

$$\delta m = Xma_1 + Yma_2 + Zma_3 \quad (m=m_1, m_2, m_3), \quad \text{Eq.2.3.46}$$

where  $m_1$ ,  $m_2$  and  $m_3$  are the numbers of the unit cell along  $a_1$ ,  $a_2$  and  $a_3$  directions, and, for calculating the scalar product  $(\mathbf{s}-\mathbf{s}_0)(\delta m' - \delta m)$ , we use the average position of the diffraction vector for the 001 reflection

$$\left\langle \frac{\mathbf{s}-\mathbf{s}_0}{\lambda} \right\rangle_{av} = l\mathbf{b}_3. \quad \text{Eq.2.3.47}$$

$$\frac{\mathbf{s}-\mathbf{s}_0}{\lambda}(\delta m' - \delta m) = l(Zm - Zm'). \quad \text{Eq.2.3.48}$$

Integrating with respect to  $dh_1$  and  $dh_2$  from  $-1/2$  to  $1/2$ ,

$$P'(2\theta) = k(\theta) \sum_{m_1} \sum_{m_2} \sum_{m_1'} \sum_{m_2'} \sum_{m'} \sum_{m_3'} \frac{\sin\pi(m_1 - m_1')}{\pi(m_1 - m_1')} \frac{\sin\pi(m_2 - m_2')}{\pi(m_2 - m_2')} \exp[2\pi i l (Zm - Zm')] \exp[2\pi i h_3 (m_3 - m_3')] \quad \text{Eq.2.3.49}$$

Except for  $m_1=m_1'$  and  $m_2 = m_2'$ , sine terms are zero. This means that we should think of the crystal in terms of columns of the cells, the columns being parallel to  $a_3$  or perpendicular to the reflecting plane 001. The summation with respect to  $m_3$  and  $m_3'$  is carried out to  $m_1$  and  $m_2$  means summation of the contributions from each column;

We now let

$$n=m_3-m_3'; \quad Z_n = Z(m_3)-Z(m_3')$$

and  $N_n(m_1m_2)$  be the number of cells with an  $n$ th neighbor in same column. Then Eq.2.3.49 becomes

$$P'(2\theta) = k(\theta) \sum_{m_1} \sum_{m_2} \sum_n N_n \langle \exp[2\pi i l Z_n] \rangle \exp[2\pi i n h_3] \quad \text{Eq.2.3.50}$$

The summation over  $m_1$  and  $m_2$  gives the number of columns  $N_1N_2$ .  $N_3$  is the average number of cells in the coherently diffracting domain. If the  $M$  in Eq.2.3.45 is the number of coherently diffracting domains in the sample and  $N$ ,  $N_3$  and  $N_n$  be averages over all the domains in the sample, Eq.2.3.45 can be expressed in the form of Fourier Series since  $Z_{-n} = -Z_n$ :

$$P'(2\theta) = k(\theta) \sum_{-\infty}^{\infty} \{A_n \cos 2\pi n h_3 + B_n \sin 2\pi n h_3\},$$

$$\text{where } A_n = \frac{N_n}{N_3} \langle \cos 2\pi l Z_n \rangle$$

$$B_n = -\frac{N_n}{N_3} \langle \sin 2\pi l Z_n \rangle. \quad \text{Eq.2.3.51}$$

We note that Eq.2.3.51 is the one dimensional Fourier analysis formula because we record power vs.  $2\theta$  which can be converted to  $h_3'$ . The coefficient  $A_n$ ,  $B_n$  are for the pure broadening. The average  $\langle \cos 2\pi l Z_n \rangle$  and  $\langle \sin 2\pi l Z_n \rangle$  are

averages over pairs of  $n^{\text{th}}$  neighbors in all columns in the sample. If positive and negative values of  $Z_n$  are equally possible for a given value of  $n$ , the sine coefficients will contribute to the line asymmetry and/or displacement. Assuming that there is no stacking fault possibility in cold worked specimen, we can use the cosine coefficient for separating the crystallite size and microstrain from pure broadened line.

#### B. Separation of size and distortion coefficient

We can represent  $A_n$  as the product of a particle size coefficient and a distortion coefficient,

$$A_n = A_n^S A_n^D \quad \text{Eq.2.3.52}$$

where

$$A_n^S = \frac{N_3}{N_n}, \quad A_n^D = \langle \cos 2\pi l Z_n \rangle \quad \text{Eq.2.3.53}$$

On the assumption that  $l$  and  $n$  are sufficiently small that the product  $l Z_n$  is also small,

$$\langle \cos 2\pi l Z_n \rangle \rightarrow 1 - 2\pi^2 l^2 \langle Z_n^2 \rangle \quad \text{Eq.2.3.54}$$

and in logarithmic form,

$$\ln \langle \cos 2\pi l Z_n \rangle = \ln(1 - 2\pi^2 l^2 \langle Z_n^2 \rangle) = -2\pi^2 l^2 \langle Z_n^2 \rangle \quad \text{Eq.2.3.55}$$

Then, Eq.2.3.55 can be written in logarithmic form:

$$\ln A_n(l) = \ln A_n^S - 2\pi^2 l^2 \langle Z_n^2 \rangle. \quad \text{Eq.2.3.56}$$

Now  $\ln A_n(l)$  can be plotted against  $l^2$  for any fixed  $n$  value. The  $l^2$  zero intercepts give the size coefficient  $A_n^S$  and we can get  $\langle Z_n^2 \rangle$  from initial slope. Since  $a_3 Z_n$  is the change in length of a column of length  $a_3 n$ , average component of strain

in the direction of the column is given  $\epsilon_L = Z_n/n$ . Thus  $\langle Z_n^2 \rangle$  can be replaced by  $n^2 \langle \epsilon_L^2 \rangle$  and then mean squared strain value ( $\langle \epsilon_L^2 \rangle$ ) is obtained. This mean squared strain is a component normal to the reflecting planes, averaged over the length squared, and averaged over all regions in the sample. If there are no strains, only small particles in the sample,  $\ln A_l$  vs.  $l^2(h^2+k^2+l^2$  in the cubic case) for each  $n$  will be horizontal line. If there is no particle size broadening but only strains in the sample, all the lines for various  $n$  intersect at  $n=0$  at  $\ln A_n(l)=0$ . We need to note that we obtain  $\langle \epsilon_L^2 \rangle^{1/2}$  from the cosine coefficients, not the average strain. From the Eq.2.3.56, we can know that only two orders of a peak are needed to separate the effects of particle size and RMS strain, and obtain  $N_n/N_{h3}$  and  $\langle \epsilon_L^2 \rangle^{1/2}$  vs.  $L$ . If the plot of  $N_n/N_{h3}$  vs.  $n$  is normalized so that its intercept at  $L=0$  is unity, the slope gives the average value ( $Deff$ ) of the length of the columns normal to the diffraction planes. This means we can determine the effective domain size in that direction. Often there is a small bend or hook in the data near  $n=0$ . This occurs from the difficulty in determining background of a broad peak. Therefore, as  $A_0$  is the area of the peak, its value will be too small. The values from beyond  $n=2-4$  should be extrapolated to  $n=0$  for normalization. As  $\ln(N_n/N_{h3})$  versus  $L$  is generally linear for  $n>2$ , such a plot is a simple procedure for this extrapolation. If strains are due to dislocation, this will be justified theoretically.[46]

### 2.3.6. Determination of dislocation density

The method of Williamson and Smallman[47] can apply to determine the dislocation density under assumption of random distribution of dislocations:

$$\rho = (\rho_D \rho_s)^{1/2}, \quad \text{Eq.2.3.57}$$

where  $\rho_D = 3/(\Delta\epsilon)^2$  is obtained from Fourier coefficient and  $\rho_s = \frac{k \langle \epsilon_L^2 \rangle}{b^2}$ ,  $k=6\pi$ , is from Gaussian strain distribution, and  $b = \frac{a}{\sqrt{2}}$  is the modulus of the Burger's vector, which is in the [110] direction for the fcc structure.

### 2.3.7. Centroid and variance method for the line broadening[48]

The centroid, or center of gravity of the X-ray line is defined by

$$\langle 2\theta \rangle = \frac{\int 2\theta I(2\theta) d(2\theta)}{\int I(2\theta) d(2\theta)}. \quad \text{Eq.2.3.58}$$

The variance, mathematically most useful in measuring the line breadth, is directly associated with the centroid  $\langle 2\theta \rangle$  as a measure of line position and defined by

$$W_{2\theta} = \langle (2\theta - \langle 2\theta \rangle)^2 \rangle = \frac{\int (2\theta - \langle 2\theta \rangle)^2 I(2\theta) d(2\theta)}{\int I(2\theta) d(2\theta)}. \quad \text{Eq.2.3.59}$$

## 2.4 Fundamentals of cold rolling

Rolling is the plastic deformation process of metal by passing it between rolls. In deforming metal by rolling, the metal is subjected to high compressive stresses from the squeezing action of the rolls and to surface shear stresses as a result of the friction between the rolls and the metal. These are a radial force,  $P_r$ , and a tangential friction force,  $F$  (Fig.2.10). The component  $P_r$  is known as the rolling  $P$ . The rolling load is the force with which the rolls press against the metal. The frictional force  $F$  is the force to draw the metal into the rolls. The angle  $\alpha$  between the entrance plane and centerline of the rolls is called the angle of contact, or angle of bite. The horizontal component of the normal force is  $P_r \sin \alpha$ , and the horizontal component of the friction force is  $F \cos \alpha$ . The limiting condition for entering metals into the rolls without any aid is

$$F \cos \alpha = P_r \sin \alpha.$$

$$\text{Thus } \frac{F}{P_r} = \frac{\sin \alpha}{\cos \alpha} = \tan \alpha = \mu.$$

Therefore,  $F = \mu P_r$  [49].

Eq.2.4.1

If  $\mu=0$ , rolling can not occur. As  $\mu$  increase progressively, larger thicken metals for rolling will be drawn into the roll throat. For the same friction condition, a large diameter roll permits a thicker metal to enter the rolls than a small

diameter roll. This is because the angle subtended from the center of the roll of the entrance plane will be the same in both cases ( $\tan \alpha$ ) but the lengths of the arcs of contacts will be appreciably different.

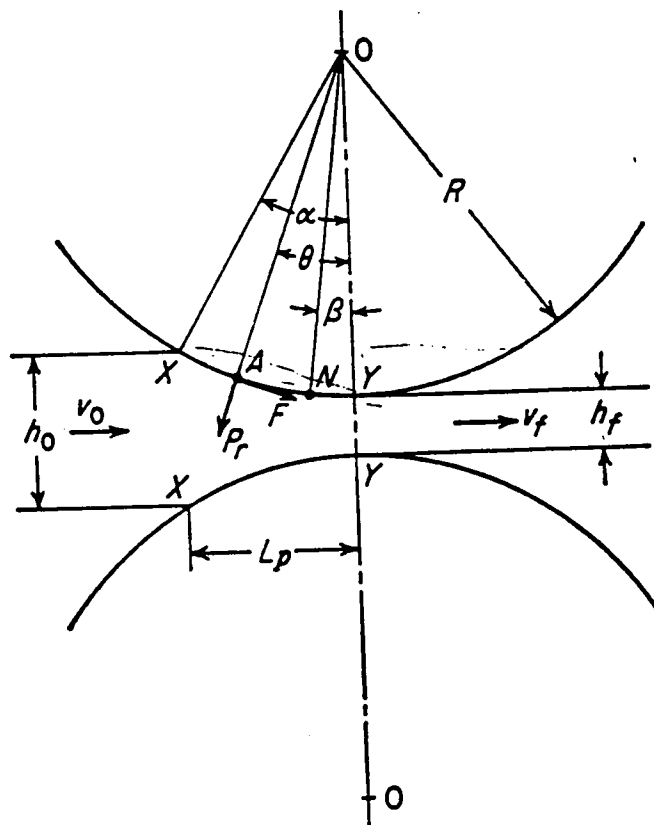


**Fig.2.10.** Forces acting during rolling.

## 2.5 Laser processing

### 2.5.1 Fundamentals of laser

Laser is a contraction of 'light Amplification by Stimulated Emission of Radiation', the process that takes place inside the device. The laser is simply stated as a beam of light which is so concentrated and powerful that it could produce power densities millions of times as intense as those on the surface of the sun.

The fundamental theory of lasers can be traced back to atomic theories by Einstein in 1917, who pointed out that controlled radiation could be obtained from an atom under certain conditions. Until the 1950s, lasers were only theory, but many scientists were working for reality. Laser history actually begins with the maser (microwave amplification by stimulated emission of radiation). In 1954, Dr. Charles H. Townes, who worked in Bell Lab., demonstrated an ammonia gas maser with adequate stimulation to sustain a maser beam of a signal equal to 24,000 megahertz. It was later discovered, in 1959, that masers could amplify light as well as microwaves, although the extension of maser principles to permit operation in the light spectrum was suggested as early as 1953. This development referred to as optical maser, with the name changed to laser in 1965. The pulsed ruby laser, built in 1960, was the first to demonstrate the monochromatic and coherent light principle.

The principle types of lasers[50] include solid state lasers(for both pulsed and continuous wave operation); gas lasers(for both pulsed and cw operation); and liquid, plastic, and Raman lasers. Each type uses various materials.

### 2.5.2 Excimer laser

Among the various lasers the excimer laser, a kind of gas lasers, is used in this experiment. The word excimer is a contraction of excited dimer, by which is meant a diatomic molecule which, whilst being stable in its excited state, is not so in its ground state. Typical of such materials are the rare gas halides such as ArF, KrF and XeCl. If we draw a diagram of energy versus atomic separation for the ground state of a normal molecule, we obtain a curve with an energy minimum occurring at the equilibrium separation of the molecule ( Fig.2.11) [51]. For example, in XeCl the following process can take place. Firstly an electron is attached, as represented by



The negative ions thus formed can then combine with positive ions,



that are present to give an excited molecule. Because of the low population in the ground state, population inversion is easily achieved. Furthermore, even when lasing commences the ground state population remains low because the ground state

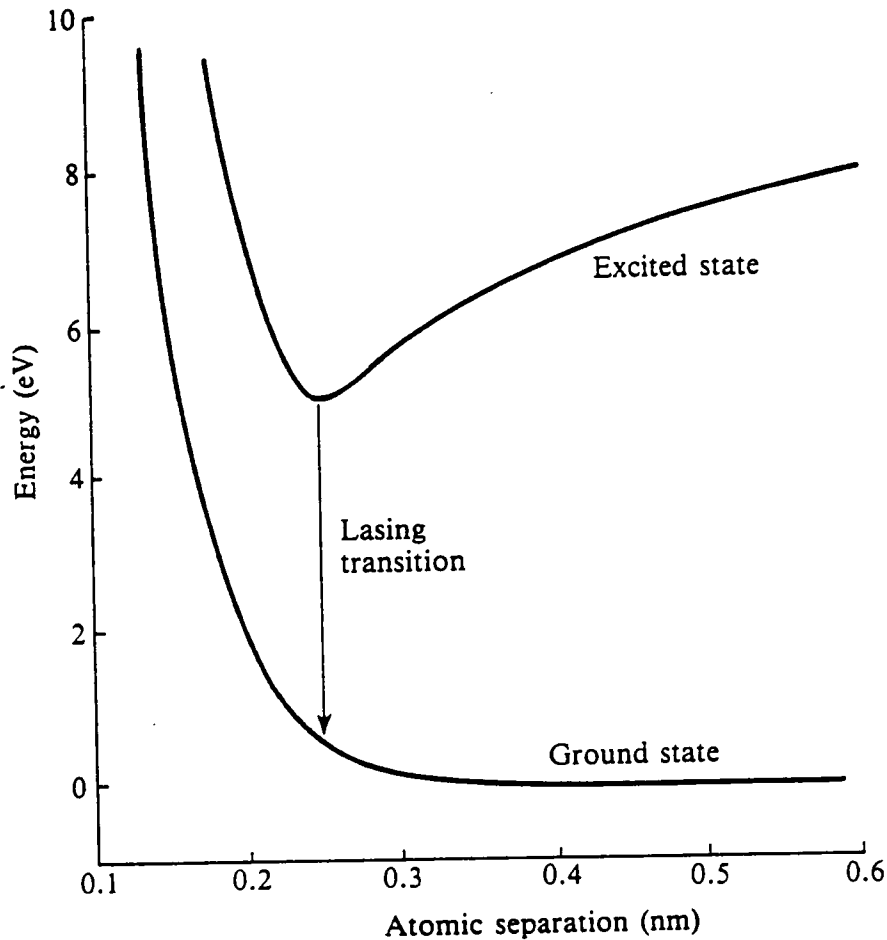


**Fig.2.11.**Potential-energy curves for the KrF excimer laser.

is inherently unstable. Once a molecule is in that state the atoms move rapidly apart and the molecule dissociates.

The beam energy per pulse per unit area normal to the beam (energy density) generated by an excimer laser is in the range of hundreds of millijoules. The initial energy density can be increased by focusing the pulse with an appropriate

system of lenses. Energy densities of more than  $10\text{J}/\text{cm}^2$  can be concentrated in small regions. The pulse duration is in the range of tens of nanoseconds and the power density can reach hundreds of megawatts. Large areas can be scanned if the work piece is mounted on the x-y translational stage. Excimer lasers operate in the ultraviolet range (UV) and the wavelength of the laser radiation depends on the gas that fills the laser cavity [52]. The energy of a nearly coherent beam incident on a material is partly absorbed by the electrons as a result of electron-photon interactions [53]. In particular in metallic materials having a high density of conduction electrons, laser light is absorbed within the first few tens of nanometers [54]. This energy is transferred as heat to the lattice very shortly ( $10^{-11} - 10^{-12}\text{s}$ ) [55]. Thus, the laser can be used as a surface heating sources.

The light is in the visible range portion. However, in the UV range the reflectivity of metals decreases considerably. For example, only 10 to 30 percent of the incident UV light is reflected by a polished copper surface, while the remaining portion of the light is absorbed in the metal within the first twenty nanometers [56, 57].

Due to recent advances in laser technology, which include increases in laser power and repetition rate, these lasers are nowadays widely used as industrial processing tools.

### 2.5.3 Thermal analysis of the process

Excimer laser heating can be treated as the one-dimensional heat diffusion problem because the irradiated area is of the order of several millimeters, while the heat affected region is only a few micrometers thick.

$$c \cdot \rho \cdot \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left( K \frac{\partial T}{\partial x} \right) + P(x, t) \quad \text{Eq.2.5.3}$$

where  $c$ ,  $\rho$  and  $K$  are the sample specific heat, density and thermal conductivity,  $P(x, t)$  represents the source term (a laser pulse). The simplest and most obvious boundary conditions refer to the initial sample temperature, the limiting temperature down in the bulk depth and the radiative loss at the surface, i.e., respectively

$$T(x, 0) = T_0 ; T(\infty, t) = T_0 ; \left. \frac{dT}{dx} \right|_{x=0} = 0 \quad \text{Eq.2.5.4}$$

where  $T_0$  is the initial temperature of the metal. In the case of melting or resolidification the condition (x-axis directed toward the sample interior);

$$\rho \Delta H_{\text{fus}} X = K_s \left. \frac{dT}{dx} \right|_{x^+(s,l)} - K_l \left. \frac{dT}{dx} \right|_{x^-(l,s)} \quad \text{Eq.2.5.5}$$

must be satisfied at the melt-solid interface. Here  $\Delta H_f$  is the heat of fusion,  $X$  is the moving rate of the interface at time  $t$ , and  $K_l$  and  $K_s$  are, respectively, the thermal conductivities of liquid and solid. Moreover,

$$X = f(T_i - T_m), \quad \text{Eq.2.5.6}$$

where  $f$  is a function of the difference between the solid-liquid interface temperature  $T_i$  and the equilibrium melting temperature  $T_m$ . The actual form of  $f$  is determined by the

crystal melting program has been developed to calculate the heat evolution during laser processing [58].

## **2.6 Phase transformation in austenitic stainless steel[59]**

### 2.6.1 Phase transformation in pure iron

Iron changes from the body-centered cubic alpha phase (ferrite) to the face-centered cubic gamma phase (austenite) at 910°C (known as the A3 temperature) and back to the body-centered cubic phase (delta ferrite) at 1400°C (the A4 temperature).

### 2.6.2 Chromium addition effect

The addition of chromium reduces the range over which the face centered cubic phase is stable; the A4 temperature drops very rapidly, and the A3 temperature, after an initial lowering, rises to meet the A4 temperature. From Fig.2.12, we see that if the chromium content exceeds about 12%, no austenitic structure can be produced at any temperature; the possibilities of quenching to produce martensite are removed(no phase transformation). That is to say, if the chromium content exceeds the limit of the gamma and gamma-plus-alpha fields, the alloy will, at all temperatures to its melting point, be body-centered cubic. Such an alloy is

therefore known as ferritic; it can not go through a martensitic transformation because it can not change its structure.

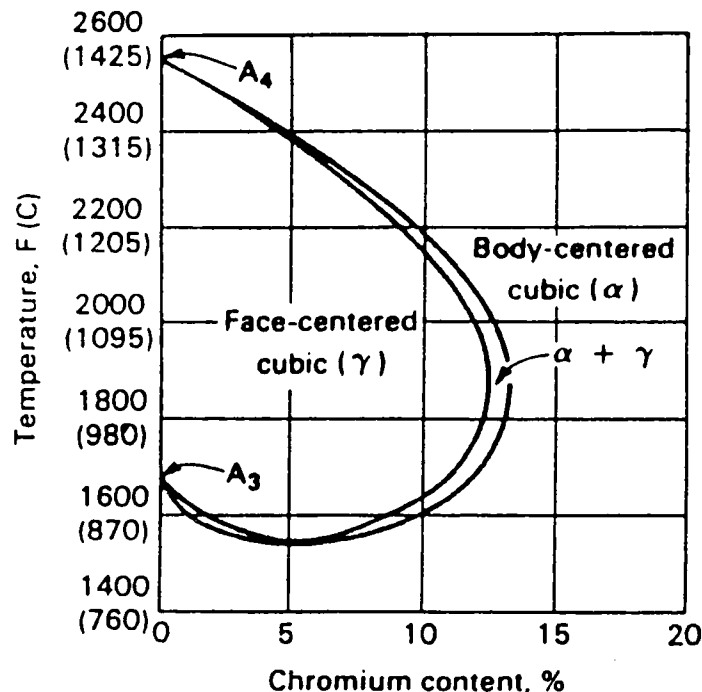


**Fig.2.12.** Partial iron chromium phase diagram.

### 2.6.3 Nickel addition effect

The effect of nickel on iron is to open up the range of stable austenite. The austenite region in an alloy of iron with 18% chromium and varying amounts of nickel is shown in the Fig.2.13. This diagram implies that at low temperatures even alloys containing quite high nickel contents will transform to ferrite. However, the transformation is so sluggish that austenite persists on cooling the alloy from

the austenite region to room temperature (and usually to subzero temperatures).

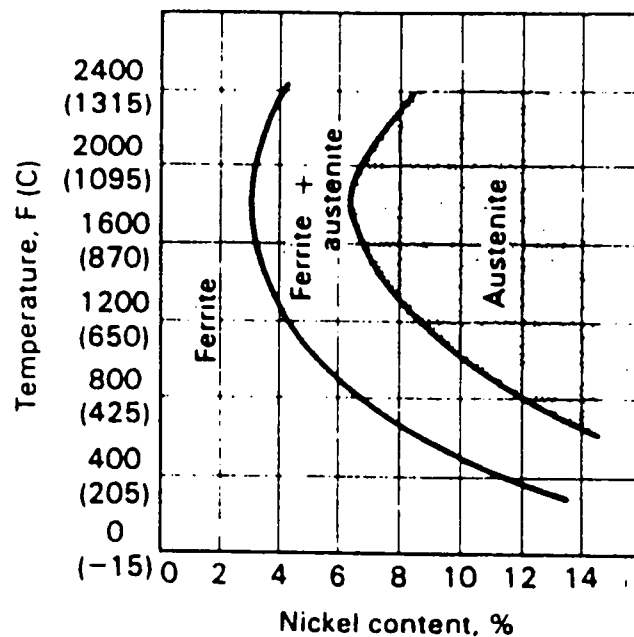


**Fig.2.13.** Effect of nickel content on the stability of austenite in an iron alloy containing 18% chromium.

#### 2.6.4 The phase of the austenitic stainless steel at room temperature

The equilibrium phase of the austenitic stainless steel (18Cr-8Ni) at room temperature is body centered cubic

phase. However, Fig.2.14 shows the composition of iron-chromium-nickel alloys (zero carbon) for which austenite is persistent at room temperature. Austenitic alloys whose composition is just sufficient to ensure the austenitic structure at room temperature (at point P at Fig.2.14) may partially transform as a result of cold work. The transformation is similar to martensitic process and the steel is substantially hardened.

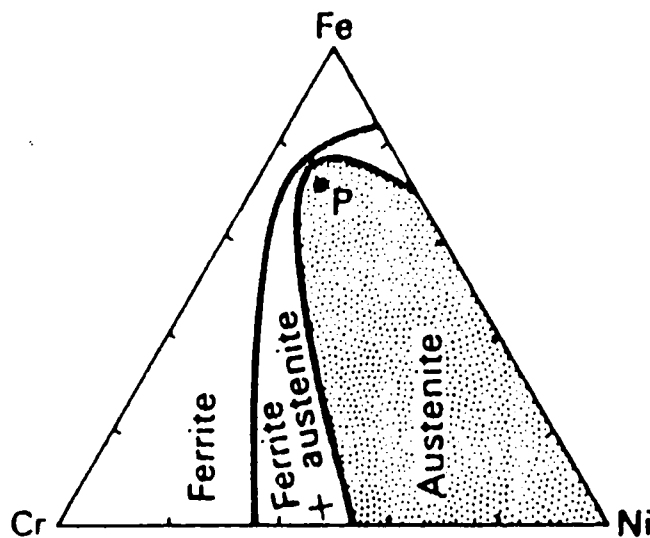


**Fig.2.14** Compositions of iron-chromium-nickel alloys for which austenite persists in room temperature. Point P indicates the position of an alloy containing 18% chromium and 8% nickel.

## CHAPTER 3

### SPECIMEN PREPARATION AND EXPERIMENTAL PROCEDURE

#### 3.1 Specimen preparation

##### 3.1.1 For residual stress analysis

316L Stainless steel was used for analysis. All specimens were machined to the approximate dimensions 2cm x 3.5cm x 0.15cm (These specimens were chosen to enable the measurement with Regaku Geiger Counter X-ray Diffractometer.), and stress-relief annealed at 1050°C for 15 minutes. After annealing, each specimen was electro-polished to remove the surface oxide layer (electrolyte; 50% sulfuric acid in the water). Then, three specimens were cold-rolled respectively to 20%, 40% and 60% reduction rates. One of the annealed specimens was taken as a standard specimen to determine stress-free d-spacing( $d_0$ ) and instrumental broadening. Then, the specimens were analyzed by x-ray diffraction to determine the macro- and micro-residual stresses.

Subsequently, all the specimens were laser-irradiated with various energy densities. Annealed specimens were laser-irradiated with the energy densities of 0.5J/cm<sup>2</sup>, 1J/cm<sup>2</sup>, 2J/cm<sup>2</sup>, 3J/cm<sup>2</sup>, 3.5J/cm<sup>2</sup> and 4J/cm<sup>2</sup>. Cold rolled specimens were laser-irradiated with energy densities of 4J/cm<sup>2</sup> and 3J/cm<sup>2</sup>. Then, X-ray diffraction process was repeated on the

laser treated specimens under the same diffraction conditions. A pulsed XeCl laser was used in the laser processing.

### 3.1.2 For melting depth measurement

One of the specimens laser-treated with the energy density of 4J/cm<sup>2</sup> was prepared to measure the melting depth metallographically. The specimen was cut and was electroplated with copper, and then hot-embedded. During hot-embedding, the specimen was fixed with holders made by coiling the sheet metals in order to hold it vertically and ball bearings were positioned around the specimen. Then the exposed areas of ball bearings during grinding and polishing were controlled in order to reduce out-of-profile effects on the specimen. Also, this preparation was aimed to reduce the edge rounding effect during grinding and polishing. After mechanical-polishing, the metallographic surface was electro-etched with 10% oxalic acid. The melting depth profile was measured with scanning electron microscopy and compared with the calculated values from the mainframe VAX cluster.

The melting depth values both measured and calculated were also compared with the X-ray penetration depths to check if X-ray could measure the residual stress covering the sufficient depth of laser affected zone.

### 3.1.3 For dislocation observation

To observe the dislocations induced during laser irradiation, specimens were prepared for TEM (Transmission Electron Microscopy). The purpose of the TEM experiment was to verify where dislocations are mainly formed, i.e., in the melting region or laser affected zone.

In order to observe the dislocations in the melted layer, micro-shield material was painted on the laser irradiated surface of the specimen and then jet polished with Tenupol-III manufactured by Struers Company.

For the observation of the dislocations in the laser affected zone, the melted layer of the specimen was removed with ion-milling machine to expose the laser affected zone. The removed side was then painted with micro-shielding material, and then jet-polished. Controlling the removal depth was performed by weighing with micro-balance. Electrolyte used was 12.5%vol. sulfuric acid in methanol [60].

After finishing the jet-polishing, the micro-shield material was removed with acetone and the specimen was further cleaned in pure ethanol.

### 3.1.4 For phase formation study of the austenitic stainless steel

316L stainless steel specimens were prepared and annealed at 1050°C for 15 minutes. Two specimens were

immersed in 10% hydro-chloric acid for 10 minutes before deposition so that specimen's surface could be cleaned and the passive film broken. After taking out the specimen from 10% HCl solution, one specimen was washed in ethanol and then placed immediately in the vacuum chamber for sputtering to prevent repassivation, while the other specimen was exposed in air for approximately 20min. to be repassivated. The surface of one substrate was ground after annealing.

The 316L stainless steel deposition was carried out in an Edward's sputtering apparatus.

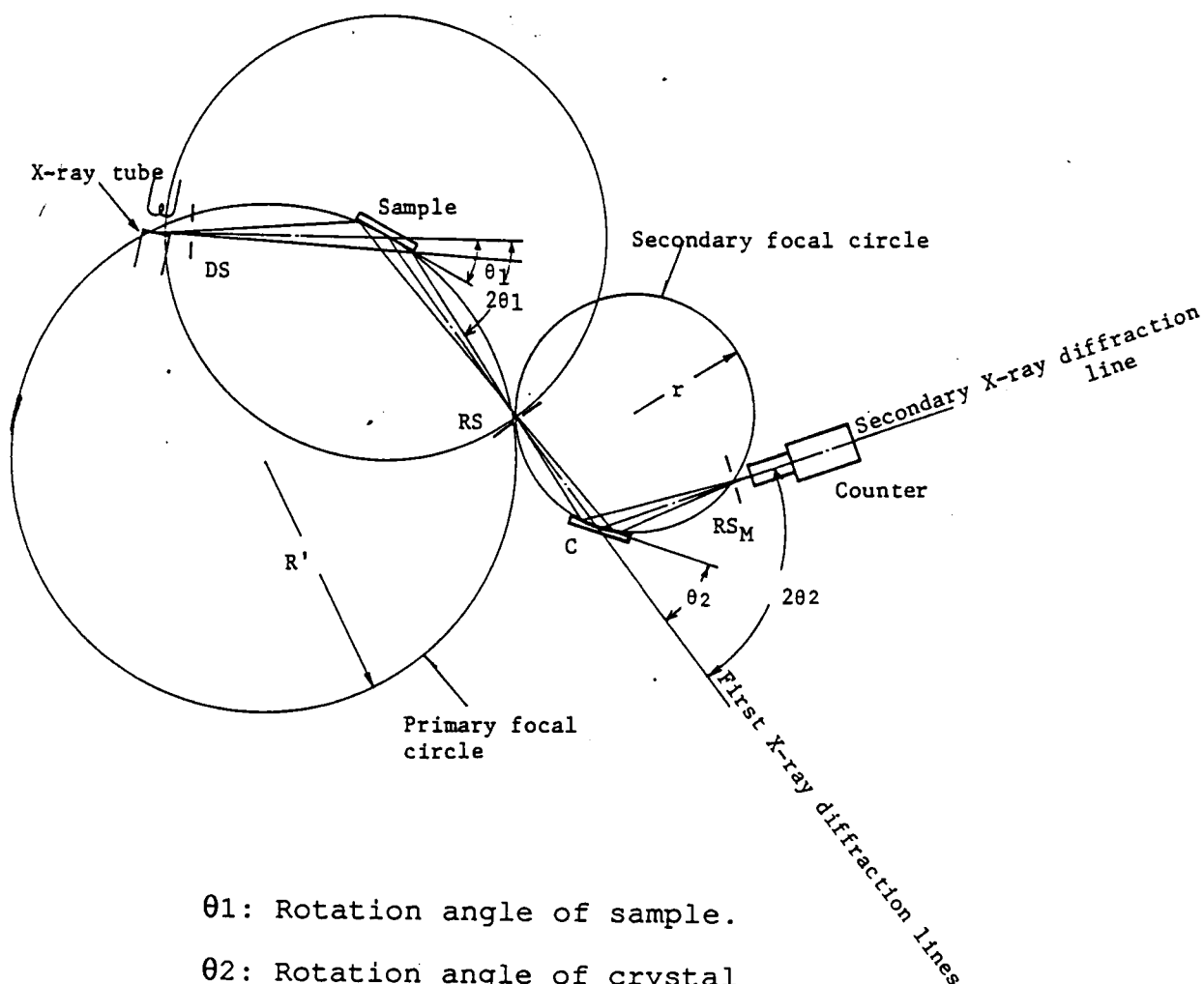
X-ray diffraction was performed on all the specimen before and after sputter deposition.

### **3.2 X-ray diffraction condition**

#### **3.2.1 Diffractometer geometry and operation principles**

The diffractometer, which is used in this experiment, is the most common apparatus used for examining diffraction patterns. It utilizes electronic counters sensitive to X-ray photons, mechanical rotation devices to rotate the specimen, and a detector to facilitate real time measurement of X-ray intensities. The geometry of a diffractometer is shown schematically in Fig.3.1. X-rays from a tube impinge on the specimen that is positioned on a holder that can rotate around an axis perpendicular to the plane of the

diffractometer. The diffracted beam is then detected by a suitable detector, which can be rotated around an axis, along the circumference of the diffractometer. The rotation of the specimen holder and detector are by means of stepping motors and gear drives and can be controlled very accurately. Slits are mounted in front of the tube and the detector, respectively, to collimate the beam and define the maximum "horizontal" divergent angles permissible in the incident and diffracted beams. Soller slits were used in the incident and diffracted beam paths for better vertical collimating. During a normal run with a flat polycrystalline sample the detector moves along the diffractometer circle to detect the diffracted beam. The holder is also rotated around an axis half the rotation angle of the detector in order to maintain focusing. In this way, the divergent incident beam is converged or focused at the receiving slit, because both sets of rays section equal arcs on the focusing circle. The monochromator is placed between the specimen holder and the detector. This is set to obtain a good peak to background ratio. The detection surface of a diffractometer (i.e., the surface that the receiving slit describes as it is rotated around the diffractometer center) is a cylindrical segment of radius  $R$  (the diffraction radius) and height  $l$  (the length of the receiving slit). The detector in a diffractometer sees only the intersection of the diffracted rays with this surface. The width of the receiving slit determines



$\theta_1$ : Rotation angle of sample.

$\theta_2$ : Rotation angle of crystal

$R'$ : Radius of primary focal circle (Goniometer radius of 185mm)

$r$  : Radius of the secondary focal circle  
(Curvature of crystal  $\times 1/2$ )

DS: Goniometer divergence slit

RS: Goniometer receiving slit

$RS_M$ : Monochromator receiving slit

C : Curved single crystal

**Fig.3.1** The geometry of the diffractometer with monochromator.

the accuracy with which the circumference of this cylindrical segment is measured, while the area of the slit controls the total intensity admitted into the detector. X-ray counters are devices that convert X-rays into a pulsating electric current, and the number of current pulses per unit time is proportional to the intensity of the X-rays entering the counter. All data obtained were collected in the PDP-11/34 system.

### 3.2.2 Diffraction condition

The specific diffraction conditions used in the experiment are present in Table 3.1. The diffractometer was aligned according to the equipment manual and checked by using the Cukal intensity peak of 420 reflection obtained from a fully annealed 316L stainless steel standard specimen at various  $\psi$  angles. When the deviations of intensity peak positions ( $\Delta 2\theta$ ) were within  $0.03^\circ$ , the diffractometer was considered to be in a well-aligned condition.

The specimens were mounted on the diffractometer such that the rolling axis corresponded to the direction  $\phi=0^\circ$ .

420 reflection was employed to analyze the macro residual stresses with various  $\psi$  tilt angles ( $\psi=0^\circ, 15^\circ, 30^\circ$  and  $45^\circ$ ), and 111 and 222 reflections were chosen to analyze micro-residual stresses and dislocation densities.

**Table 3.1**

Operation conditions used in X-ray diffraction

|                       |                                                 |
|-----------------------|-------------------------------------------------|
| Diffractometer        | Rigaku Geiger Count<br>Diffractometer           |
| Characteristic X-rays | CuK $\alpha$                                    |
| Monochromatization    | Curved Single crystal<br>graphite monochromator |
| Divergence angle      | 0.5°                                            |
| Preset time           | 10 seconds                                      |
| Step size             | 0.01°                                           |
| Tube voltage          | 40KV                                            |
| Tube current          | 30mA                                            |

### 3.3 Analytical procedures for residual stresses

#### 3.3.1 Background subtraction from intensity data

The background was determined by averaging the first and the last fifty intensity data for each diffraction pattern, and subtracted from each intensity value.

#### 3.3.2 Analytical procedures for macro-residual stress

##### 3.3.2.1 Least squares parabolic fitting

Intensity points which encompass 85% of the maximum intensity value were selected for parabolic fit. In determining intensity peak position ( $2\theta_p$ ) Eq.2.3.16 was used with the least squares fitting method. Detailed procedure for the least squares parabola fitting is given below [61].

Assuming that an odd number ( $2n+1$ ) of two theta ( $2\theta_j$ ) points is spaced apart at equal intervals ( $\delta$ ), Eq.2.3.16 can be written by

$$I_j = a + b\delta j + c\delta^2 j^2 \quad \text{Eq.3.3.1}$$

where  $j$  is two theta interval number with respect to the working origin,  $\delta$  is the  $2\theta$  interval between the data points.

Using least squares method the parameter,  $S$ , reaches a minimum at the best estimates for  $a$ ,  $b$  and  $c$ .  $S$  can be written as

$$S = \sum (a + b\delta j + c\delta^2 j^2 - I_j) \quad \text{Eq.3.3.2}$$

where  $n$  is from the  $2n+1$  points used in the fit. By definition the minimization occurs when the partial derivative of  $S$  with respect to each parameter is equal to zero, i.e.,

$$\partial S / \partial a = 0, \partial S / \partial b = 0 \text{ and } \partial S / \partial c = 0 . \quad \text{Eq.3.3.3}$$

Solving equations resulting from Eq.3.3.3 simultaneously, we can determine parameters  $a, b$  and  $c$ . Then, using Eq.3.6.6 the maximum for the parabola can be obtained when

$$\partial I_i / \partial 2\theta_i = 0 . \quad \text{Eq.3.3.4}$$

The solution of Eq.3.3.4 is the two theta peak position ( $2\theta_p$ ).

These procedures were programmed and calculation was done on the mainframe VAX cluster. Fig.3.2 (a) and (b) show examples of the least squares parabola fitting plots with the top 15% intensity data. The intensity peak for parabolic fit was normally  $K\alpha_1$ . However, when the intensity peak profile was so broadened due to deformation and  $K\alpha_1$  could not be distinguished from total peak, averaged  $k\alpha$  wavelength was chosen to determine d-spacing.

#### 3.3.2.2 Residual stress calculation

The residual stresses were calculated using Eq.2.3.10. The slopes were obtained from the least squares linear fitting method for all the  $d$  vs.  $\sin^2\psi$  plots. Young's modulus and Poisson's ratio ( $\nu$ ) used were 193Gpa and 0.28 respectively [62].

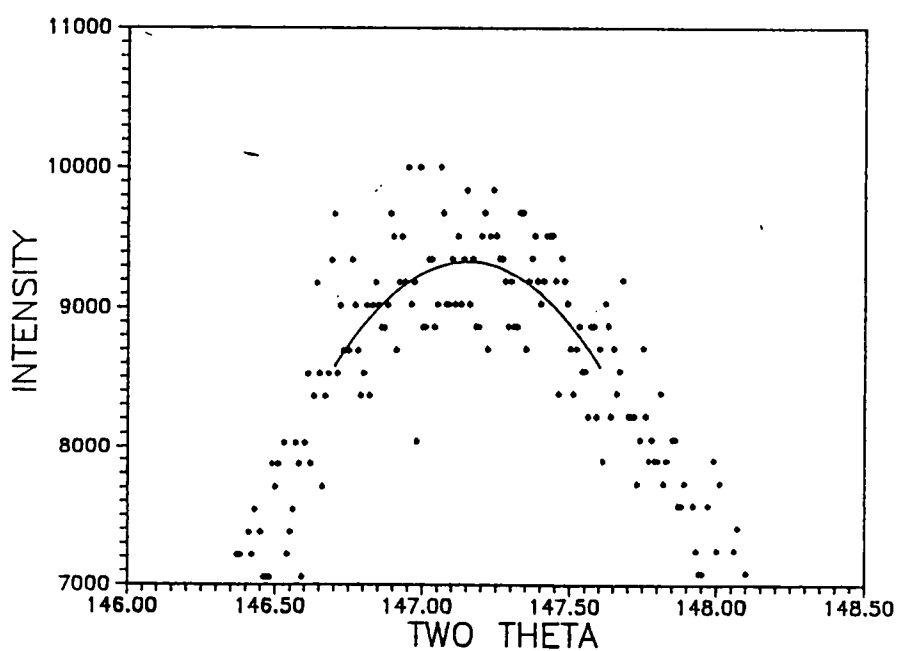
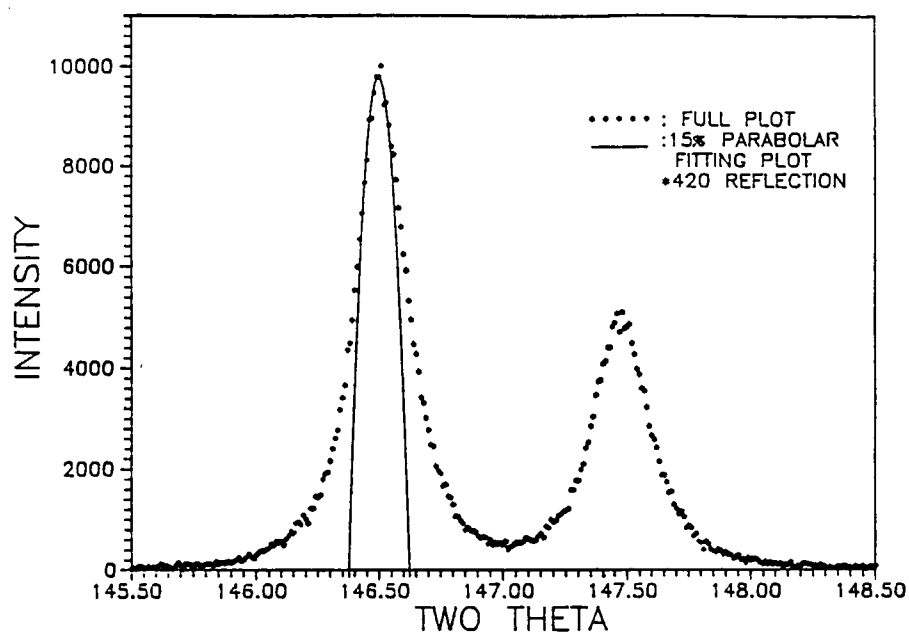


**Fig.3.2.** An example of the least squares parabolic fitting method for the top 15% of max. intensity; a) a view from a full scan, b) a view from the top 15% intensity data.

### 3.3.3 Analytical procedure for micro-residual stress

#### 3.3.3.1 Line broadening determination

The line broadening was measured using Eq.2.3.58 and Eq.2.3.59.

#### 3.3.3.2 Separation of the pure line broadening -- the method of Stoke's correction

For better Fourier analysis, the method of Rachinger correction was used to eliminate the  $K\alpha_2$  peak from the total peak because the computation can be greatly simplified if the peak profile is symmetrical. Fig.3.3 shows examples of the Rachinger correction. This method renders not only better Fourier analysis but also larger limit of the particle size to be analyzed. It is reported that the peaks to be analyzed should be at least 20% broader than the standard peaks, otherwise the analytical procedures will result in considerable scatter. This means that a particle size larger than about  $1000\text{\AA}$  can not be measured by Fourier analysis method of the total intensity peak. By eliminating  $K\alpha_2$  peak from the total peak, particle size to be analyzed can be increased to about  $2000\text{\AA}$ . The error in the particle size and in the strains are generally within 10% in this case [63].

With the  $K\alpha_1$  peak separated by the method of Rachinger correction, Fourier transformation was then performed.

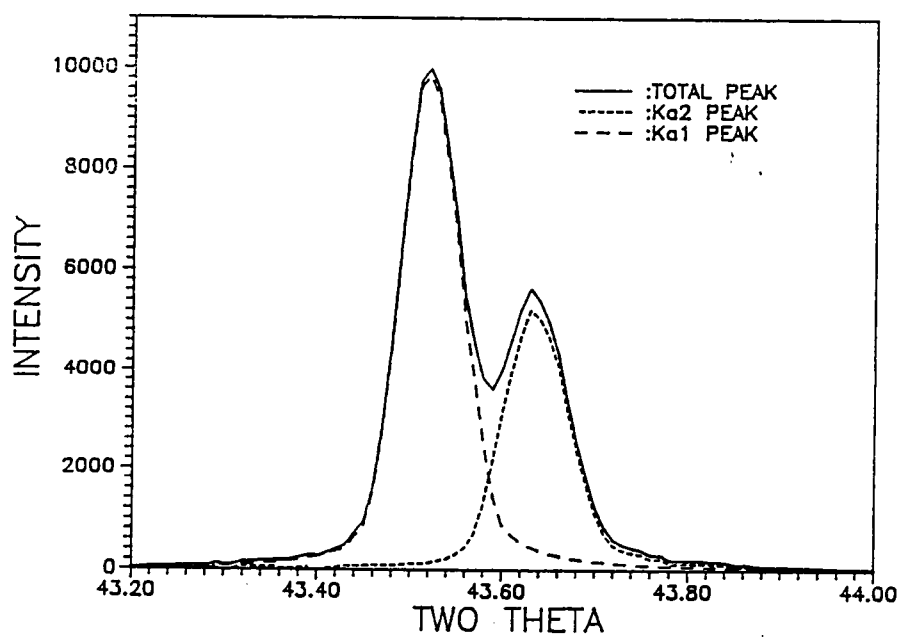
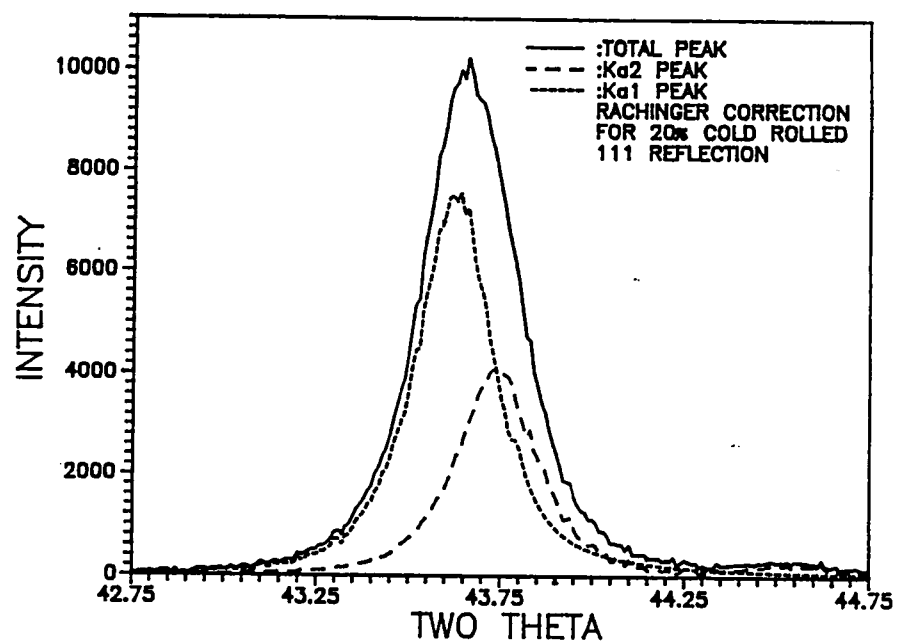


Fig.3.3. Examples of the Rachinger correction.

Fourier coefficients of any real functions ( $g(x)$  and  $h(x)$ ) were obtained by replacing the integral for these coefficients by a sum for practical evaluation on a computer. 600 intervals were used to integrate the intensity profile. The computation was performed as follows:

$$\text{For the instrumental function } g(x),$$

$$G(\zeta) = 1/a \sum g(x) \exp(2\pi i n x/a) \quad \text{Eq.3.3.5}$$

Then;

$$G(\zeta) = a/600 \times 1/a \times \sum g(t) \exp(2\pi i n t/600),$$

or

$$G(\zeta) = 1/600 \times \sum g(t) \exp(2\pi i n t/600), \quad \text{Eq.3.3.6}$$

where the sum is over integers  $t$  in interval  $a$ . Similarly, we can determine Fourier coefficient  $H(\zeta)$  for the measured function  $h(x)$ ,

$$H(\zeta) = 1/600 \sum h(t) \exp(2\pi i n t/600).$$

Since  $G(\zeta)$  and  $H(\zeta)$  consist of real and imaginary parts, we can get the Fourier cosine and sine coefficients using Eq.2.3.29. Thus, the Fourier coefficients for the series  $H_r, H_i, G_r, G_i$  are given by

$$\begin{aligned} H_r &= 1/600 \sum h(t) \cos(2\pi t n/600) \\ H_i &= 1/600 \sum h(t) \sin(2\pi t n/600) \\ G_r &= 1/600 \sum g(t) \cos(2\pi t n/600) \\ G_i &= 1/600 \sum g(t) \sin(2\pi t n/600). \end{aligned} \quad \text{Eq.3.3.7}$$

With these Fourier coefficients each line profile becomes to have its own particular function. Fig.3.4 shows examples of reconstructed intensity profiles with Fourier coefficients.

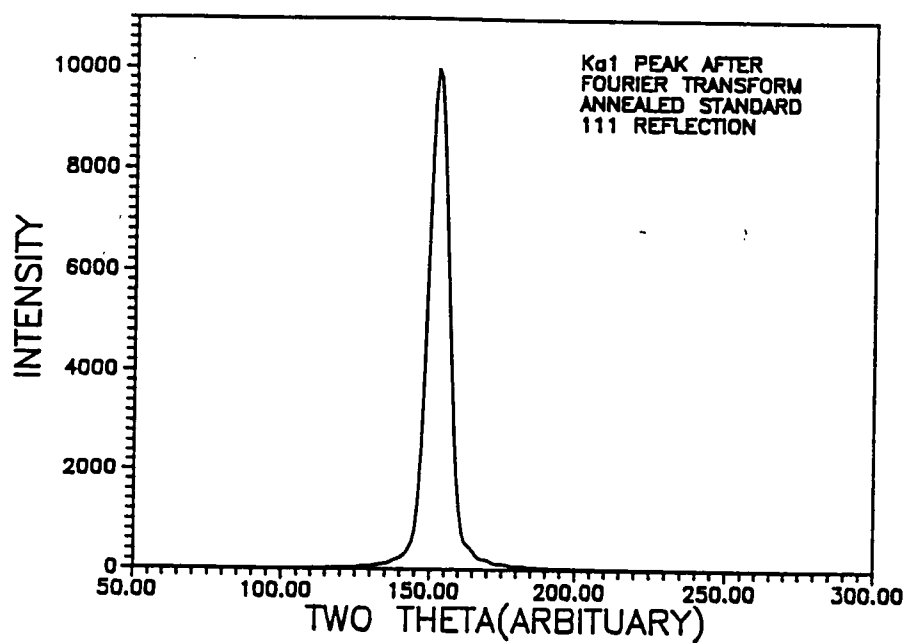
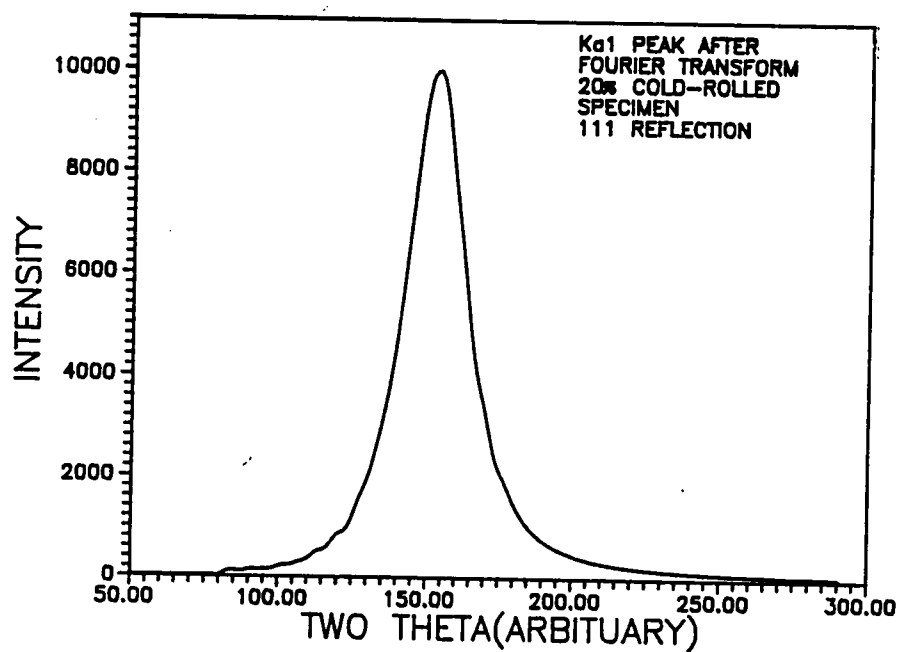


**Fig.3.4.** Examples of intensity peak profiles reconstructed using Fourier coefficients.

Consequently, from the Fourier coefficient  $G(\zeta)$  for the instrumentally broadened peak profile  $g(x)$  and the Fourier coefficients  $H(\zeta)$  for the peak profile  $h(x)$ , the corrected Fourier coefficient  $F(\zeta)$  for pure line profile can be obtained using Eq.2.3.32. Then, the pure diffraction profile  $f(x)$  can be computed as follows;

$$f(x) = 1/600 \times \sum F(\zeta) \exp(-2\pi i n t / 600) = 1/600 \times \sum F_r \cos(2\pi n t / 600) + 1/600 \times \sum F_i \sin(2\pi n t / 600). \quad \text{Eq.3.3.8}$$

Fig.3.5 to 3.6 show examples of the procedures to get the pure broadened line profiles constructed by the corrected Fourier coefficients.

### 3.3.3.3 Separation of the particle size and the micro-strain using pure broadened peak

In order to get the information about the crystallite size effect and the lattice strain effect on the line broadening the crystallite size and lattice strain were separated using the Warren's powder pattern power theorem. The computational procedures are given as follows.

From the Warren's power theorem the experimentally observed power  $P'(2\theta)$  can be expressed as a Fourier series. From the Eq.2.3.51,

$$P'(2\theta) = K(\theta) \sum (A_n \cos 2\pi n h^3 + B_n \sin 2\pi n h^3).$$

If we express the pure broadened peak profile by introducing fictitious quantities  $a_3, a_3^*, l', h_3'$ , the new angular variable is now given as

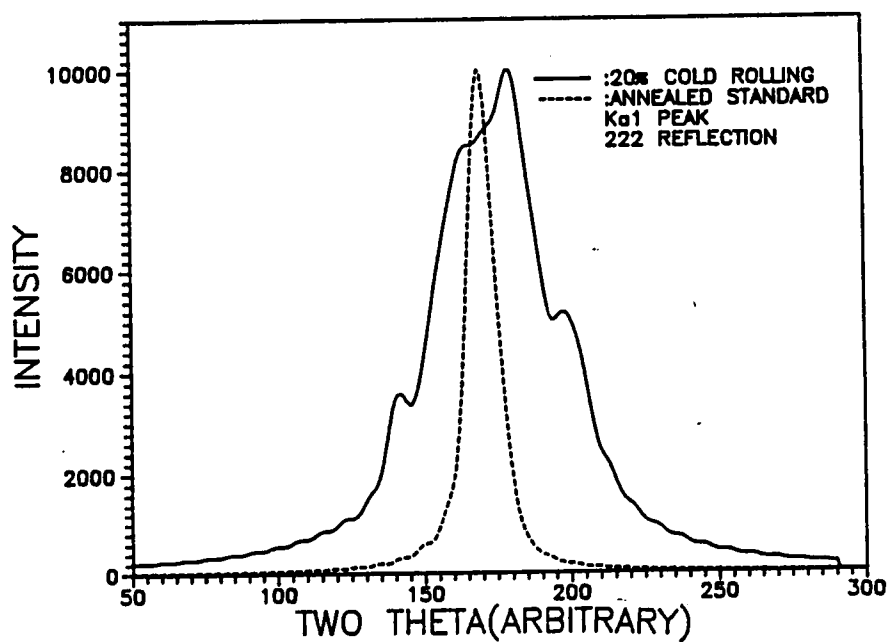
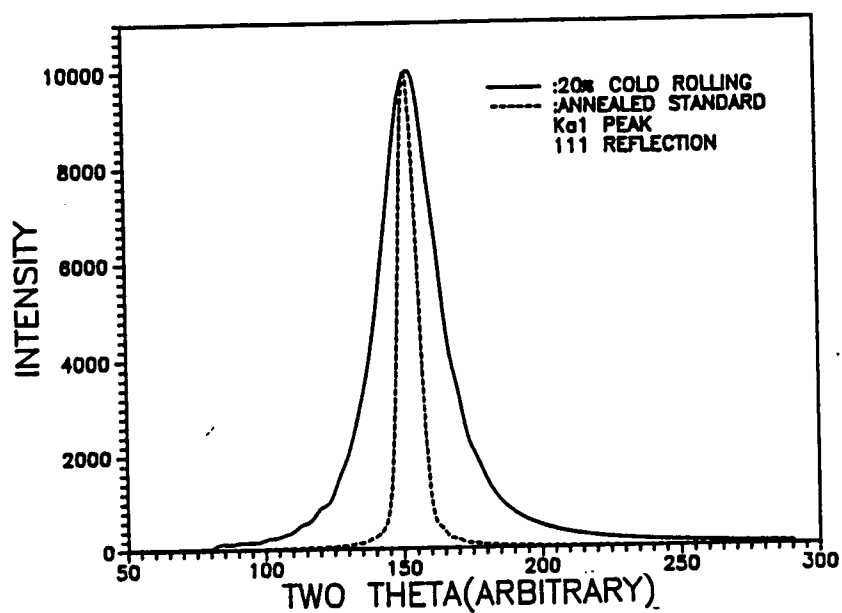


**Fig.3.5.** Comparison of experimental peak with instrumental peak, reconstructed by Fourier coefficients.

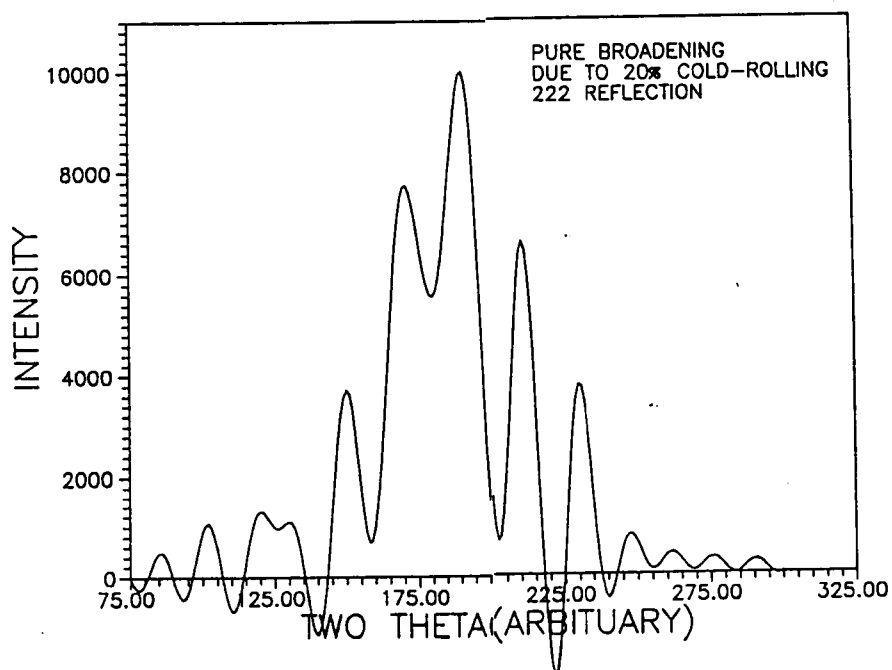
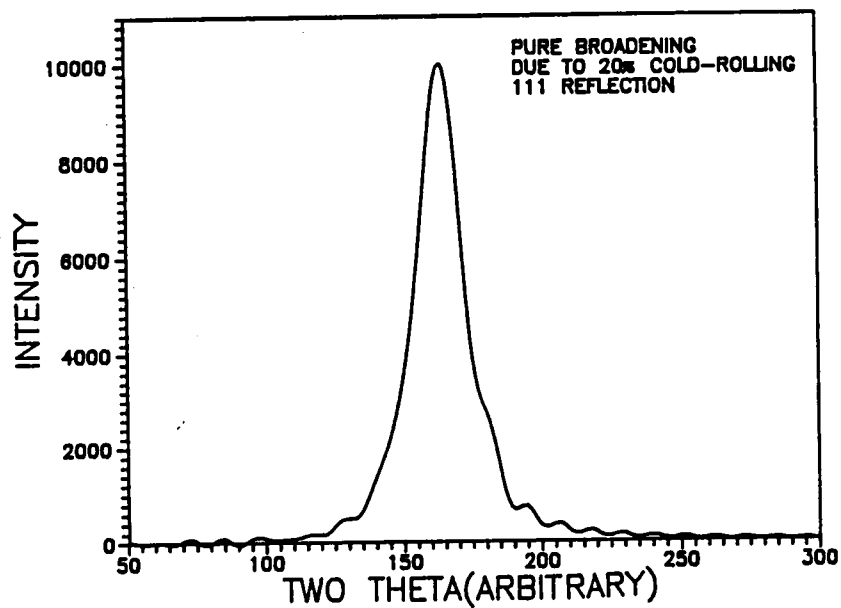


**Fig.3.6.** Examples of the pure broadened line profile constructed by corrected Fourier coefficient after Stoke's correction.

$$h_3' = 2a_3' \times \sin\theta/\lambda. \quad \text{Eq.3.3.9}$$

At the peak position,  $\theta = \theta_0$  and we let  $h_3' = l'$ . Next, a large interval in  $h_3'$ , extending from  $l' - 1/2$  to  $l' + 1/2$ , is selected to include the entire line profile sufficiently. With the angle  $\theta_1$  and  $\theta_2$  corresponding, respectively, to the limits  $l' - 1/2$  and  $l' + 1/2$ , we have

$$\begin{aligned} l' - 1/2 &= 2a_3'/\lambda \times \sin\theta_1 \\ l' &= 2a_3'/\lambda \times \sin\theta_0 \\ l' + 1/2 &= 2a_3'/\lambda \times \sin\theta_2, \end{aligned} \quad \text{Eq.3.3.10}$$

from which

$$1/2 = 2a_3'/\lambda(\sin\theta_2 - \sin\theta_1). \quad \text{Eq.3.3.11}$$

Now,  $a_3'$  can be determined from Eq.3.3.11 because  $\lambda, \theta_1$  and  $\theta_2$  are known. Since  $L = n'a_3'$  is real distance in the reflection plain normal direction, we may multiply all  $n'$  values by the value  $a_3'$  obtained from Eq.3.3.11. Rewriting Eq.2.3.56,

$$\ln A_n(l) = \ln A_n^s - 2\pi^2 h_o^2 \langle Z_n^2 \rangle.$$

Since the cosine coefficient of the corrected peak profile corresponds to  $A_n(l)$ , the size coefficient  $A_n^s$  and values of  $-2\pi^2 \langle Z_n^2 \rangle$  for the fixed value  $n$  can be determined by  $h_o^2 (= h^2 + k^2 + l^2)$  zero intercepts and the slope respectively by using the plot of the  $\ln A_n(l)$  versus  $h_o^2$ . Since the change in length of a column of length  $a_3 n$ ,  $\epsilon L = Z_n/n$ ,  $\langle Z_n^2 \rangle$  can be replaced by  $n^2 \langle \epsilon L^2 \rangle$ . Thus the initial slopes of the  $\ln A_n(l)$  versus  $h_o^2$  plots give values of  $-2\pi^2 h_o^2 n^2 \langle \epsilon L^2 \rangle$ . Then, the root mean squared (RMS) strain components  $\langle \epsilon L^2 \rangle$  can be calculated.

Fig.3.7 shows an example of the logarithmic plot for

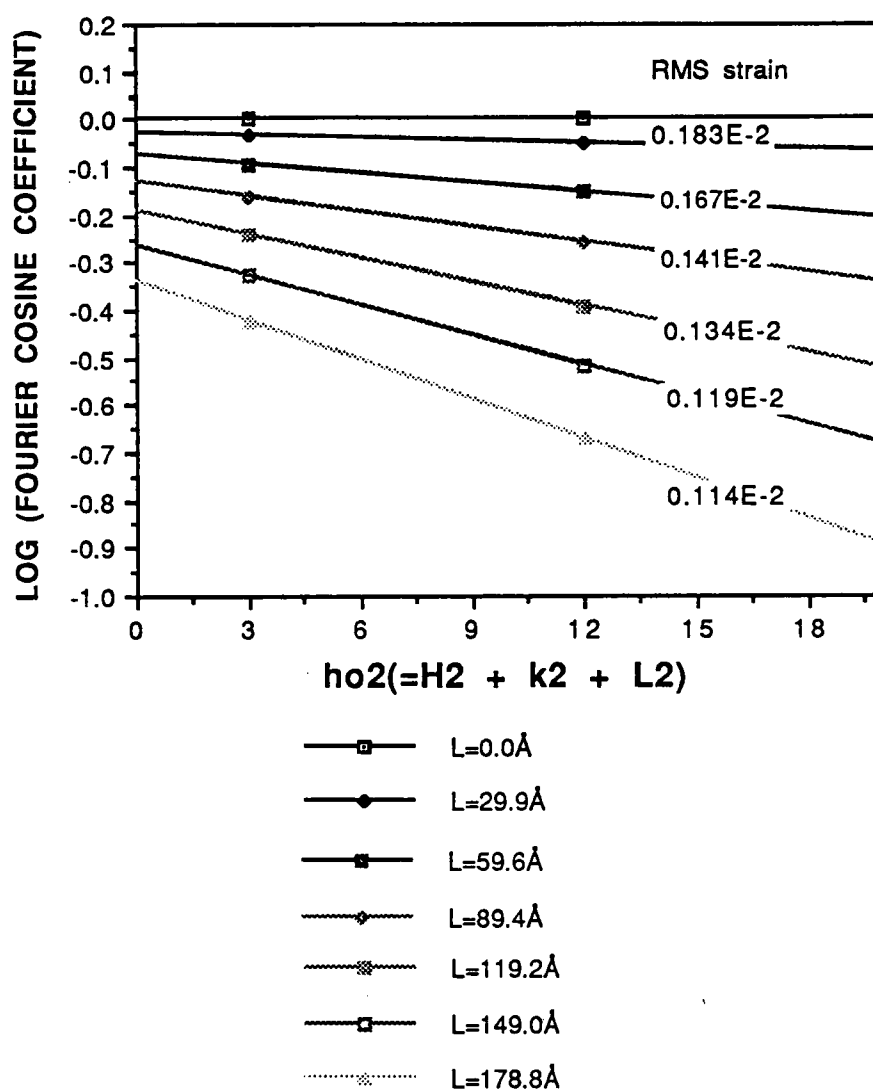


**Fig.3.7.** An example of logarithmic plot to separate the crystallite size and RMS strain using Fourier cosine coefficient. From the slope the RMS strain is obtained.

separating crystallite size and distortion effect. From this kind of plot the RMS strain is obtained. Then, by plotting the size coefficients  $AL^S$  as a function of  $L$  an effective crystal size ( $D_{eff}$ ), a composite of the true mean dimension  $D$  and the faulting effect, can be obtained from the abscissa intercepts of the initial slope. Fig.3.8 shows such an example of the plot to obtain the  $D_{eff}$ . Then Eq.2.3.57 can be used to calculate dislocation densities. The dislocation densities can be obtained from either the crystallite size or the RMS strain. Both can then be combined. Comparing these two values information about the dislocation clustering can be obtained.

### **3.4 Calculation of melting depth and laser affected zone**

In the calculation of the melting depth and thermal gradient, a computer program utilizing the finite element method (FEM) was prepared. The variables used in the calculations are below:

.Thermal conductivity(cal/m.sec.K): Melting range;26.9

Liquid(1427°C);27.24

.Density(g/cm<sup>3</sup>): Solid; 8

Melting;7

Liquid(at 2227°C);6.61

.Specific heat(J/kg.K): Solid;500

Liquid;920J/kg.K

- .Melting range: 1376°C - 1400°C
- .Boiling temperature: 2723°C(pure iron)
- .Heat of fusion: 516 cal/cm<sup>3</sup>(Pure iron)
- .Heat of evaporation:12.6x10<sup>3</sup> cal/cm<sup>3</sup>(Pure iron)
- .Reflectivity: 0.15
- .Absorption coefficient: 8x10<sup>5</sup>

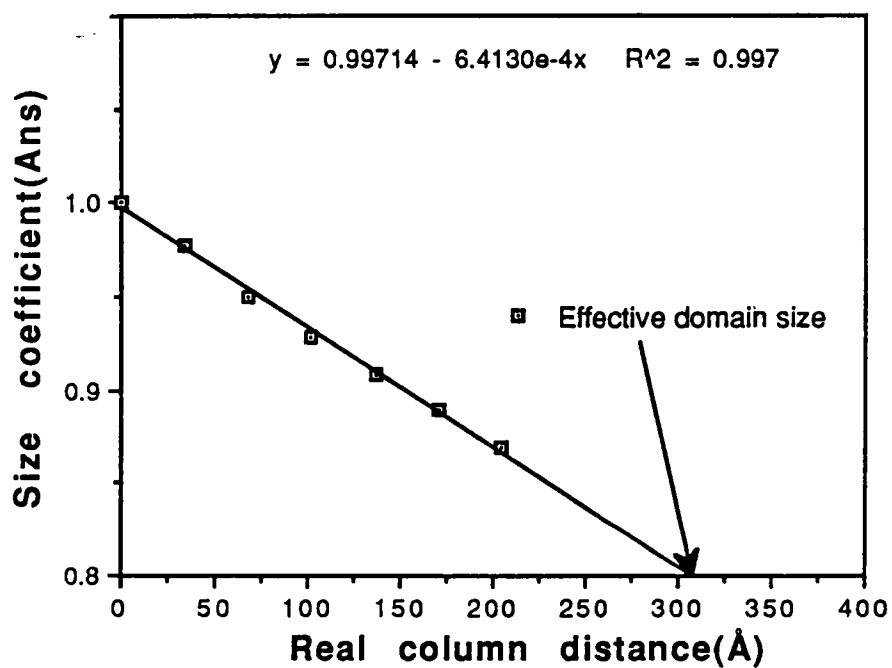
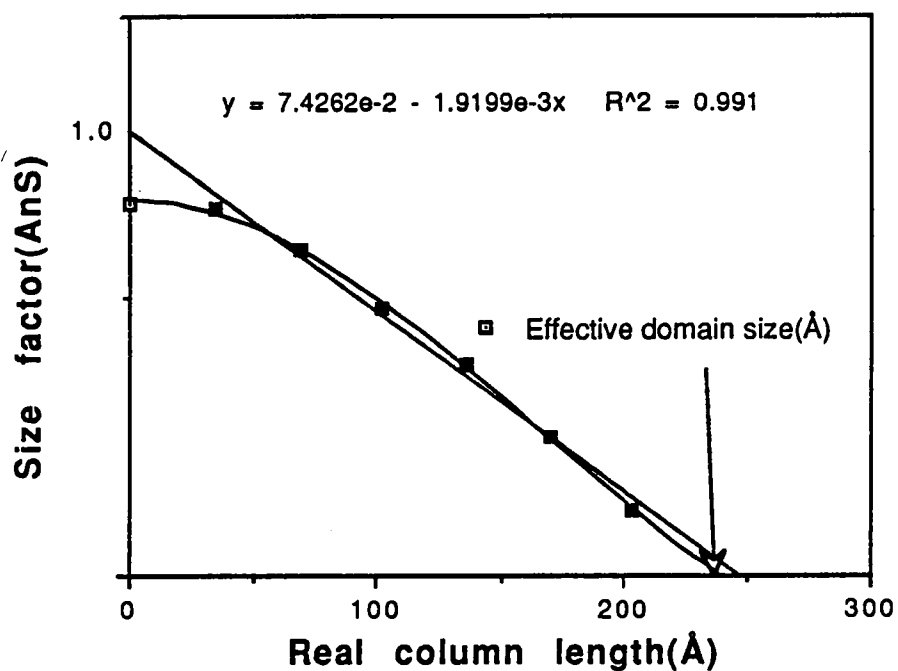


**Fig.3.8.** Plots of cosine size coefficient( $AL^S$ ) vs. real column length( $L$ ) (examples of the plot to get the effective crystallite size).

## CHAPTER4

## RESULTS

### 4.1 Calibration method

Fig.4.1 shows the intensity profiles of the 420 reflection scanned at various  $\psi$  angles after calibration and Fig.4.2 shows their least squares parabola fitting plots. As a result of the calculation to determine the peak position their deviations ( $\Delta 2\theta_p$ ) appeared to be within  $0.02^\circ$  including out-of-para-focusing errors. That means that the diffractometer was well-aligned.

At the para-focusing condition ( $\psi=0^\circ$ ) the stress-free d-spacing ( $d_0 = 0.80463 \text{ \AA}$ ) for 420 reflection was obtained.

### 4.2 Macro-residual stress analysis

#### 4.2.1 Peak scans

In the Fig.4.3 the intensity peak profiles of the 420 reflection are plotted as a function of two theta values for various  $\psi$  tilt angles for each specimen.

#### 4.2.2 Parabola fitting curves

The least squares parabola fitting curves for various  $\psi$  tilt angles used in determining the intensity peak position for each specimen are shown in Fig.4.4.

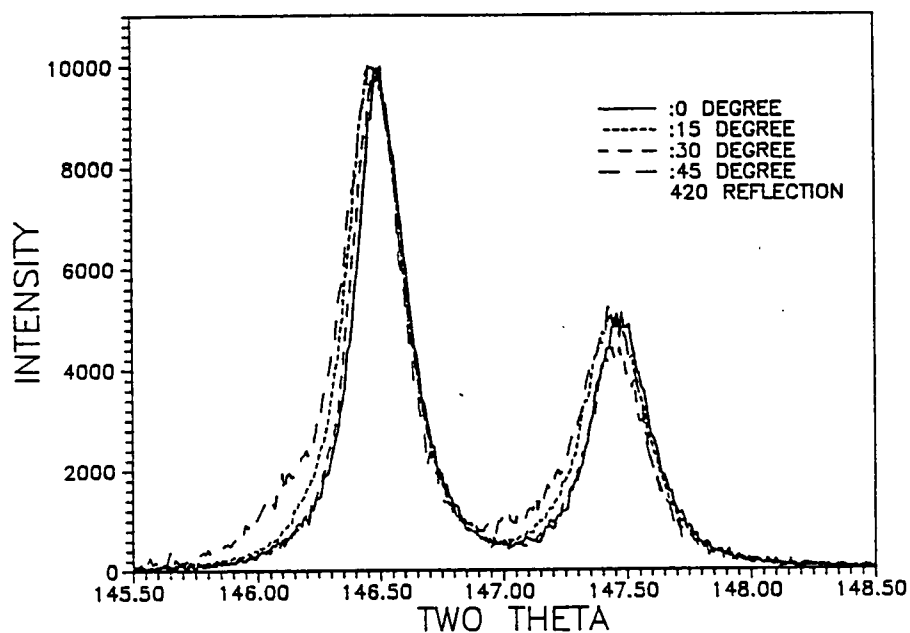


**Fig.4.1.** Peak scans of 420 reflection at various  $\psi$  angles for a fully annealed specimen after calibration.

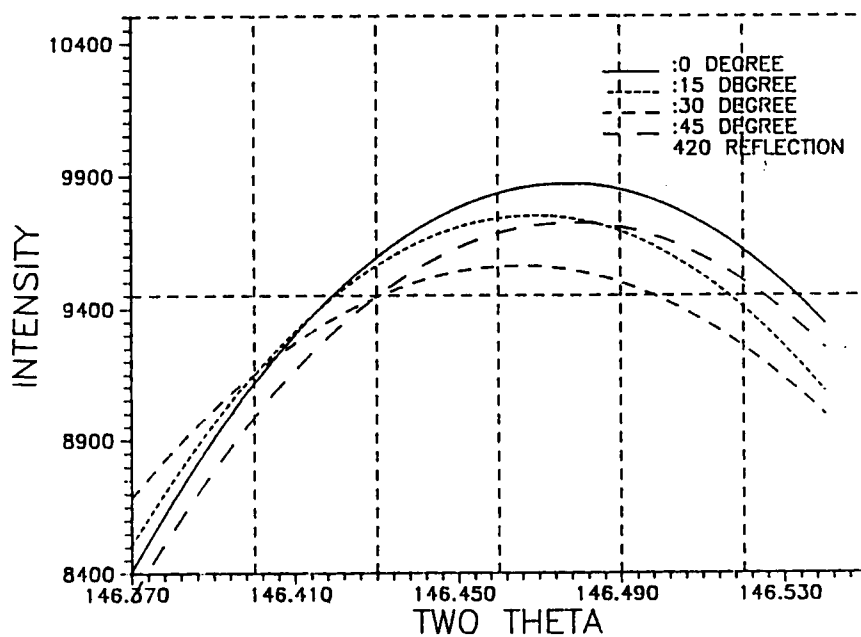


**Fig.4.2.** Least squares parabola fitting curves for the peaks from Fig.4.1.

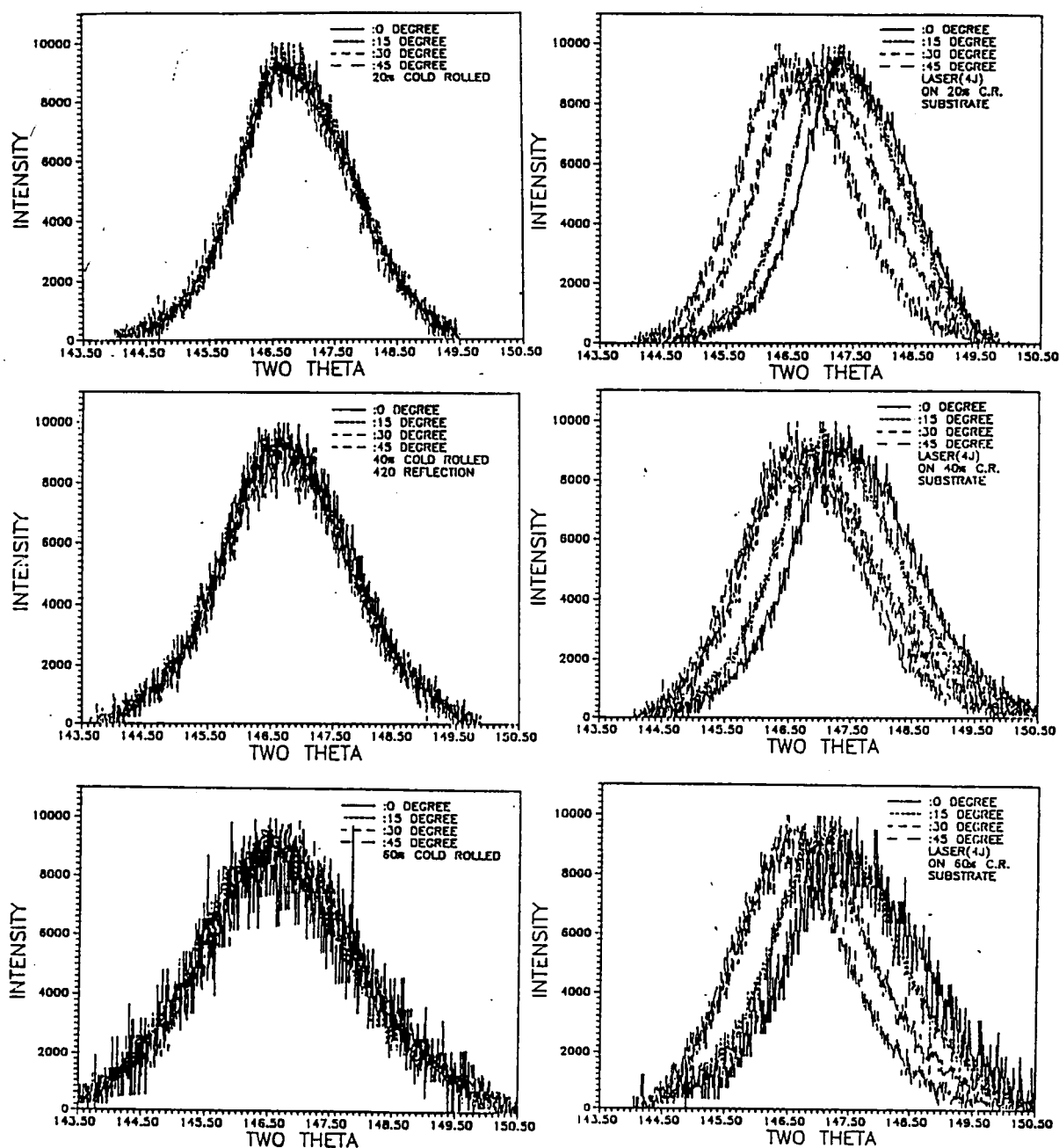


**Fig.4.3.** Peak scans of 420 reflections for the specimens used in the experiment.

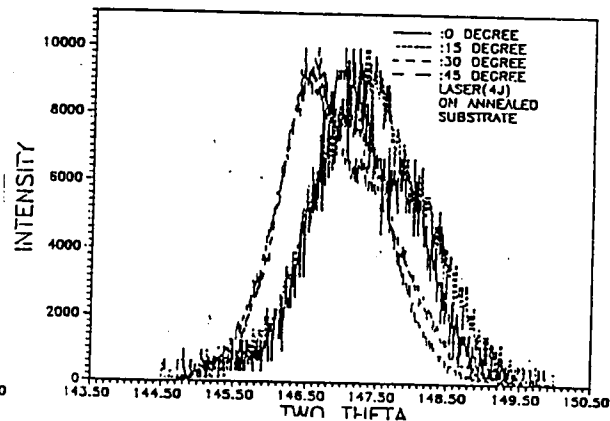
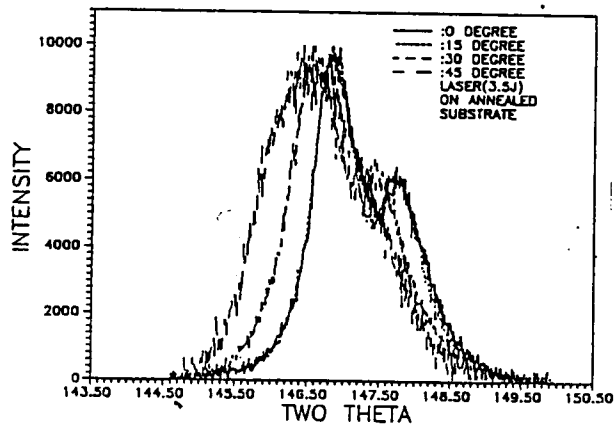
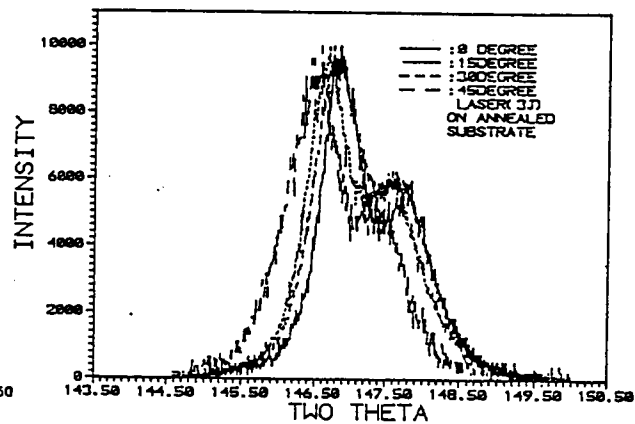
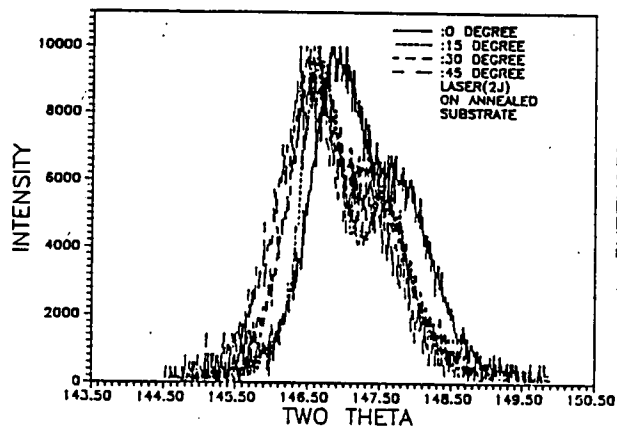


Fig.4.3. Continued

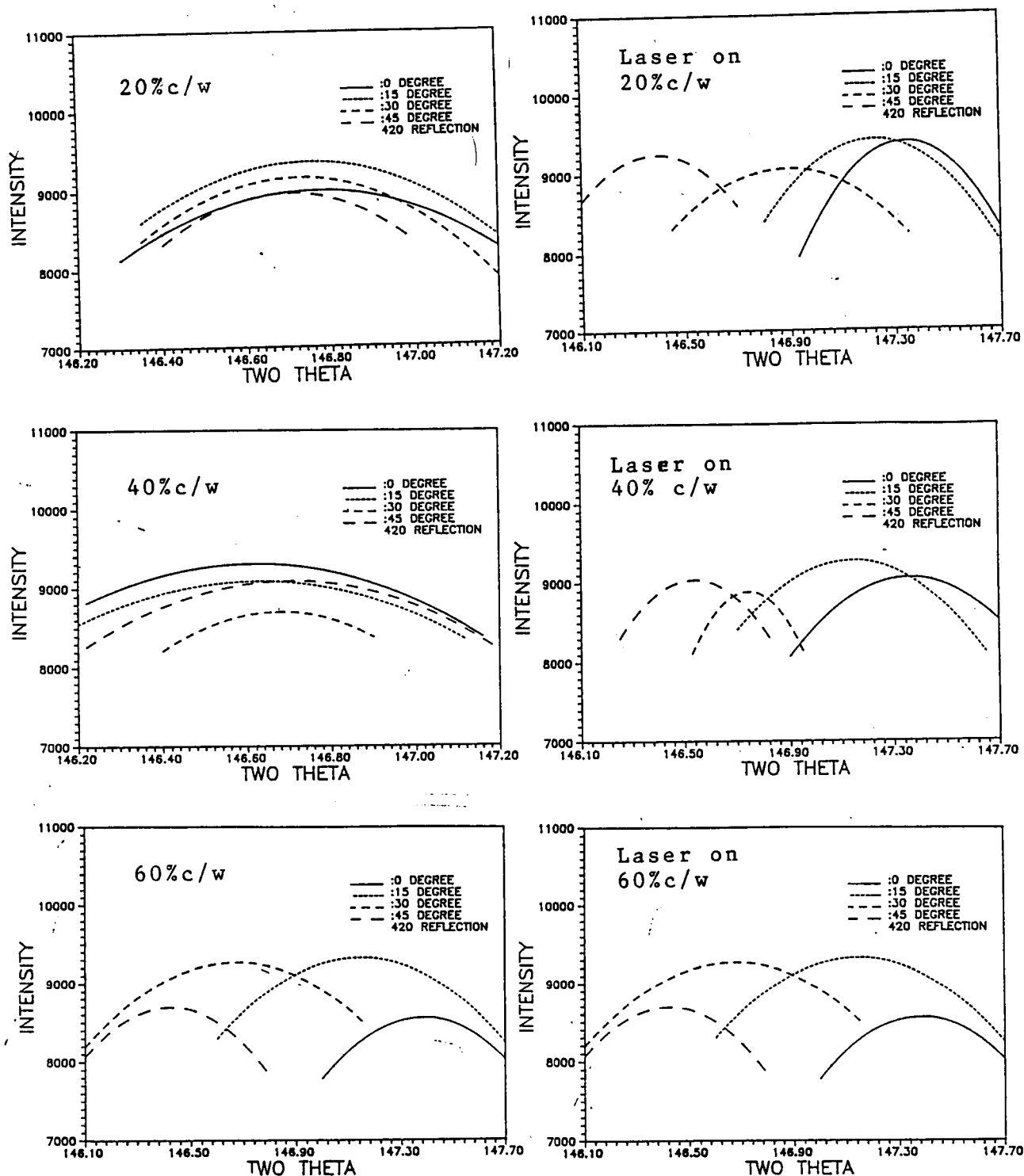


**Fig.4.4.** Least squares parabola fitting curves for the peak scans shown in the Fig.4.3.

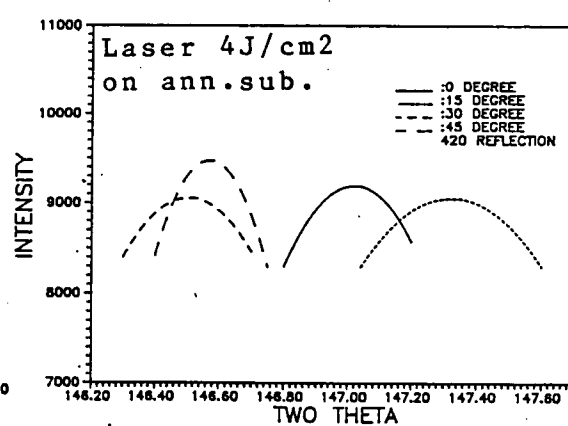
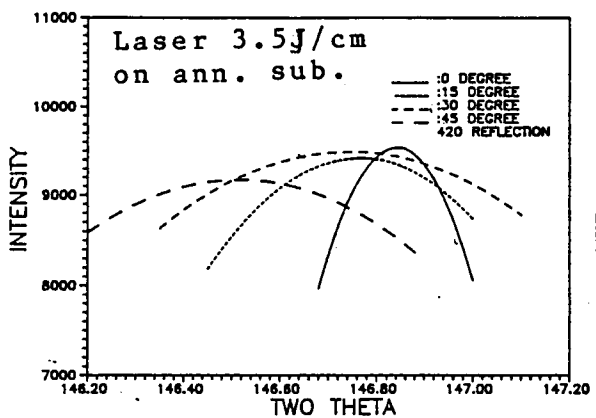
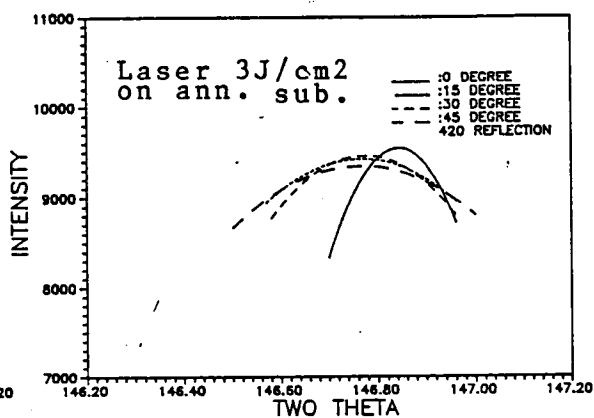
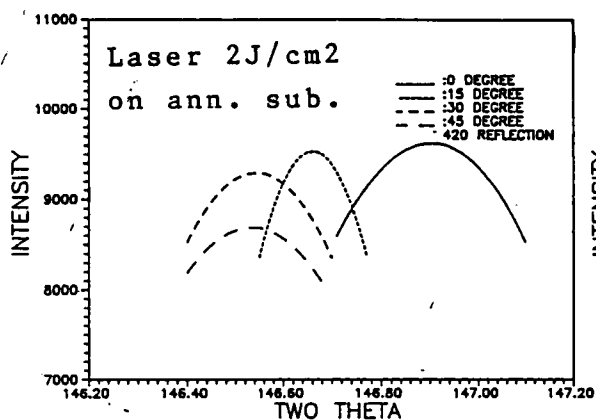


Fig.4.4. Continued

#### 4.2.3 d versus $\sin^2\psi$ plot

The plot of interplaner spacing( $d$ ) versus  $\sin^2\phi$  for the annealed and cold rolled specimens is shown in the Fig.4.5. The data were fitted to a straight line by using the least square method. As already shown in the Eq.2.3.9, we can determine the stress state in the surface of a specimen from the slope of the fitted line. When the slope is positive, the stress is tensile, while the stress is compressive when the slope is negative. In the case of Fig.4.5, when the 316L stainless steel samples are 20% cold rolled, tensile stresses are developed. On the other hand, for 40% and 60% cold rolled specimens, the stresses developed are compressive in nature.

Fig.4.6 shows the plot of the ' $d$ ' versus  $\sin^2\phi$  at various  $\phi$  tilts for specimens laser-treated with an energy density of  $4\text{J/cm}^2$  after annealing and cold-rolling. The macroresidual stresses become tensile after irradiation in all the specimens studied. Fig.4.6 shows that independently of the initial state of the stresses(viz. tensile, compressive or no-stresses) all the stresses in the stainless steel specimens become tensile with almost the same magnitude of stresses after irradiation.

Fig.4.7 shows the effect of the laser energy density on the residual stress for annealed specimens.

The values of residual stresses for each specimen, calculated using Eq.2.3.10 with  $\phi=0$ , are tabulated in table 4.1.

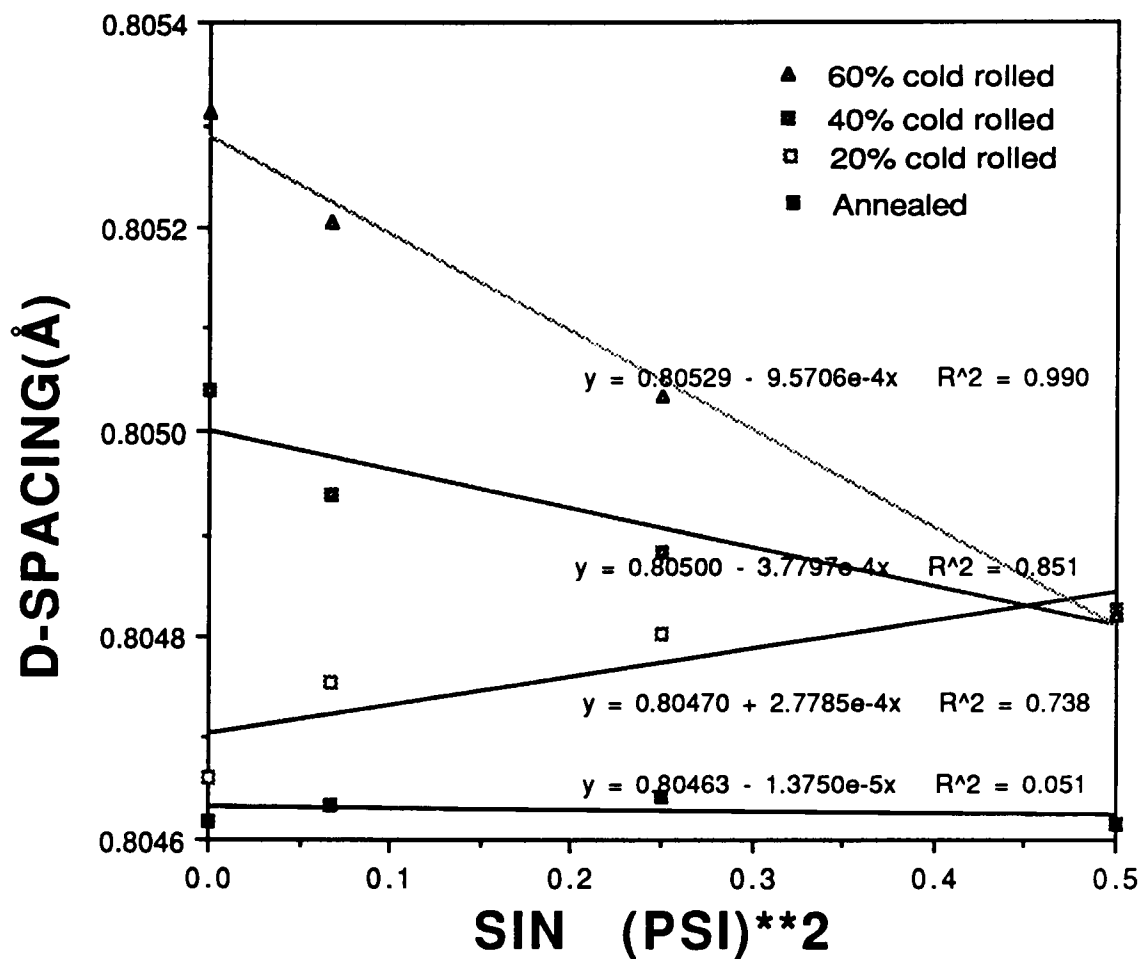


**Fig.4.5.** Plots of  $d$  vs.  $\sin^2\psi$  for annealed and cold rolled specimens.

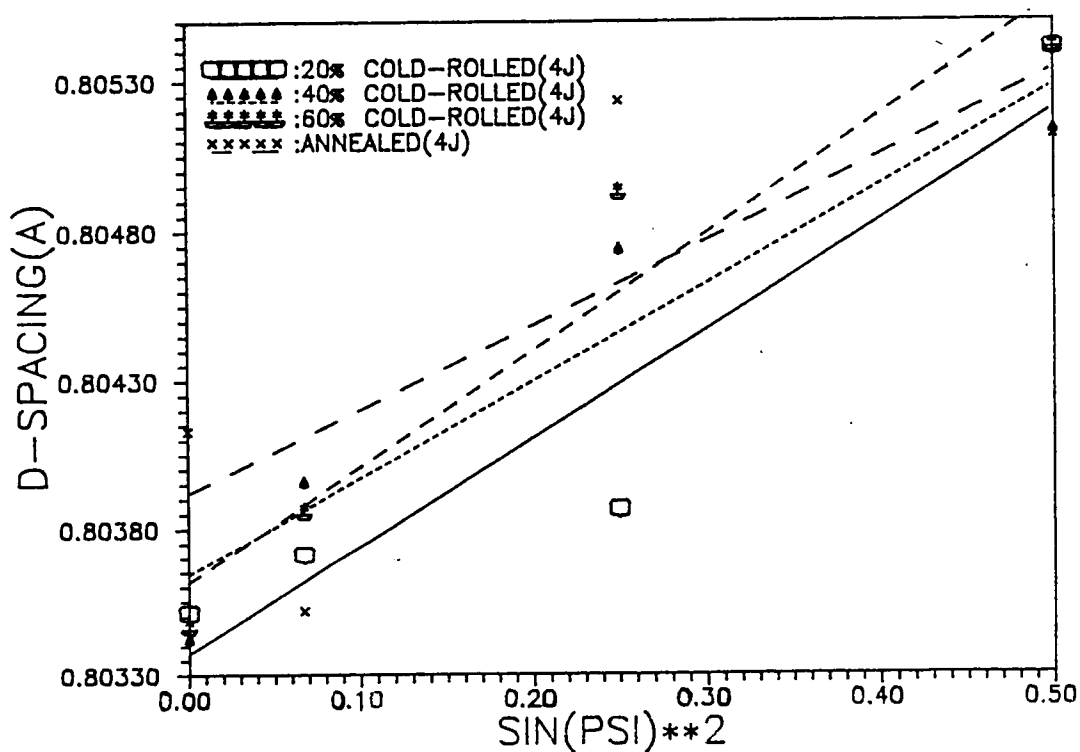
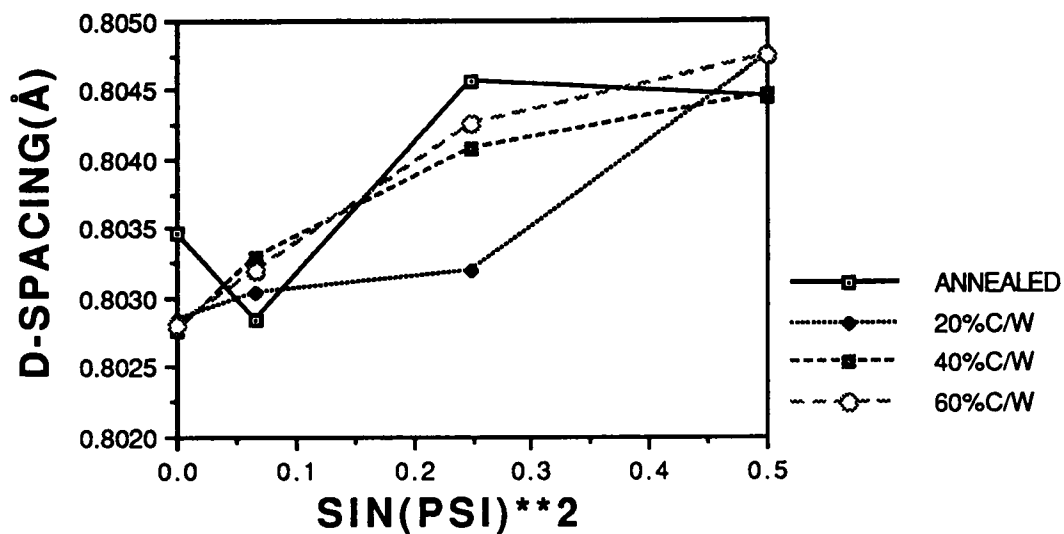


**Fig.4.6.** Plots of  $d$  vs.  $\sin^2\psi$  for annealed and cold rolled specimens after laser irradiation with  $4\text{J}/\text{cm}^2$  energy density (above; interpolation, below; least square fitting).

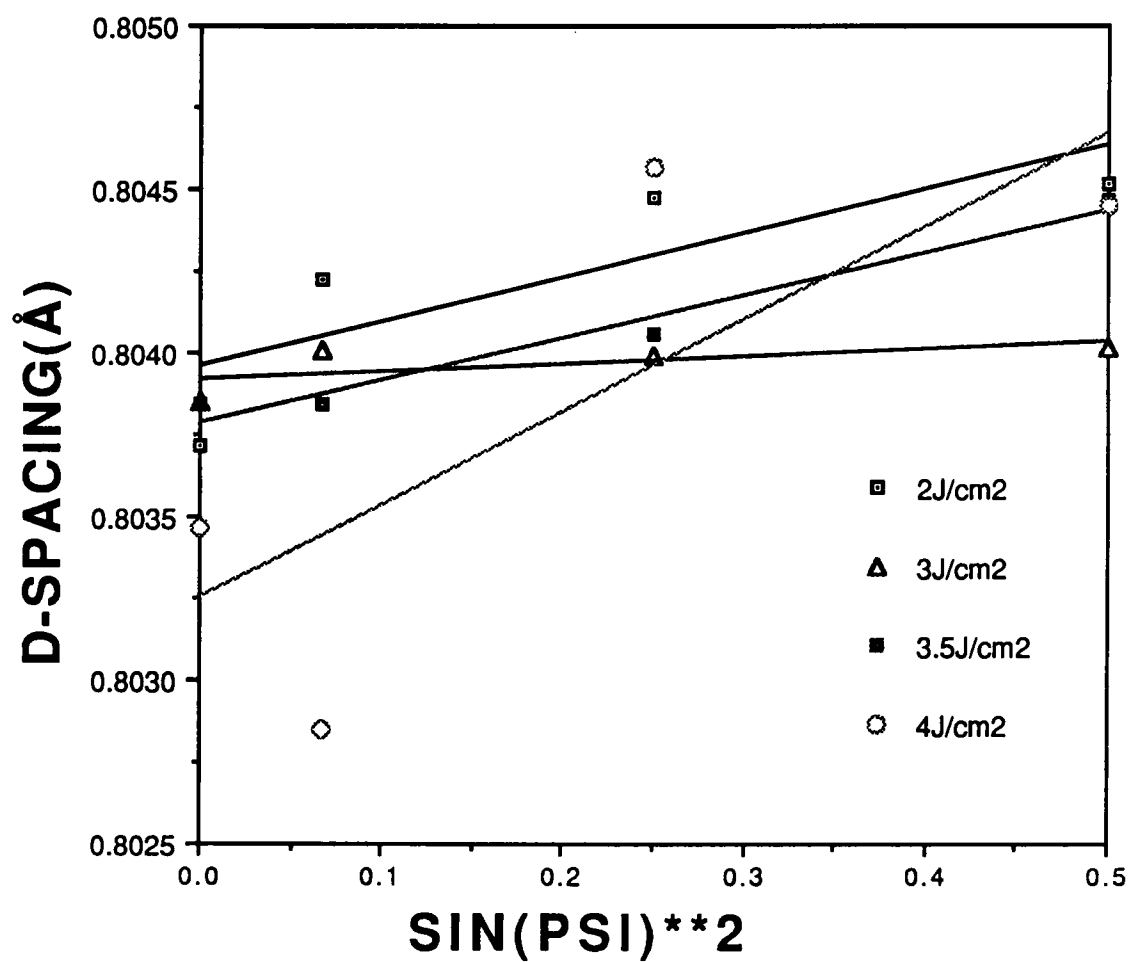


**Fig.4.7.**Plots of  $d$  vs.  $\sin^2\psi$  for annealed specimens after laser treatment with various energy densities.

**Table 4.1**

Macro-residual stress determined using  $\sin^2\psi$  method

| Specimen<br>description | Residual stress<br>before laser |                      | Residual stress<br>After laser |                      |
|-------------------------|---------------------------------|----------------------|--------------------------------|----------------------|
|                         | Mpa                             | x10 <sup>3</sup> psi | Mpa                            | x10 <sup>3</sup> psi |
| Ann. & 0.5J             |                                 |                      | 43.2                           | 6.3                  |
| Ann. & 1.0J             |                                 |                      | 71.4                           | 10.4                 |
| Ann. & 2.0J             |                                 |                      | 253.6                          | 36.8                 |
| Ann. & 3.0J             |                                 |                      | 244.9                          | 35.5                 |
| Ann. & 3.5J             |                                 |                      | 41.9                           | 6.1                  |
| Ann. & 4.0J             |                                 |                      | 530.8                          | 77.0                 |
| 20% cold-rolling        | 52.1                            | 7.6                  | 735.2 (4J)                     | 106.7                |
| 40% cold-rolling        | -70.8                           | -10.3                | 685.5 (4J)                     | 99.5                 |
| 60% cold-rolling        | -179.3                          | -26.0                | 613.8 (4J)                     | 89.1                 |

The calculations of residual stresses presented here were performed by making two main assumptions:

- 1) Plain stress condition (viz.  $\sigma_3 = \sigma_{13} = \sigma_{23} = 0$ ) because the X-ray penetration depth is approx. 2-3 $\mu$ m thick.
- 2) The specimen was regarded to be isotropic.

A more detailed analysis of the validity of these assumptions is given in the next chapter.

Fig.4.8 shows that the residual stresses increased in the specimens irradiated with lower energy densities and remained approximately constant at higher energy densities.

After laser irradiation the stresses in all the samples are not only tensile but also very high. In contrast to this result, for the poly-crystalline silicon materials neither peak shift nor line broadening were detected after laser treatment. In Fig.4.9 the residual stresses are plotted as a function of cold rolling before and after laser treatment.

### **4.3 Micro-residual stress analysis**

#### **4.3.1 Peak scans**

Fig.4.10 shows the line profiles for the 111 and 222 reflections for annealed, cold rolled and laser treated specimens used in this experiment.

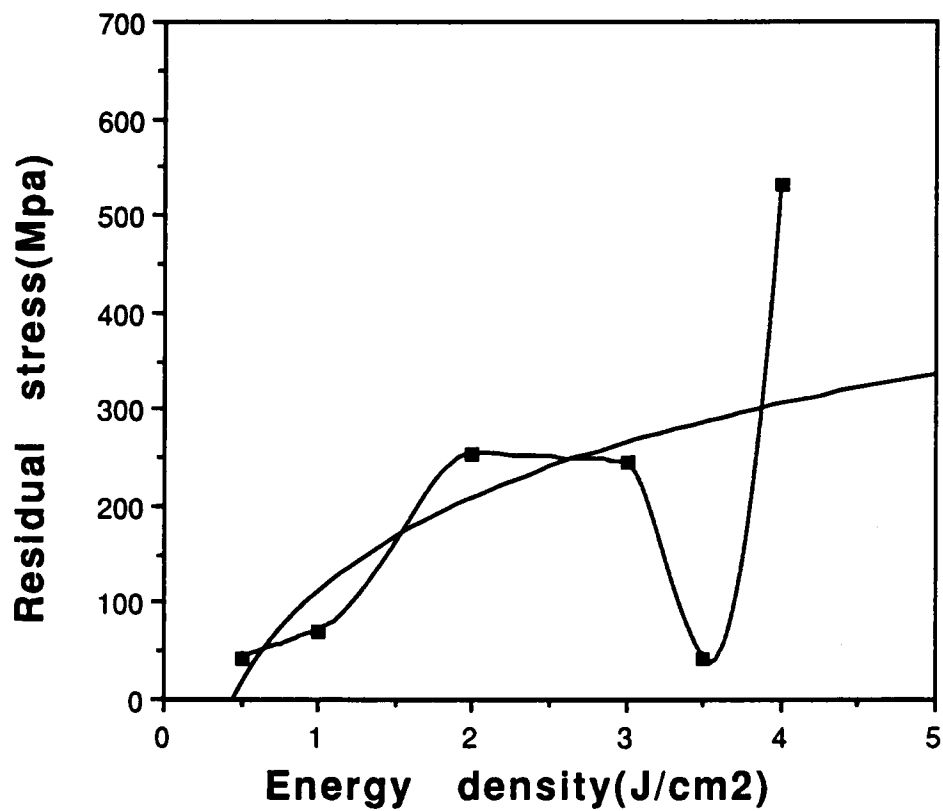


**Fig.4.8.** Plot of the residual stress versus laser energy density for annealed specimen.

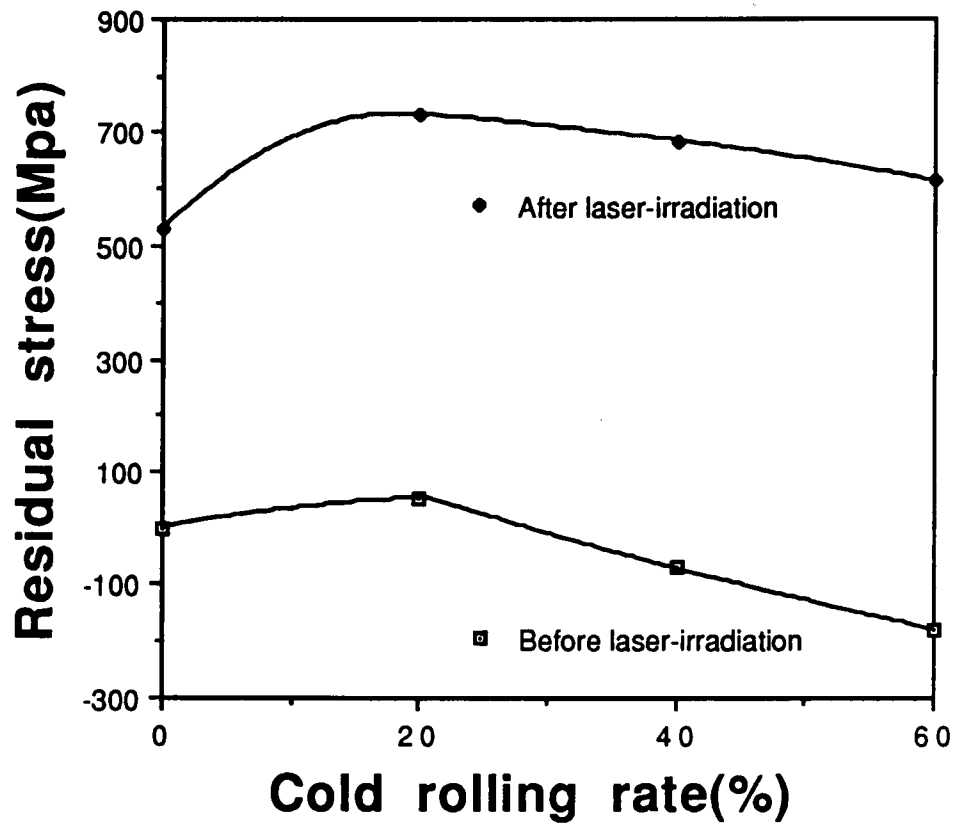


**Fig.4.9.** Plot of the residual stress versus cold rolling before and after laser treatment.

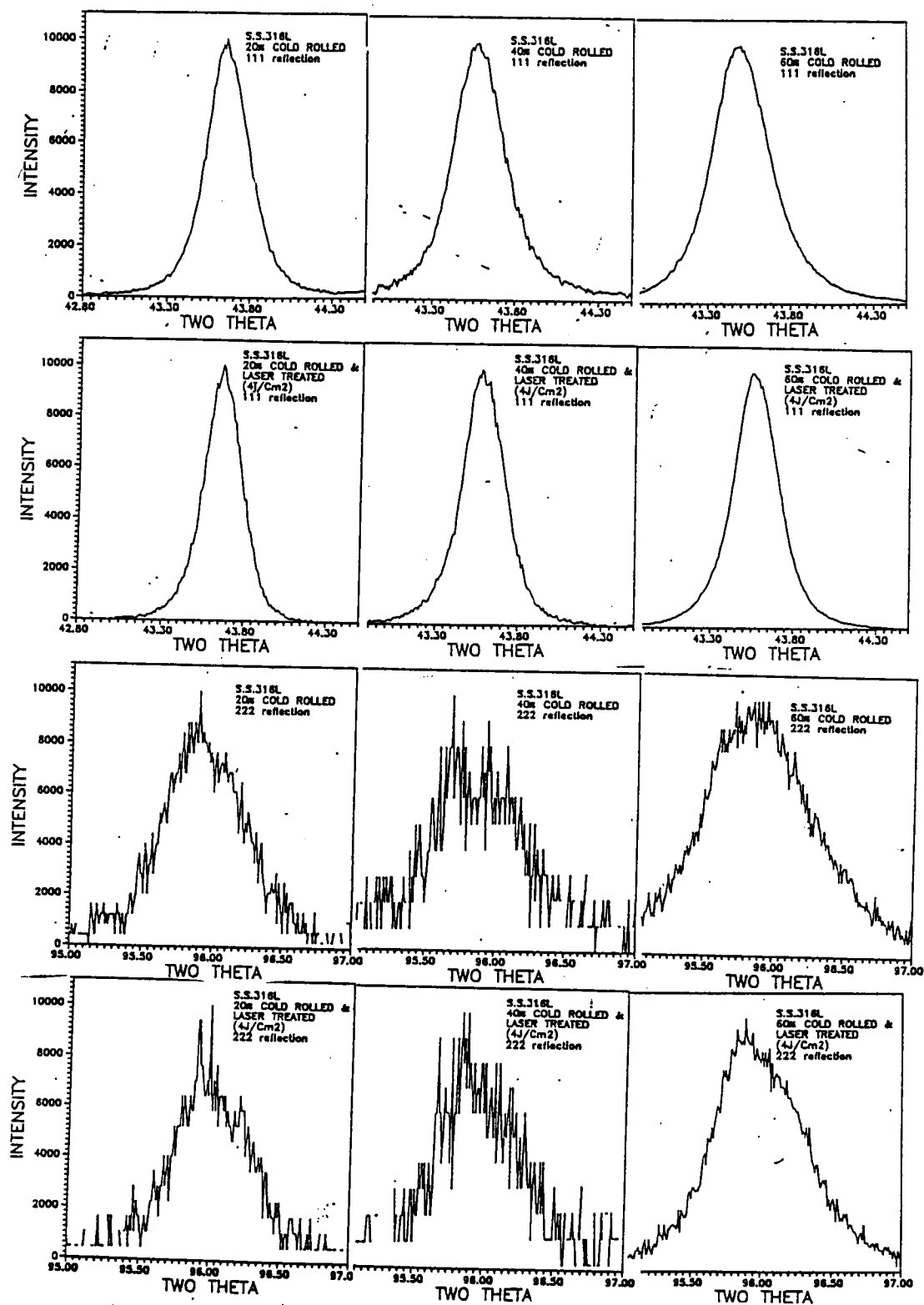


**Fig.4.10.**Intensity peak profiles of 111 and 222

reflections used in determining the micro-residual stresses.

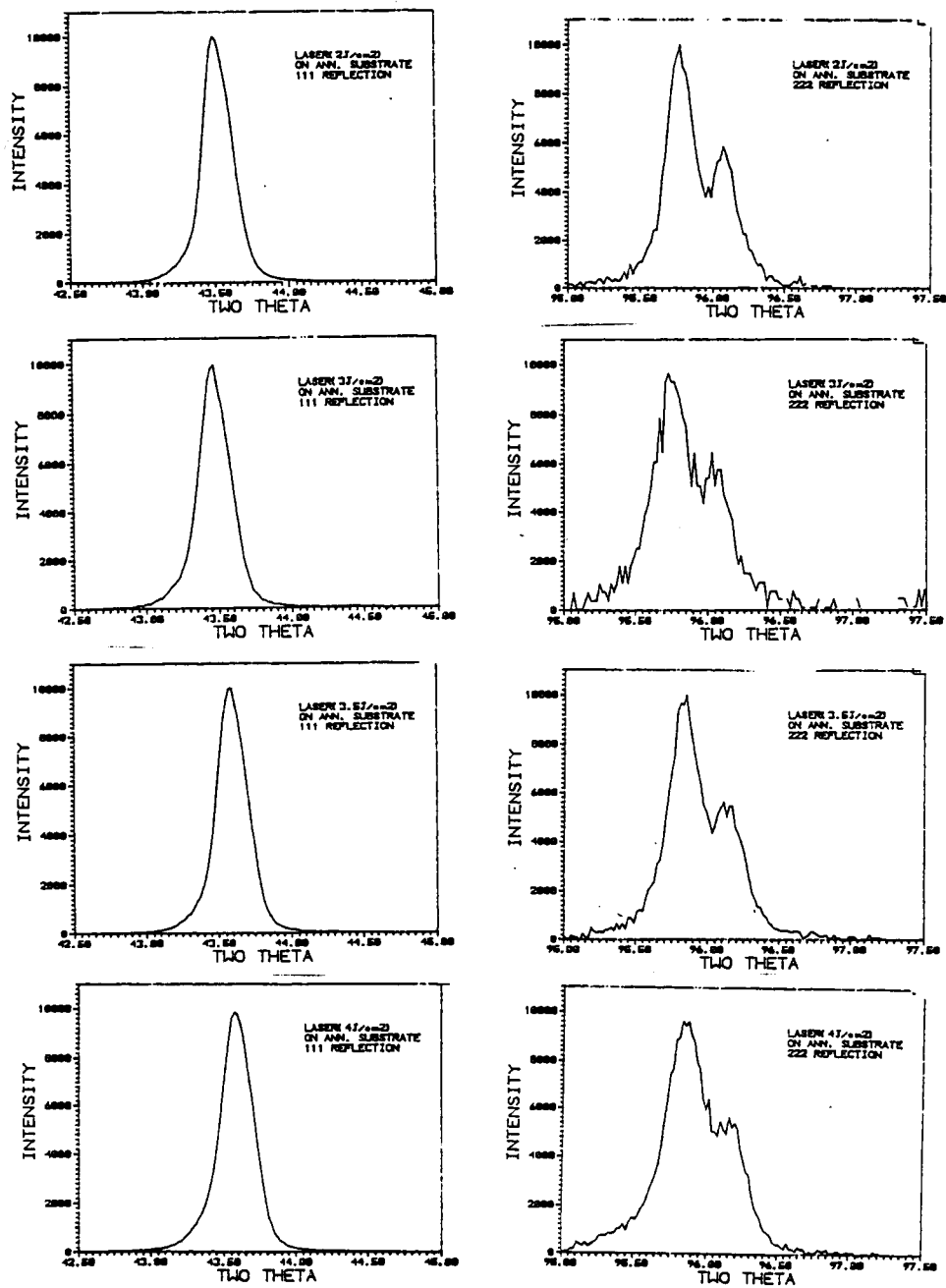


Fig.4.10. Continued

#### 4.3.2 Line broadening measurement

The line broadening was evaluated using the variance and Fourier method. Table 4.2 lists the variance values for the 111 and 222 reflections for annealed and cold rolled specimens before and after laser irradiation.

As is to be expected, the variance increased as the amount of cold working increased (Fig.4.11).

Fig.4.12 shows the variance change as a function of laser energy density for laser-treated annealed substrates at various energy densities. The variance of the irradiated annealed samples increases as the energy density increases.

For the cold rolled specimens the variance was decreased as a result of the laser irradiation with  $4\text{J}/\text{cm}^2$  and the difference of the variance became larger for the higher cold rolled specimen (Fig.4.13).

When the 60% cold rolled specimen was laser irradiated with  $3\text{J}/\text{cm}^2$  and  $4\text{J}/\text{cm}^2$ , it appeared that the higher laser energy density reduced the variance value more.

#### 4.3.3. Separation of the crystallite size and RMS strain

The Fourier analysis method developed by Warren and Averbach was used to separate the crystallite size and RMS strain from the pure broadened line profile and the method was explained in the section 3.3.3.3. Stoke's correction has been checked to ensure the accuracy of the calculation

**Table 4.2**

Variance comparison before and after  
laser treatment

| Specimen<br>description | Reflection<br>plane | Variance ( $\times 10^{-4}$ ) |                | Laser<br>energy<br>density<br>(J/cm <sup>2</sup> ) | Variance<br>change<br>( $\times 10^4$ ) |
|-------------------------|---------------------|-------------------------------|----------------|----------------------------------------------------|-----------------------------------------|
|                         |                     | Before<br>laser               | After<br>laser |                                                    |                                         |
| Annealed<br>#1          | 111                 | 237                           | 269            | 2                                                  | 32                                      |
|                         | 222                 | 541                           | 910            |                                                    | 369                                     |
| Annealed<br>#2          | 111                 | 160                           | 226            | 3                                                  | 66                                      |
|                         | 222                 | 426                           | 727            |                                                    | 301                                     |
| Annealed<br>#3          | 111                 | 173                           | 232            | 3.5                                                | 59                                      |
|                         | 222                 | 404                           | 716            |                                                    | 312                                     |
| Annealed<br>#4          | 111                 | 213                           | 263            | 4                                                  | 50                                      |
|                         | 222                 | 442                           | 809            |                                                    | 357                                     |
| Cold-rolled<br>20%      | 111                 | 691                           | 485            | 4                                                  | -206                                    |
|                         | 222                 | 1294                          | 987            |                                                    | -307                                    |
| Cold-rolled<br>40%      | 111                 | 839                           | 457            | 4                                                  | -382                                    |
|                         | 222                 | 1870                          | 1182           |                                                    | -688                                    |
| Cold-rolled<br>60%      | 111                 | 928                           | 465            | 4                                                  | -463                                    |
|                         | 222                 | 2746                          | 1647           |                                                    | -1099                                   |
|                         | 111                 |                               | 514            | 3                                                  | -414                                    |
|                         | 222                 |                               | 1848           |                                                    | -898                                    |

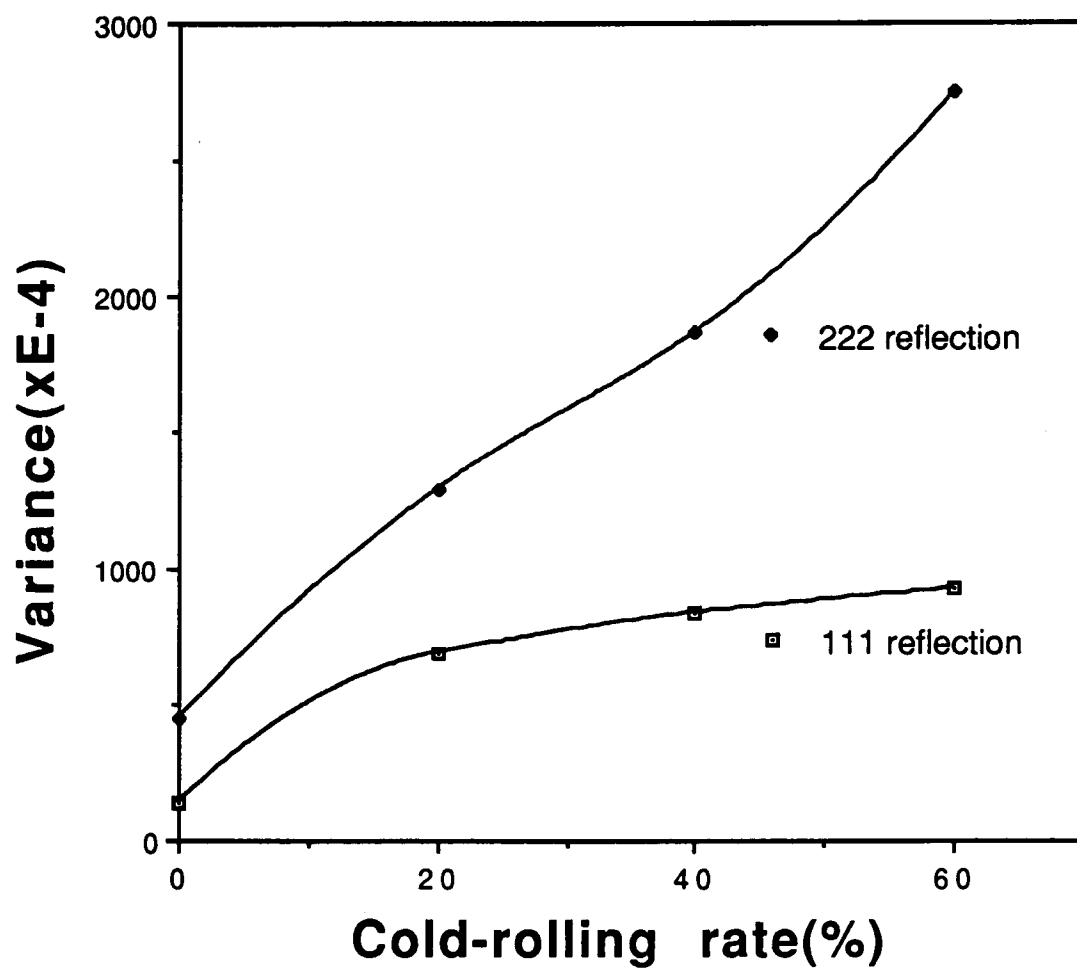


**Fig.4.11.** Plot of variance vs. cold rolling.

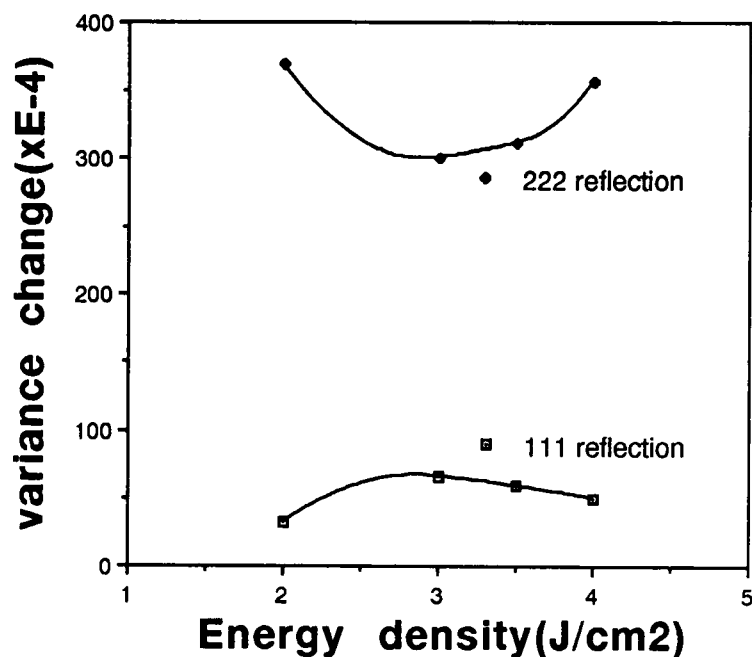


Fig.4.12. Variance change vs.laser energy density for the annealed substrate after laser irradiation with various energy densities.

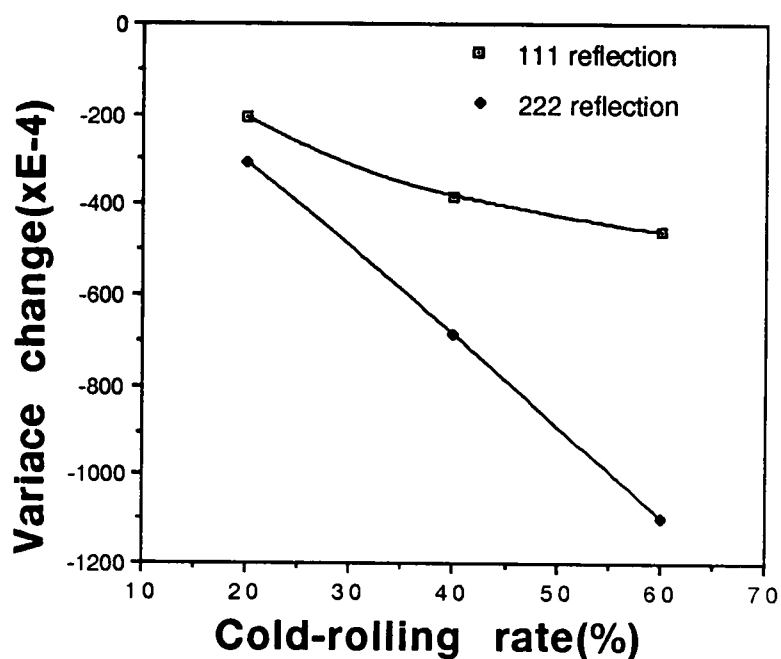


Fig.4.13. Plot of the variance change vs. percent of cold work for cold rolled specimens after laser irradiation at 4J/cm<sup>2</sup> energy density.

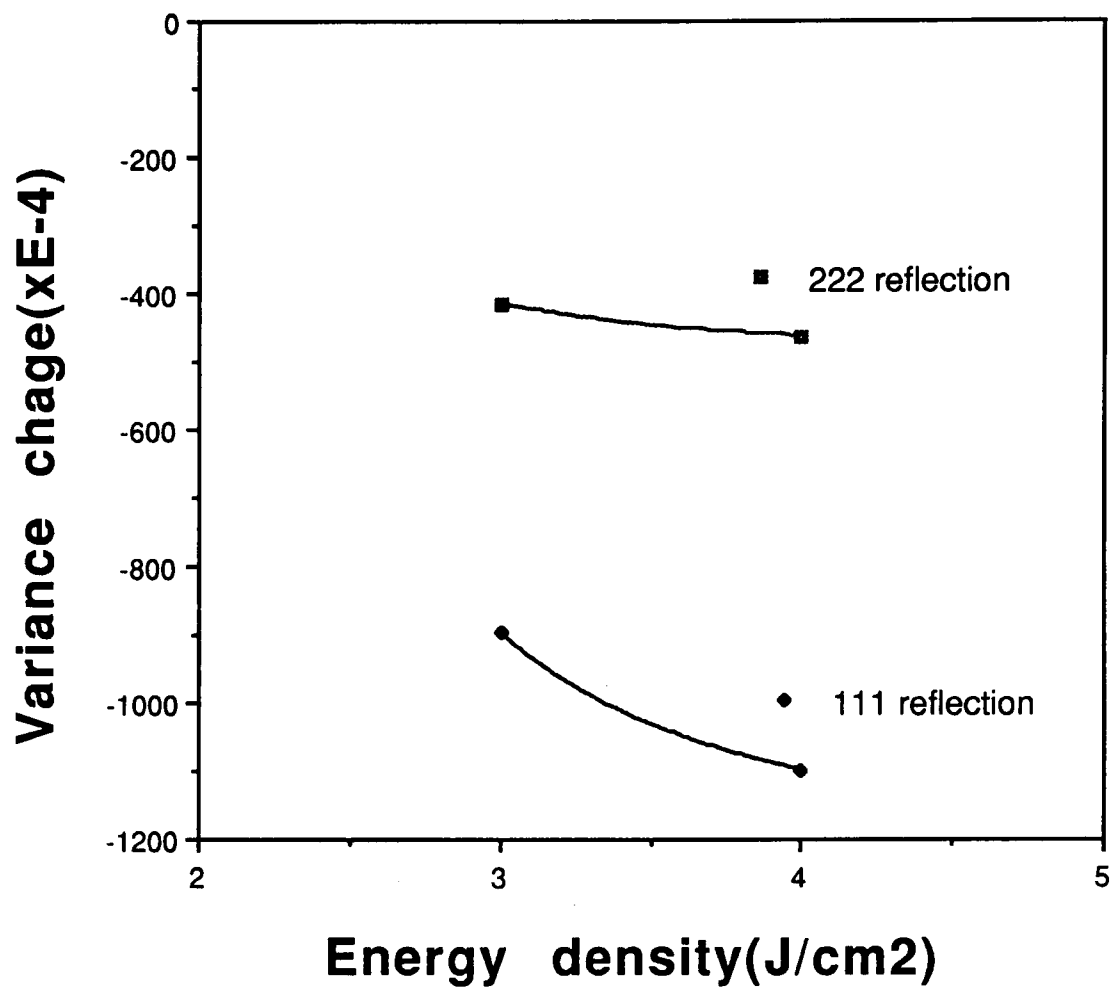


**Fig.4.14.** Plot of the variance change vs. different energy densities for 60% cold rolled specimen.(Fig.4.15). All the plots used in separating the size effect and the lattice strain effect are shown in Fig.4.16.

Table 4.3 lists RMS strain and effective crystallite size values for the annealed specimens after laser irradiation and table 4.4 tabulates RMS strain and effective crystallite size values for cold rolled specimens before and after laser irradiation. For all cases the Fourier size coefficients and RMS strain values increased as the real column length increased. That means that the contributions to the line broadening as a result of cold rolling and laser treatment are due to both the mosaic structure and non-uniform elastic lattice strain.

For the  $2\text{J}/\text{cm}^2$  laser treated sample the effective crystallite size is very large and in  $3\text{J}/\text{cm}^2$ ,  $3.5\text{J}/\text{cm}^2$  and  $4\text{J}/\text{cm}^2$  energy densities the particle sizes become smaller and almost constant (Fig.4.17). This means that in lower energy density the contribution to the line broadening is mainly due to lattice strains and in higher energy densities both the particle size and the lattice strain can affect the line broadening.

Fig.4.18 shows that the lattice strain effect to the line broadening increases as laser energy densities increase.

The RMS strains for cold rolled specimens are almost the same as a result of the laser irradiation except for the 40% cold rolled specimen as shown in Fig.4.19.

The effective crystallite size for the cold rolled specimens increased after laser irradiation with  $4\text{J}/\text{cm}^2$  energy density as shown in the Fig.4.20.

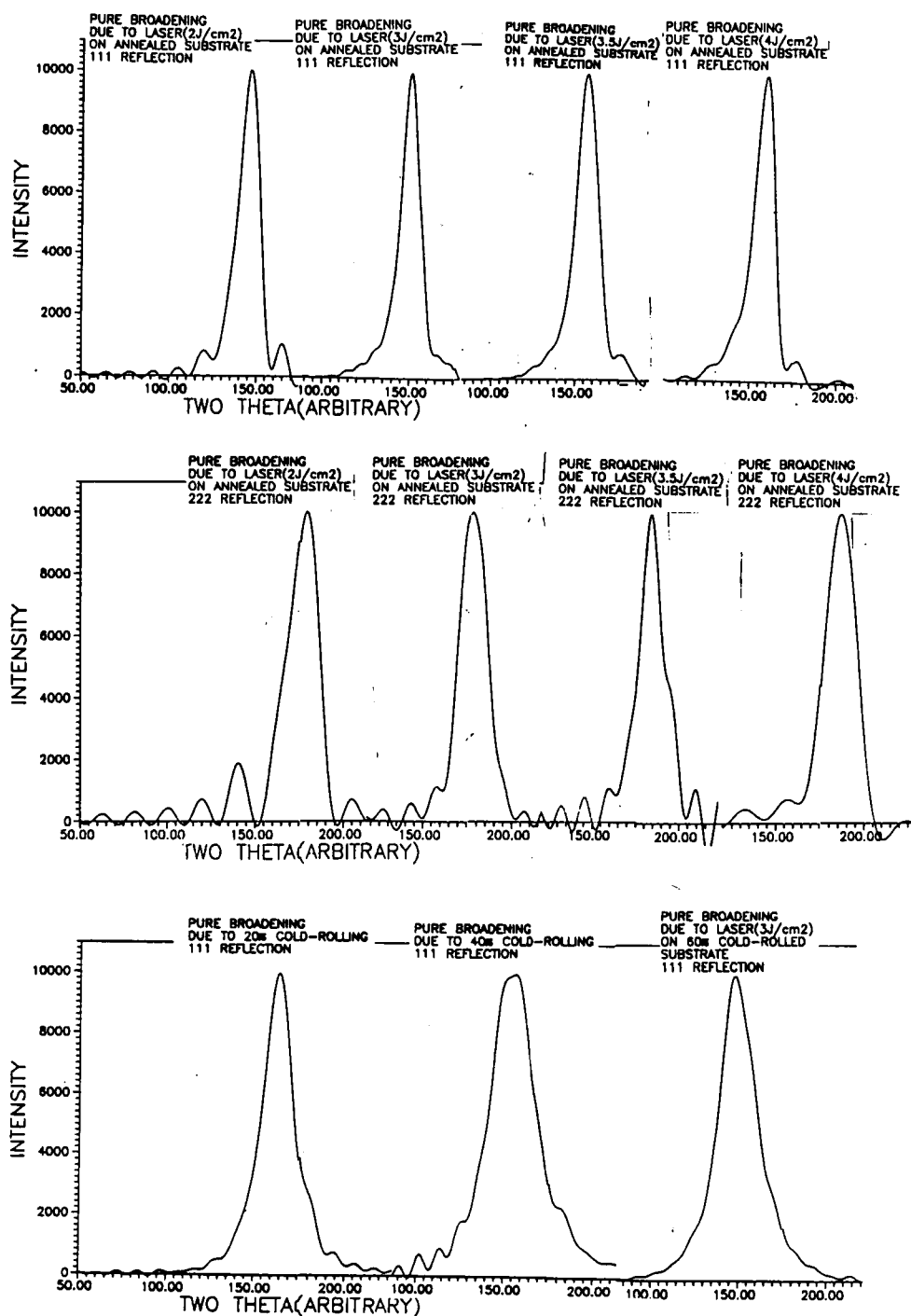


**Fig.4.15.** Pure broadened  $K\alpha_1$  line profiles after Stoke's correction. (The integrated intensity should be consistent with the variance values determined from the total peaks.)

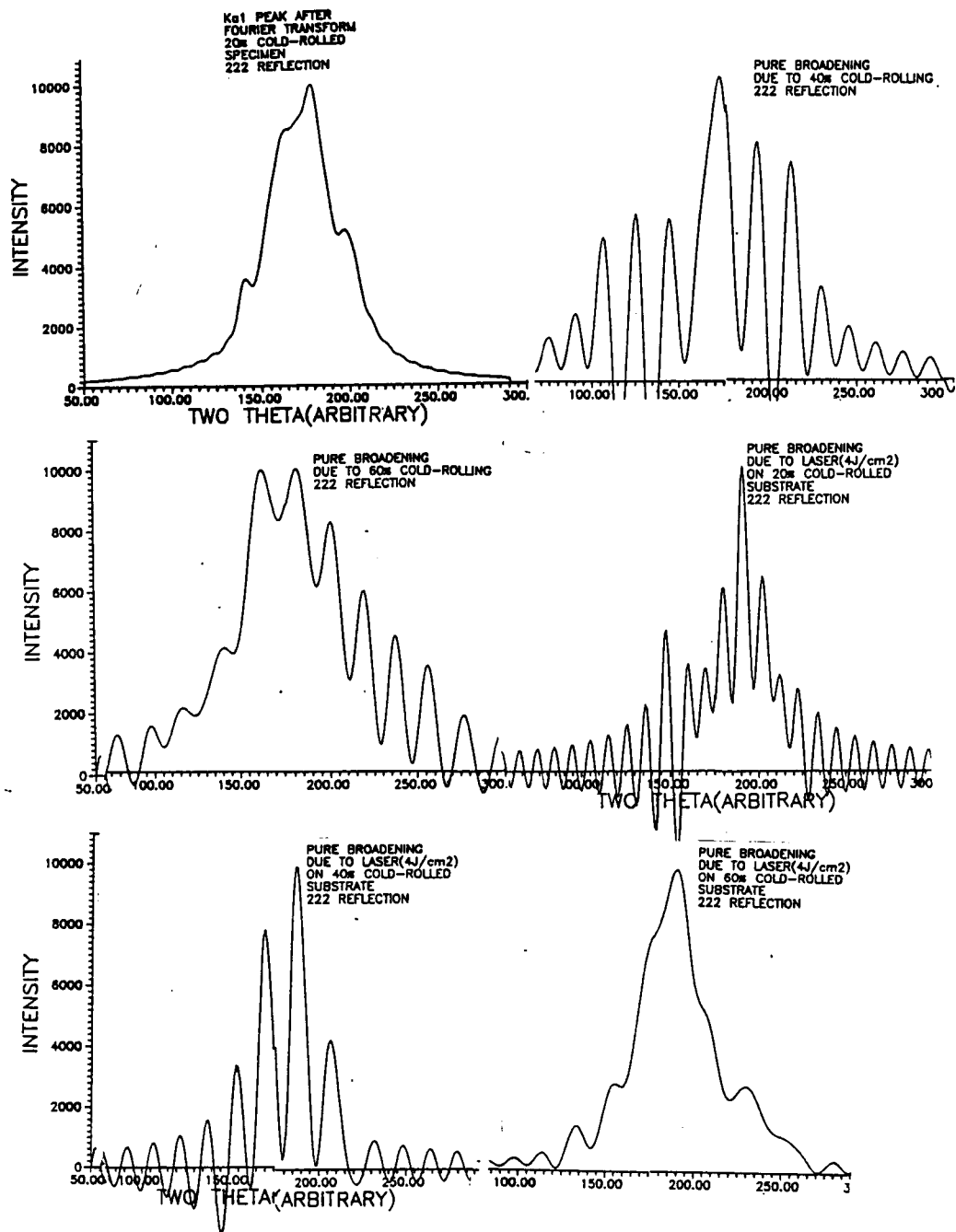


Fig. 4.15. Continued

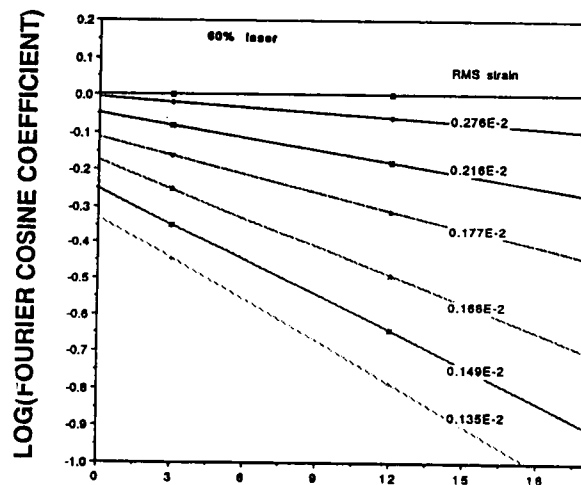
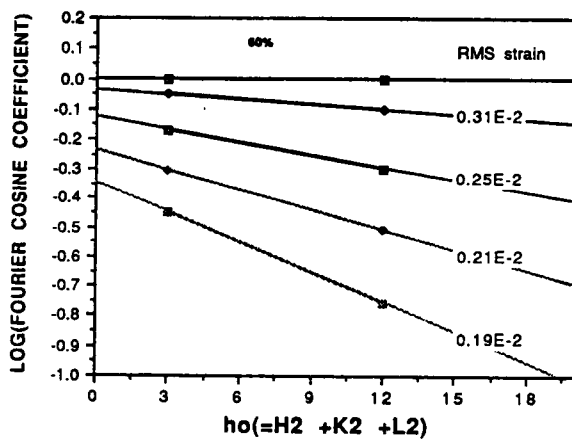
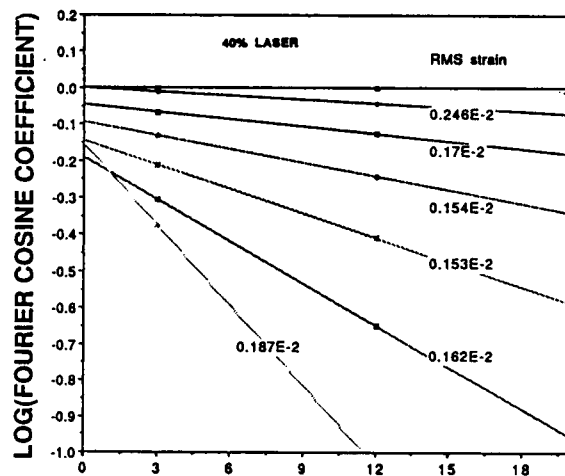
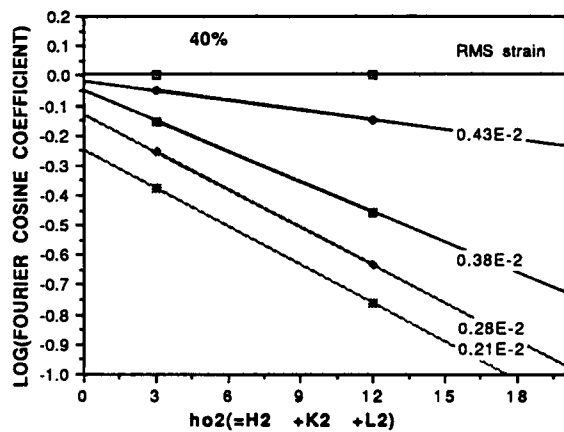
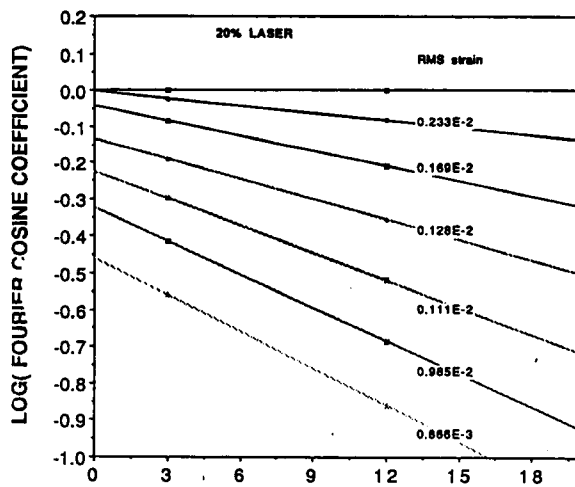
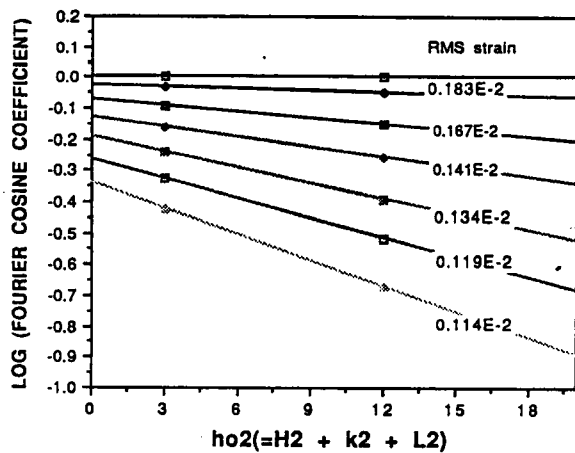


**Fig.4.16.** Plots used to separate the crystallite size and the lattice strain.

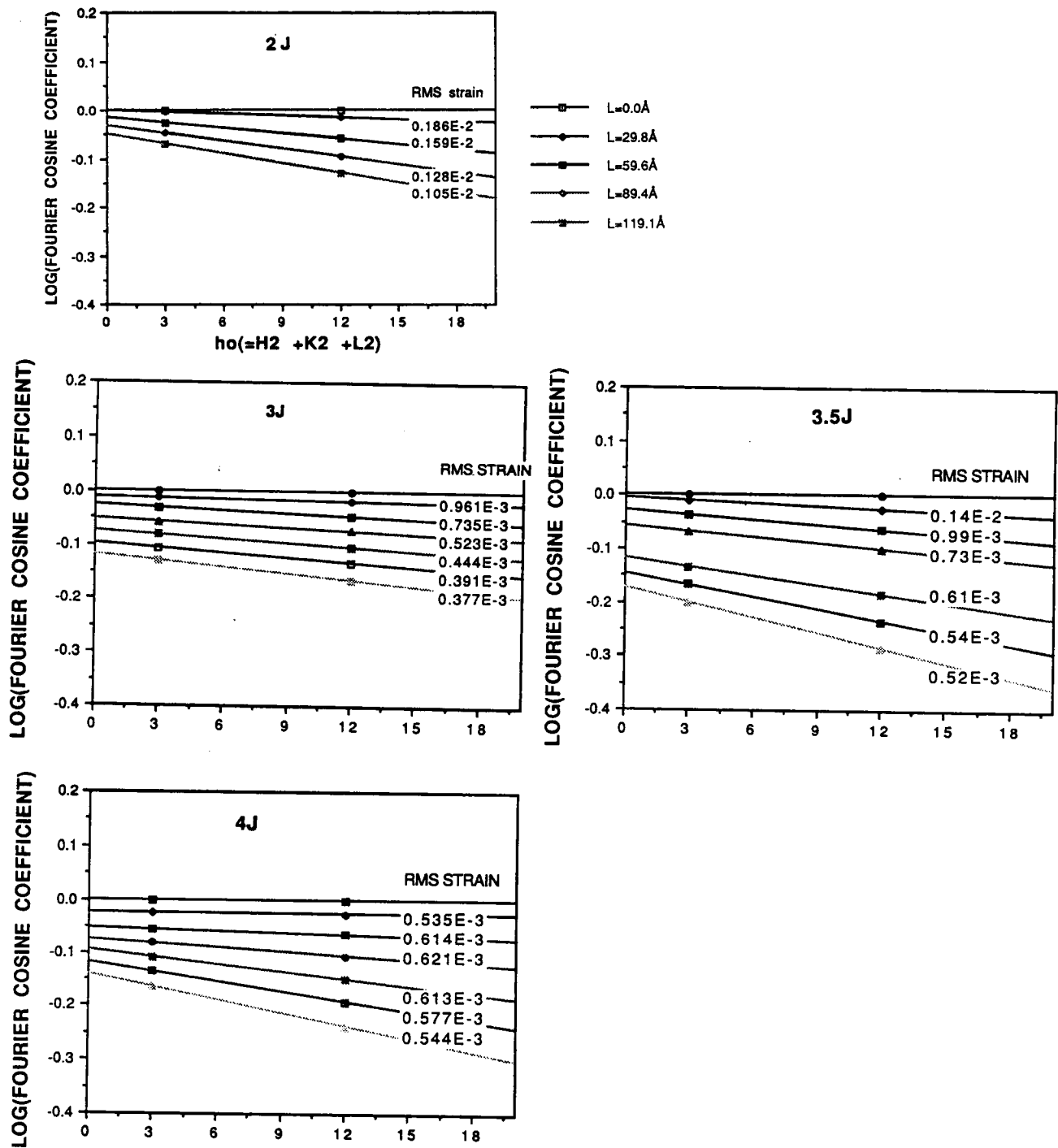


Fig.4.16. Continued

**Table 4.3**

Effective crystallitic sizes and RMS strains for  
the specimens laser-treated on annealed substrates

| Specimen<br>description                            | Laser Energy density (J/cm <sup>2</sup> ) |        |        |        |
|----------------------------------------------------|-------------------------------------------|--------|--------|--------|
|                                                    | 2                                         | 3      | 3.5    | 4      |
| Effective<br>crystallite<br>size ( $D_{eff}$ ) (Å) | 3.76E4                                    | 1.1E3  | 1.57E3 | 6.1E2  |
| <u>Column</u><br><u>depth</u><br>L=34.8Å           | 1.9E-3                                    | 9.6E-4 | 1.5E-3 | 5.4E-4 |
| <u>RMS strain</u> L=68.1Å                          | 1.6E-3                                    | 7.4E-4 | 9.9E-3 | 6.1E-4 |
| L=102.1Å                                           | 1.3E-3                                    | 5.2E-4 | 7.3E-4 | 6.2E-4 |
| L=136.1Å                                           | 1.1E-3                                    | 4.4E-4 | 6.1E-4 | 6.1E-4 |

**Table 4.4**

Effective crystallite sizes and RMS strains for  
the specimens cold-rolled and laser-treated on  
the cold-rolled specimens

| specimen            | Cold rolled specimen |        |        | laser treated on the cold<br>rolled specimen (J/cm <sup>2</sup> ) |         |        |        |
|---------------------|----------------------|--------|--------|-------------------------------------------------------------------|---------|--------|--------|
|                     | 20%                  | 40%    | 60%    | 20%(4)                                                            | 40%(4)  | 60%(4) | 60%(3) |
| description         |                      |        |        |                                                                   |         |        |        |
| Effective           |                      |        |        |                                                                   |         |        |        |
| crystallite size(A) | 3.8E2                | 2.3E2  | 2.8E2  | 1.4E3                                                             | 3.7E2   | 1.2E3  | 5.8E3  |
| — Column            |                      |        |        |                                                                   |         |        |        |
| — depth             |                      |        |        |                                                                   |         |        |        |
| L=34.4A             | 2.7E-3               | 2.3E-3 | 3.1E-3 | 2.7E-3                                                            | 1.19E-3 | 3.2E-3 | 3.1E-3 |
| RMS strain L=68.1A  | 1.9E-3               | 1.5E-3 | 2.5E-3 | 2.1E-3                                                            | 1.18E-3 | 2.7E-3 | 2.3E-3 |
| L=102.1A            | 1.5E-3               | 1.1E-3 | 2.1E-3 | 1.7E-3                                                            | 1.2E-3  | 1.7E-3 | 1.8E-3 |
| L=136.2A            | 1.2E-3               | 6.9E-4 | 1.9E-3 | 1.4E-3                                                            | 1.4E-3  | 1.6E-3 | 1.7E-3 |

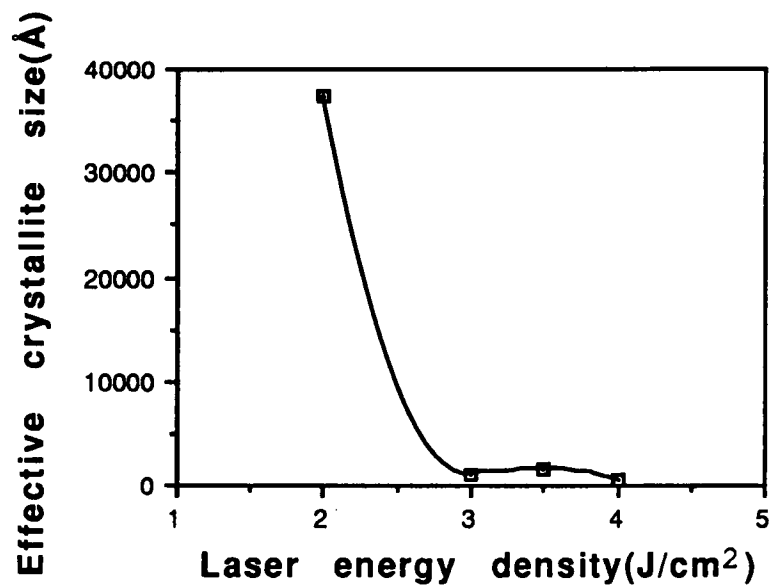


**Fig.4.17.** Plot of the effective crystallite size versus laser energy density for the annealed specimen.

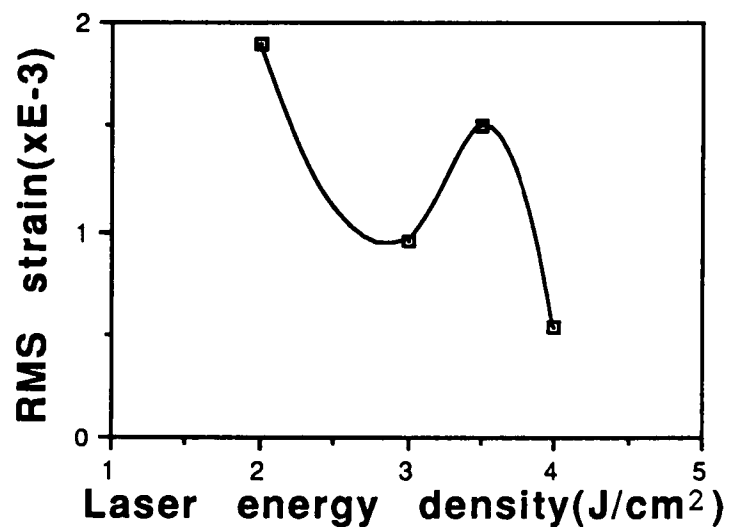


**Fig.4.18.** Plot of the RMS strain versus laser energy density for the annealed specimen.

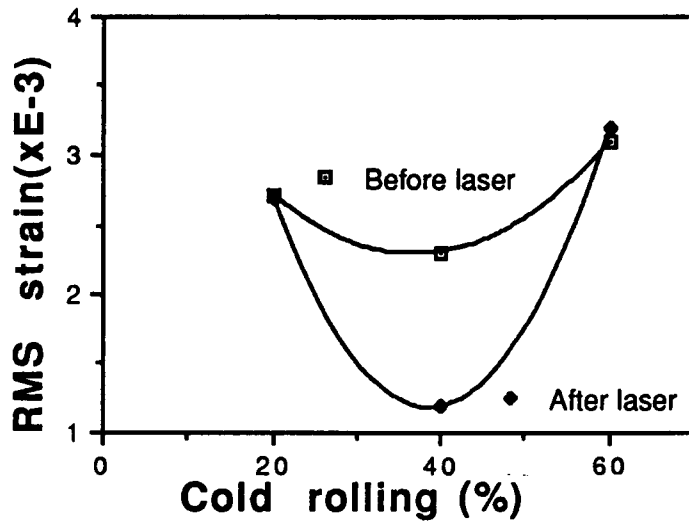


Fig.4.19. Variation of the RMS strain versus cold rolling rate before and after laser irradiation.

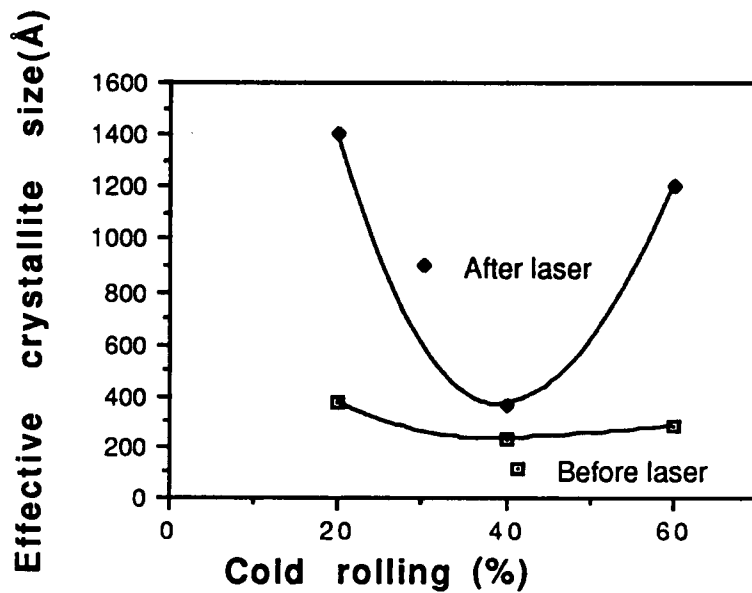


Fig.4.20. Variation of the effective crystallite size versus cold rolling rate before and after laser irradiation.

Thus, we can say that for the cold rolled specimen the laser irradiation increases the effective crystallite size and does not change the RMS strains to a significant amount.

#### 4.3.4. Dislocation density analysis

There are two different ways in which the dislocation density can be calculated;

- i) using crystallite size
- ii) using the RMS strain.

The dislocation density values calculated from the crystallite size ( $D_d$ ) and RMS strains ( $D_s$ ) are shown in Table 4.5.

The ratio of  $D_s/D_d$  as a function of the laser energy density is plotted in Fig.4.21 for laser treated pre-annealed samples. For the lower energy densities, the dislocations are mostly generated by the lattice strains. For the higher laser energy densities, the contribution to the dislocation density due to the small size of the crystallites is more important than the lattice strain contribution.

The dislocation density ratio between dislocation densities before and after laser treatment against cold rolling rate for cold rolled specimens is shown in Fig.4.22. The lattice strain effect is approximately the same, but, the crystallite size effect decreases for all the specimens.

Table 4.6 lists the average dislocation density values ( $\sqrt{D_d \times D_s}$ ) obtained from the dislocations calculated from the

**Table 4.5**

Dislocation density (from RMS strain and small crystal size) variation before and after laser treatment

| Specimen description | Laser energy density (J/cm <sup>2</sup> ) | Dislocation density calculated from RMS strains (Ds) |             | Dislocation density change | Dislocation density calculated from Effective crystallite size (Dd) |             | Dislocation density change |
|----------------------|-------------------------------------------|------------------------------------------------------|-------------|----------------------------|---------------------------------------------------------------------|-------------|----------------------------|
|                      |                                           | Before laser                                         | After laser |                            | Before laser                                                        | After laser |                            |
| Annealed #1          | 2                                         |                                                      | 1.01E11     | 1.01E11                    |                                                                     | 9.56E6      | 9.56E6                     |
| Annealed #2          | 3                                         |                                                      | 2.69E11     | 2.69E11                    |                                                                     | 1.91E10     | 1.91E10                    |
| Annealed #3          | 3.5                                       |                                                      | 6.20E11     | 6.20E11                    |                                                                     | 7.64E9      | 7.64E9                     |
| Annealed #4          | 4                                         |                                                      | 8.33E9      | 8.33E9                     |                                                                     | 7.5E10      | 7.5E10                     |
| cold rolled 20%      | 4                                         | 2.11E11                                              | 2.20E11     | 9.0E9                      | 1.57E11                                                             | 1.01E10     | -1.47E11                   |
| cold rolled 40%      | 4                                         | 1.54E11                                              | 4.16E10     | -1.12E11                   | 4.51E11                                                             | 1.77E11     | -2.74E11                   |
| cold rolled 60%      | 4                                         | 2.78E11                                              | 3.02E11     | 2.4 E10                    | 2.73E11                                                             | 2.47E10     | -2.48E11                   |
|                      | 3                                         |                                                      | 2.73E11     | -5.0 E9                    |                                                                     | 5.15E10     | -2.21E11                   |

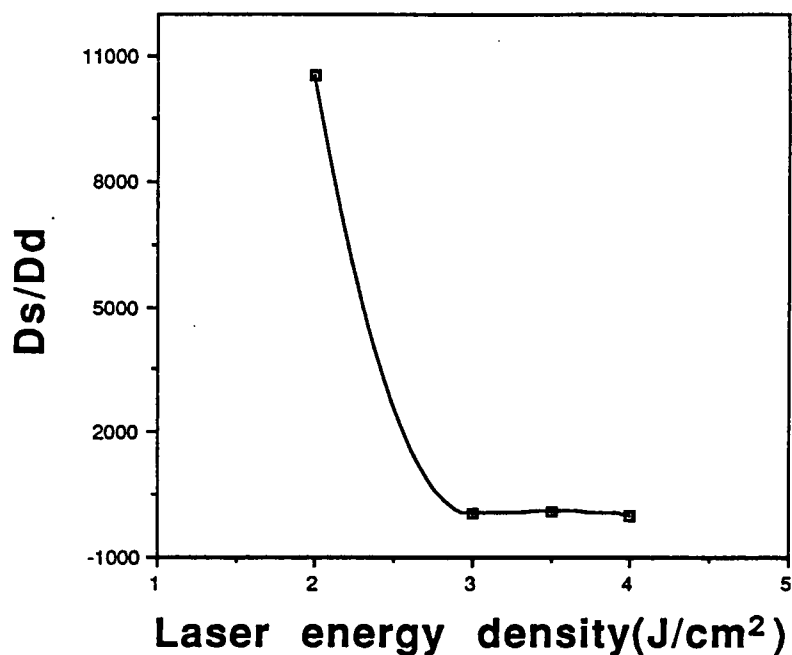


**Fig.4.21.** Plot of the  $D_s/D_d$  versus laser energy density ( $D_s$ , the dislocation density from the lattice strain;  $D_d$ , the dislocation density from crystallite size).

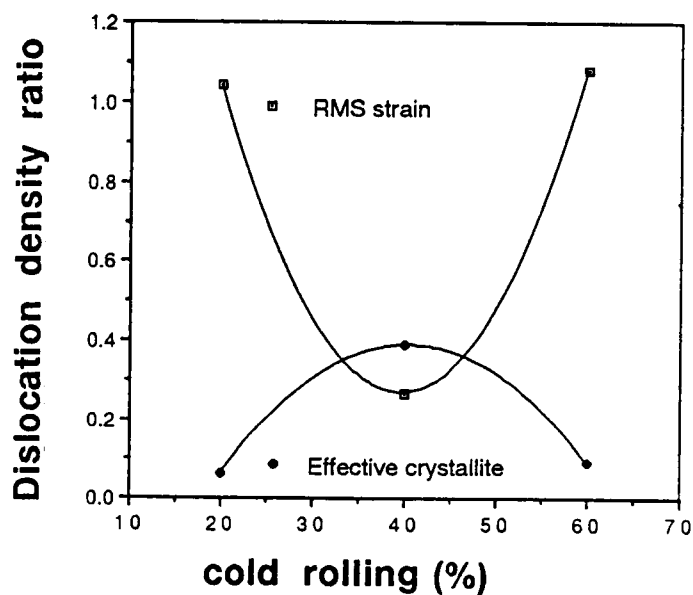


**Fig.4.22.** Plot of the dislocation density ratio (between dislocation densities before and after laser treatment) versus cold rolling rate in terms of RMS strain and effective crystallite size.

crystallite size( $D_d$ ) and RMS strains( $D_s$ ) for each specimen. Fig.4.23 thru Fig.4.26 are for the averaged dislocation densities.

Dislocation density was plotted against cold rolling rates before and after laser irradiation with an energy density of  $4\text{J}/\text{cm}^2$  in Fig.4.23 . For the cold rolled specimens the dislocation densities were increased with increasing cold rolling rates, while the dislocation densities were decreased after laser irradiation for all the cold rolled specimens.

The plot of the dislocation density change versus cold rolling is shown in Fig.4.24. Dislocation densities due to the laser treatment were decreased with an increase in cold rolling rates.

The dislocation density is plotted as a function of laser energy densities( $3\text{J}/\text{cm}^2$  and  $4\text{J}/\text{cm}^2$ ) for the 60% cold rolled specimen in Fig.4.25. The higher laser energy produces a greater reduction in the dislocation density.

Fig.4.26 shows that the dislocation density increases with an increase in the laser energy density for the annealed substrates.

#### **4.4 Microstructural view**

##### **4.4.1 Melting depth profile**

The cross-sectional views of the rapidly solidified surface layers due to laser-irradiation are shown in the

**Table 4.6**

Average dislocation density variation before and  
after laser treatment

| Specimen<br>description | Laser<br>energy<br>density<br>(J/cm <sup>2</sup> ) | Average dislocation<br>density ( $\sqrt{D_s \times D_d}$ ) |                | Dislocation<br>density<br>change |
|-------------------------|----------------------------------------------------|------------------------------------------------------------|----------------|----------------------------------|
|                         |                                                    | Before<br>laser                                            | After<br>laser |                                  |
| Annealed #1             | 2                                                  |                                                            | 9.79E8         | 9.79E8                           |
| Annealed #2             | 3                                                  |                                                            | 2.26E10        | 2.26E10                          |
| Annealed #3             | 3.5                                                |                                                            | 2.17E10        | 2.17E10                          |
| Annealed #4             | 4                                                  |                                                            | 2.49E10        | 2.49E10                          |
| cold rolled 20%         | 4                                                  | 1.8E11                                                     | 4.7E10         | -1.33E11                         |
| cold rolled 40%         | 4                                                  | 2.6E11                                                     | 8.55E10        | -1.75E11                         |
| cold rolled 60%         | 4                                                  | 2.76E11                                                    | 8.64E10        | -1.89E11                         |
|                         | 3                                                  |                                                            | 1.19E11        | -1.57E11                         |

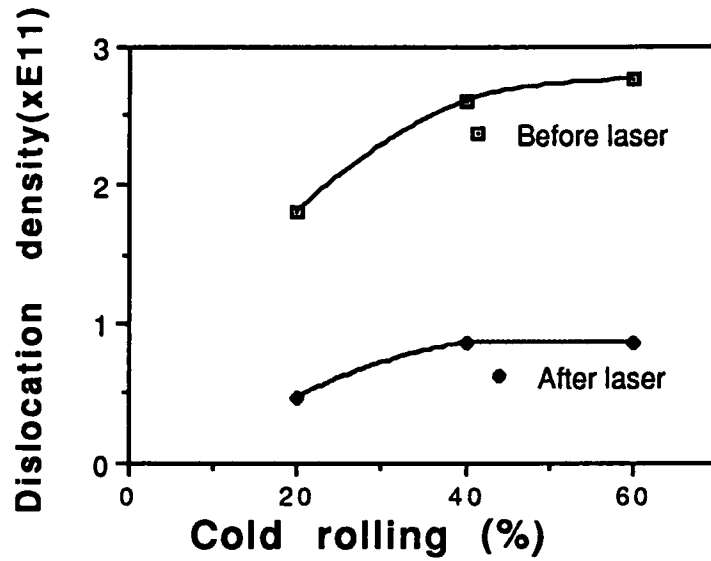


**Fig.4.23.** Plot of the dislocation density versus cold rolling rate before and after laser irradiation with 4J/cm<sup>2</sup> energy density.

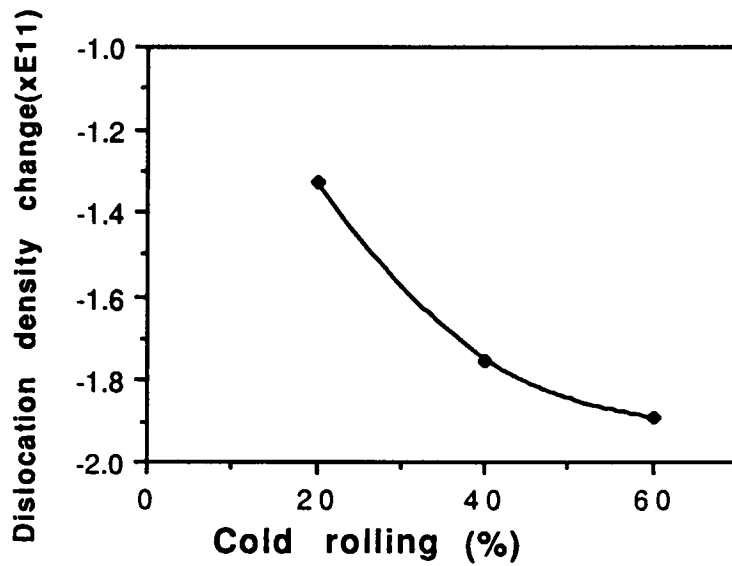


**Fig.4.24.** Plot of dislocation density change versus cold rolling rate due to laser treatment with 4J/cm<sup>2</sup> energy density.

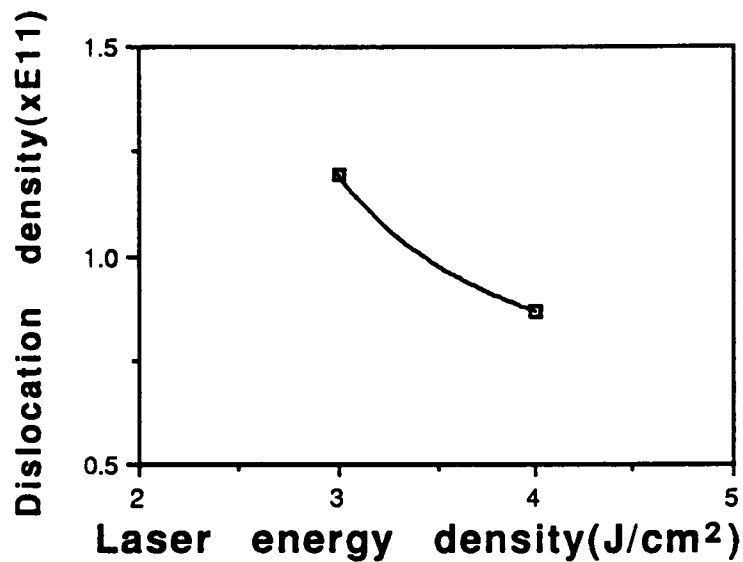


**Fig.4.25.** Plot of dislocation density versus laser energy density for 60% cold rolled specimen laser irradiated.

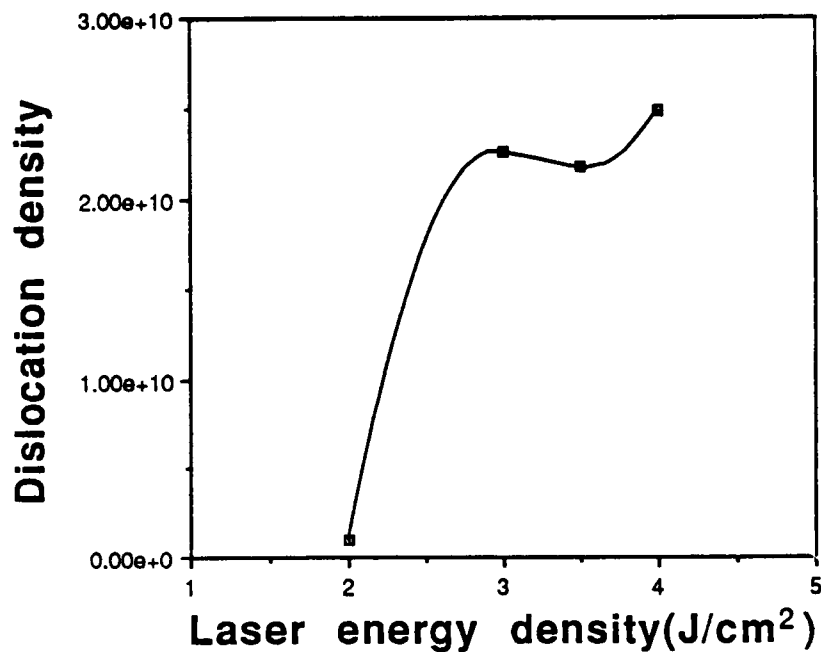


**Fig.4.26.** Plot of the the dislocation density vs. laser energy density for the annealed substrate.

Fig.4.27 (a) and (b). We can see that an average melt depth is approximately  $0.35\mu\text{m}$  and the thickness of the melting depth is non-uniform (Fig.4.27(b)).

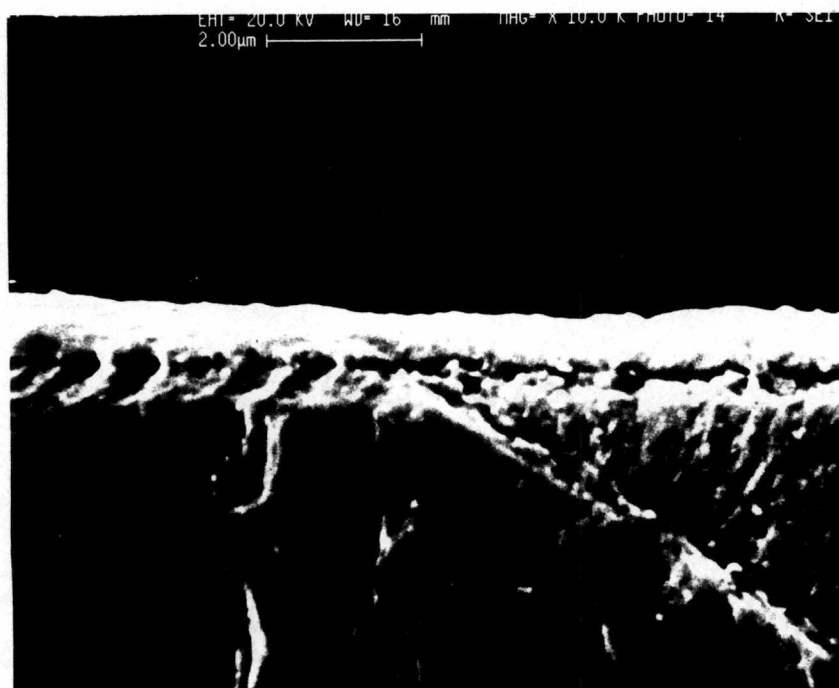
#### 4.4.2 Dislocation observation

Fig.4.28 shows dislocations in the as-received specimens and Fig.4.29 shows the dislocations in the laser affected zone. Some dislocations were seen in the as-received specimen. In the melted region no dislocation was seen, while numerous dislocations were found in the laser affected zone after laser-irradiation with an energy density of  $4\text{J}/\text{cm}^2$ .

#### 4.5 Results of the computer simulation for the melting depth and thermal gradient

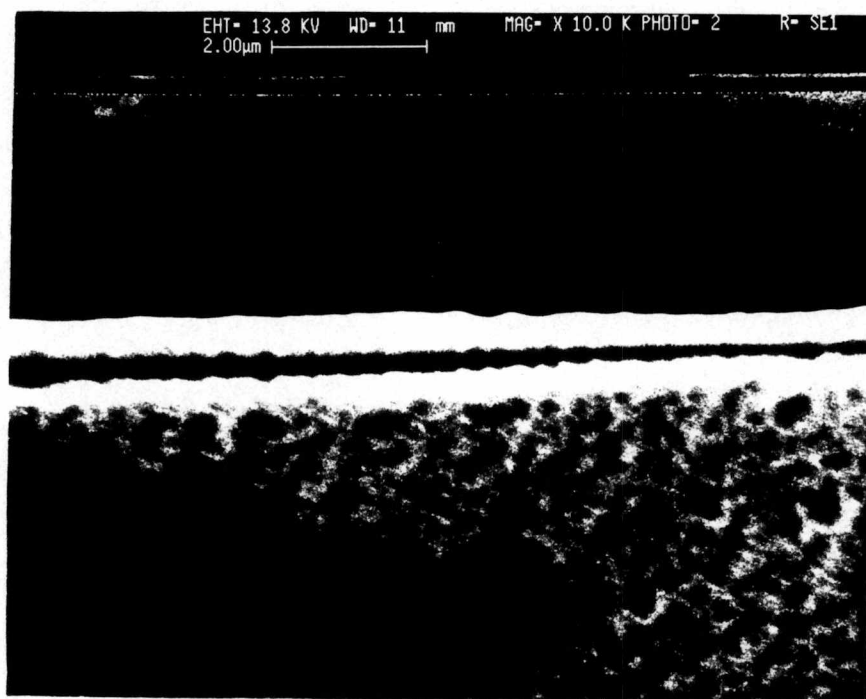
The plot of the melting and vaporization depth versus laser energy density is shown in the Fig.4.30. Vaporization depth increases continuously as the laser energy increases, while the melted depth are approximately constant.

Fig.4.31 shows temperature distributions as a function of the distance from the interface between liquid and solid for the various laser energy densities ( $2\text{J}/\text{cm}^2$ ,  $3\text{J}/\text{cm}^2$ ,  $3.5\text{J}/\text{cm}^2$  and  $4\text{J}/\text{cm}^2$ ). It appears that the laser affected zone depths are almost the same for the different energy densities.



(a)

**Fig.4.27.**Cross sectional view of the solidified surface layer during laser treatment with  $4\text{J}/\text{cm}^2$  energy density from SEM.



(b)

Fig.4.27. Continued



**Fig.4.28.** Dislocations from the as-received specimen  
from TEM.(x80000)

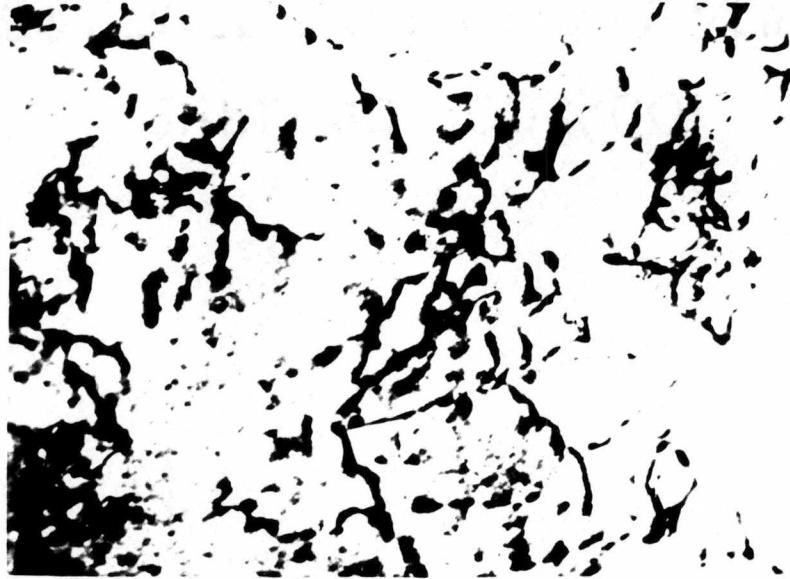


**Fig.4.29.** Dislocations in the laser affected zone from TEM after laser irradiation with  $4\text{J}/\text{cm}^2$  energy density ( $\times 80000$ )

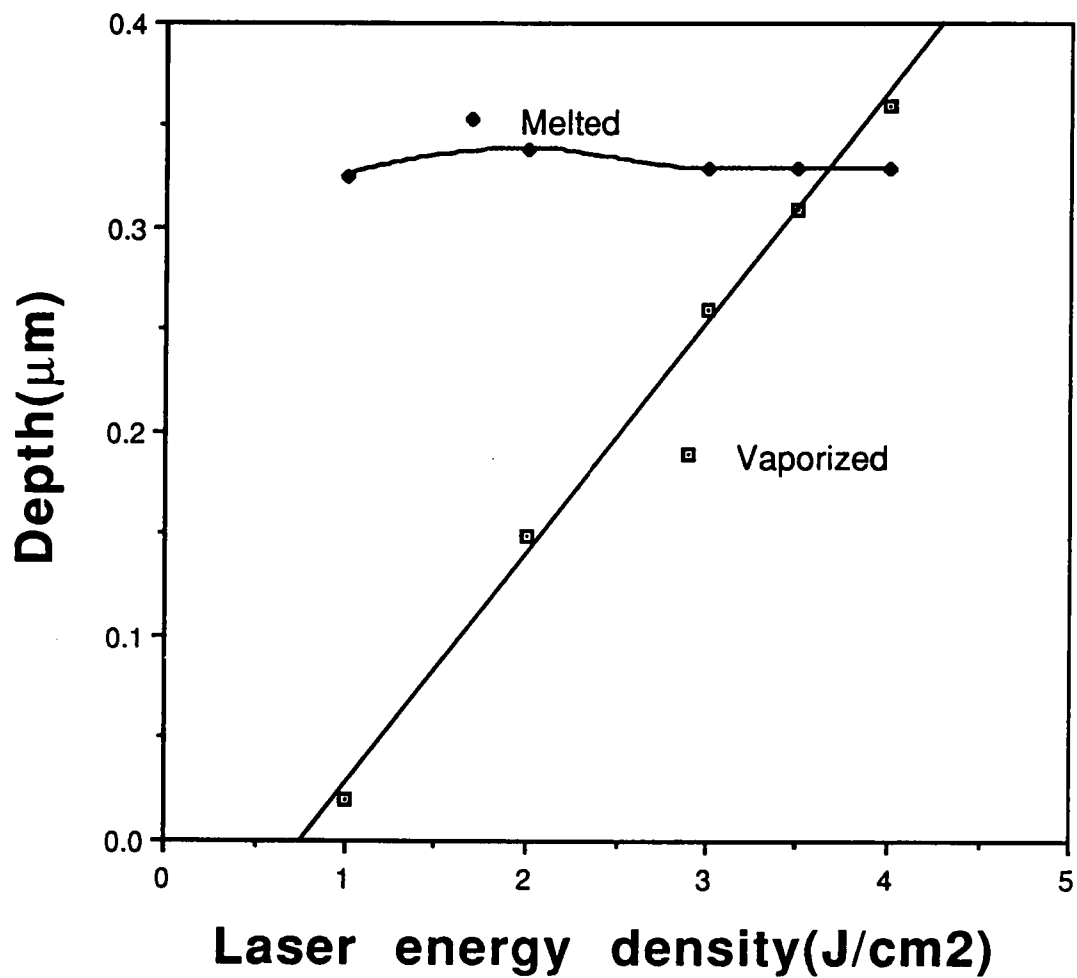


**Fig.4.30.** Plot of the melted depth and vaporized depth versus laser energy density.

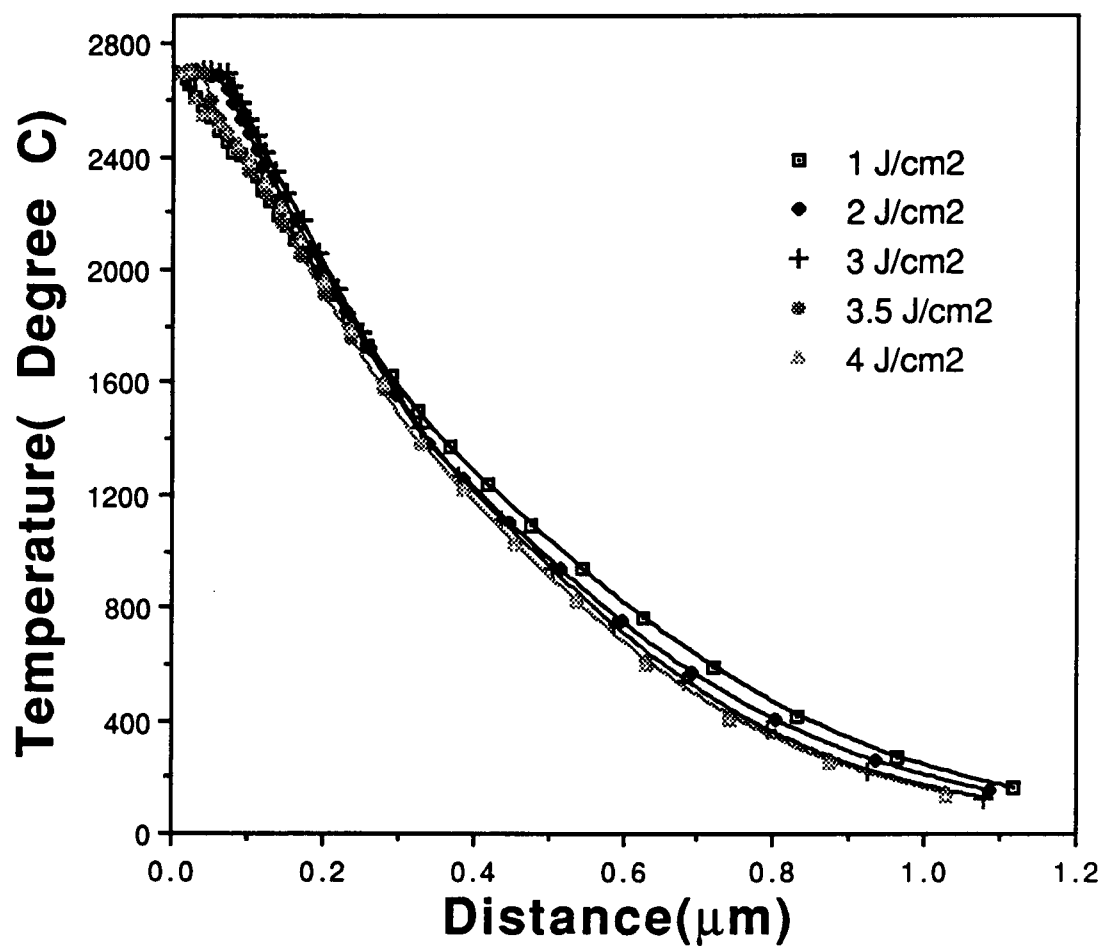


**Fig.4.31.**Plot of the temperature versus laser affected zone depth obtained from the computer simulation.

#### **4.6 Crystal structure of stainless steel deposited on austenitic stainless steel**

Fig.4.32 and 4.33 show the diffraction pattern of an annealed substrate before and after deposition. Fig.4.34 and 4.35 represent diffraction patterns of grown substrates before and after deposition. In both cases after sputter deposition the 200 BCC reflection appears, indicating the formation of a BCC phase in the deposited film.

In some experiments the oxide layer was chemically removed and the substrate was quickly inserted into the vacuum chamber for sputtering in order to prevent the formation of more oxide film. Film deposited in substrate where the oxide layer was removed had a FCC crystal structure.

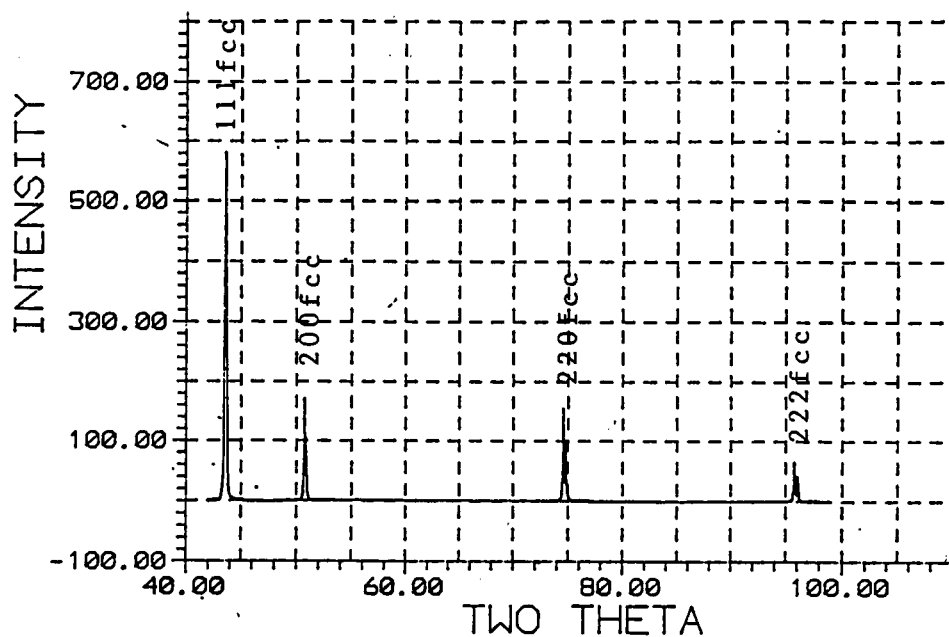


**Fig.4.32.** Diffraction pattern for an annealed substrate for the sputter deposition

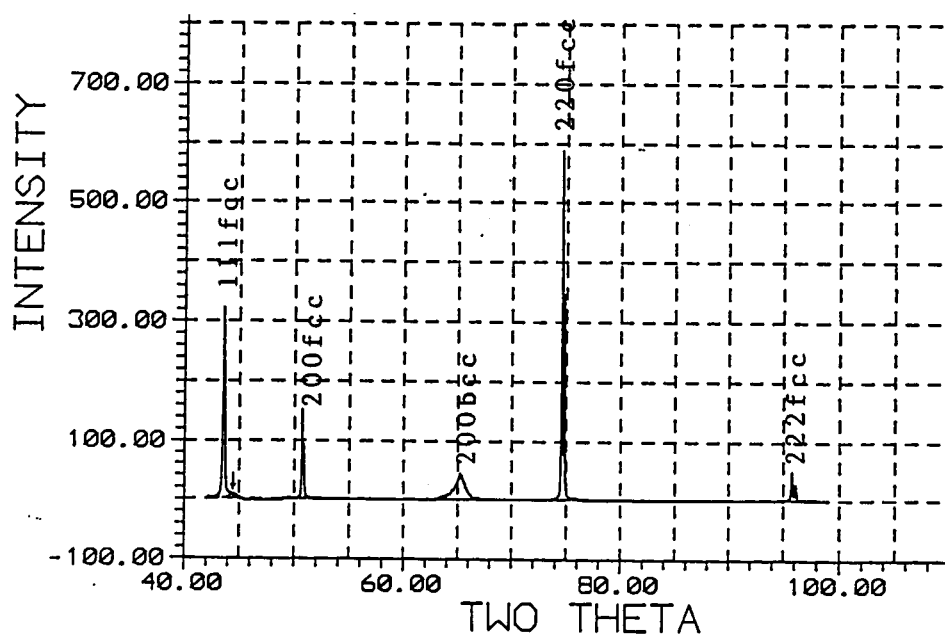


**Fig.4.33.** Diffraction pattern for a deposited film on a annealed substrate.

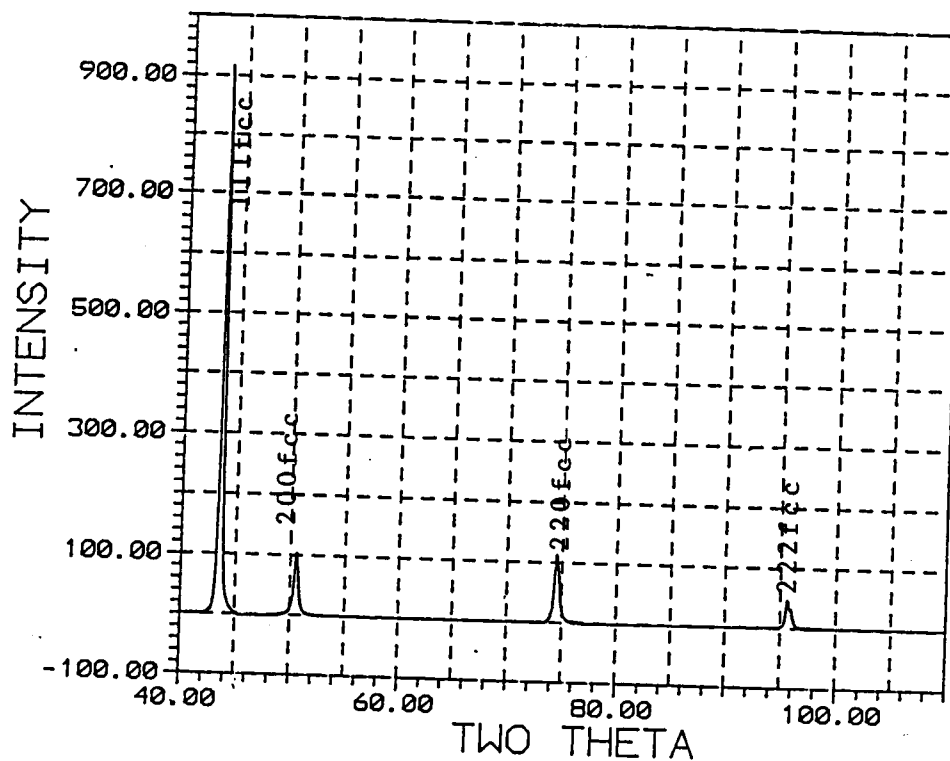


Fig.4.34. Diffraction pattern for a ground substrate after annealing

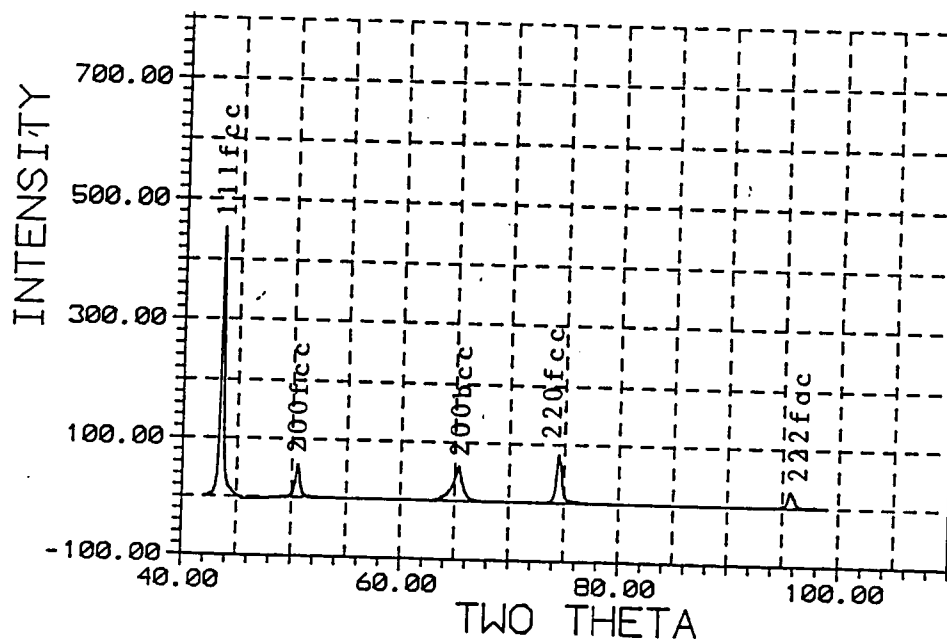


Fig.4.35. Diffraction pattern for a deposited film on a ground substrate.

## CHAPTER 5

### DISCUSSION

#### 5.1 Macro-stress analysis

##### 5.1.1 Residual stress during cold rolling operation

The rolling operation is a combination of a squeezing action and a frictional action. The specimen is forced into the rolls gap by frictional work that generates shear stresses on the surface of the specimen. Therefore, the surface is deformed more than the center of the rolled sheet. And, the surface residual stress should be compressive. However, in the case of 20% cold-rolled specimen, the surface is under tensile stress (Fig.4.5). This result indicates that during the rolling process the plastic flow in the surface was less than in the center. For 40% and 60% cold rolled specimens the residual stresses are compressive in nature. The distribution of the stresses during the rolling operation is a function of a number of variables such as the specimen thickness, the roll diameter and the degree of reduction[64].

##### 5.1.2 The effects of laser irradiation on the surface normal component of the residual stress

In the biaxial stress condition the possible " $d$ " vs.  $\sin^2\psi$  plots are a straight line (Fig.2.8(a)) and in the existence of the shear stresses in the surface normal

direction  $\psi$  splitting in the "d" vs.  $\sin^2\psi$  plot can be obtained (Fig. 2.8(b)). Under the inhomogeneous stress condition we expect the oscillatory plot (Fig. 2.8(c)).

In the X-ray stress analysis, biaxial stress condition is usually assumed, because 70% of the integrated intensity comes from approximately  $2.8\mu\text{m}$  of the surface layer (420 reflection) (see Fig. 5.1). The diffracted area is approximately  $3.5 \times 10^7 \mu\text{m}^2$  (Fig. 5.2) and the ratio of the area to the penetration depth is about  $1.24 \times 10^7$ . Moreover, the tilting of the specimen axis produces a lower depth of penetration of the X-ray resulting in even a larger ratio of the area to the penetration depth. Under these conditions, in the diffracted volume plane stress condition can be assumed ( $\sigma_{33} = \sigma_{13} = \sigma_{23} = 0$ ).

There is a slight oscillation in the "d" vs.  $\sin^2\psi$  plot as a result of cold rolling as shown in Fig. 4.5. In the polycrystalline materials we can always expect such a slight oscillatory "d" vs.  $\sin^2\psi$  plot as a result of plastic deformation due to non-uniform plastic deformation and elastic incompatibility that are a function of the texture and the grain shape. Shear stresses in the surface normal direction due to cold rolling can also give rise to the splitting in "d" vs.  $\sin^2\psi$ . However, in this case the stress variation in the surface normal direction due to cold rolling is almost negligible in the diffraction layer.

After laser treatment the oscillation in the data for a certain specimen is larger than that of cold rolling as shown in Fig.4.6. This indicates that as a result of the laser treatment significant inhomogeneous stresses and elastic incompatibility took place even in the diffraction volume.

At equal level of stresses the amount of plastic deformation is a function of sample temperature. The portions of the specimen located far from the surface will deform less than those closer to the surface. The thermal gradient is extremely steep ( $10^7 \text{ }^\circ\text{K/cm}$ ) and will produce non-uniform plastic deformation. It can be concluded that the extremely steep gradient generated during laser processing will produce non-uniform residual stresses. Another effect on the non-uniform plastic deformation may be the non-uniform cross-sectional distributions of the laser beam energy during laser irradiation.

In both cold rolling and laser irradiation, in general we can also consider anisotropic properties of material on the oscillatory "d" vs.  $\sin^2\psi$  plot. However, non uniform plastic deformation and elastic incompatibility due to the anisotropic property of a material may not cause such a big oscillatory plot as shown in Fig.4.6 unless the orientations of the grains are extremely preferred.

Noyan et al. have recently tried to interpret such a oscillatory data (denoting non-uniform stress distribution in the material) by a computer simulation in terms of local

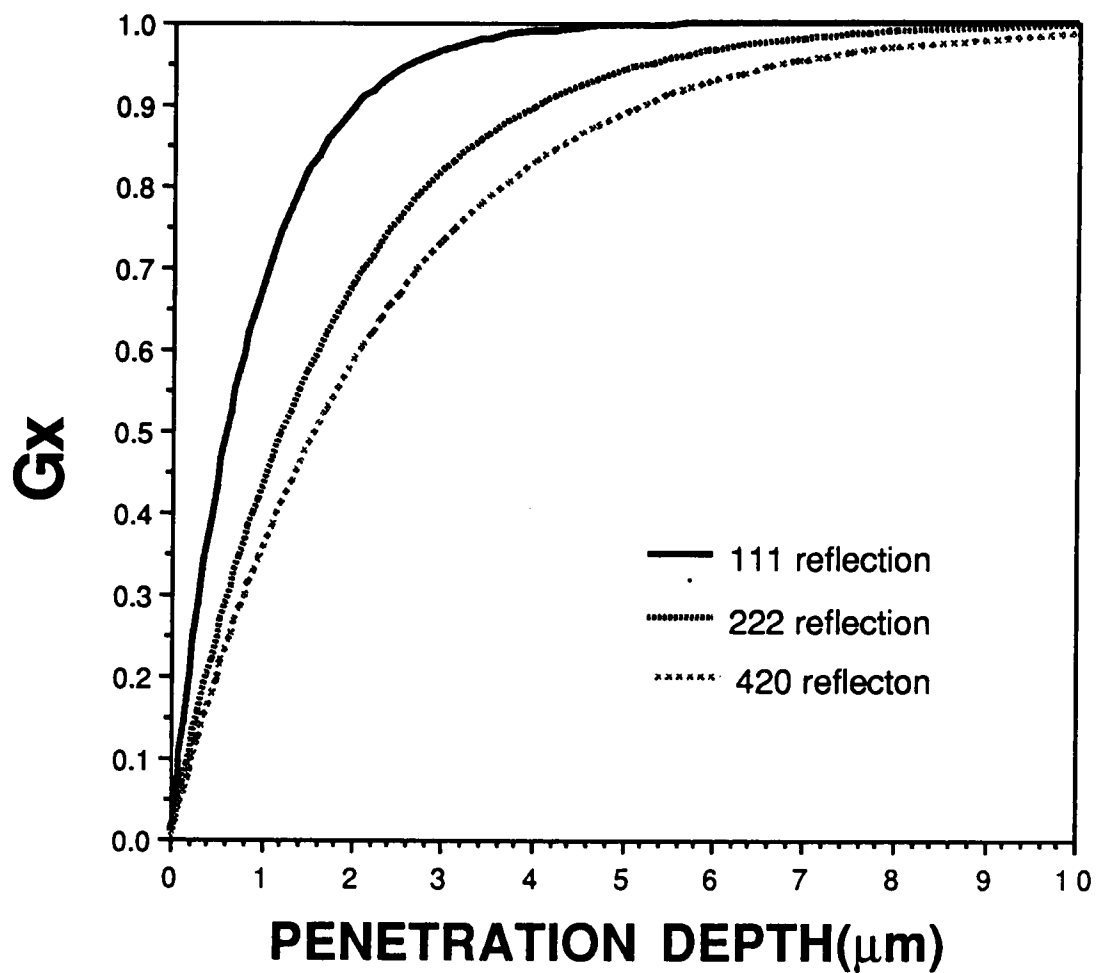
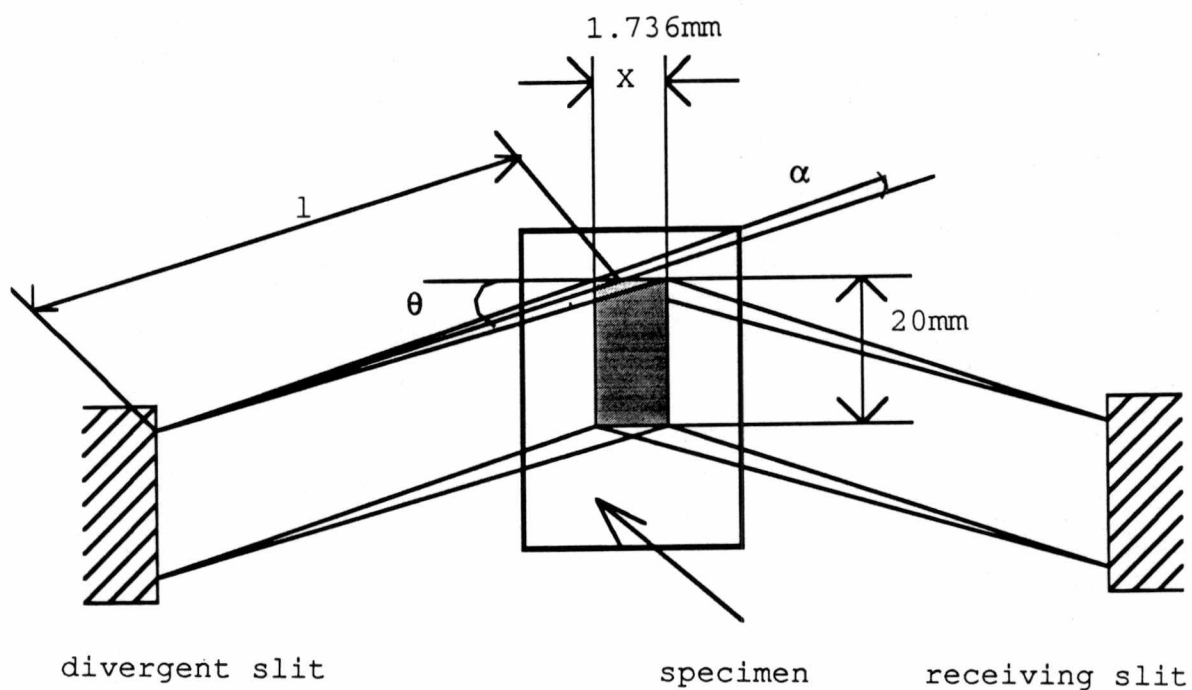


**Fig.5.1.** Plot of the fraction of the total integrated intensity (Gx) versus X-ray penetration depth( $\mu\text{m}$ ) for 316L stainless steel.



diffraction area =  $3.472 \times 10^7 \mu\text{m}^2$

formulae:

$X = (1/\sin(\theta+\alpha) + 1/\sin(\theta-\alpha)) l \sin\alpha + \text{aperture width}$   
 width(=0.05mm for  $1/2^\circ$  divergence slit)

$2\alpha = \text{divergent angle} (\alpha=0.25)$

$\theta = \text{incident beam angle} (=73.35^\circ; \text{bragg's angle})$

$l = \text{Goniometer radius} (=185\text{mm})$

**Fig.5.2.**X-ray diffracting area for 420 reflection.

yielding (non-uniform plastic deformation) [65].

The actual residual stress states in the sample that is possible with interpretation of oscillatory data are very complicate.

In this research the least squares linear fitting methods wrer thus used for all the case, and determined residual stress values are, therefore, the average values in the diffraction volume.

#### 5.1.3 Residual stresses during laser treatment

It is clear from the experimental results that independently of the initial state of stresses or amount of cold work of the sample, laser processing always generates residual stresses that are tensile in nature. The magnitudes of the stresses are almost independent of the initial states of the samples. The way these residual stresses are induced in the steel is analyzed below.

1) During heating: Molten metal can not give any stress on the non-melted area. However, complex transient thermal stress can occur in a specimen due to the localized heating. During laser heating the surface is hotter than the interior and regions closer to the surface will tend to elongate more than regions that are deeper inside, depending on the thermal expansion coefficient of the materials. The inner layers always act as a constraint against the outer layer leaving the region closer to the surface in compression. The yield

strength of the surface will be lower than the inner layers due to the thermal gradient. When the thermal stress due to the constraining action of the interior exceeds the yield strength of the materials, the surface will plastically deform resulting in a decrease in the length. In such a way the surface and the interior tend to have the same length although the surface is at higher temperature during heating (Fig.5.3).

2) During cooling: If there is no plastic deformation during cooling, the elastically elongated layers due to the surface layer being at higher temperature will contract more than the internal layers. Therefore, the resultant surface residual stress will be tensile.

However, some plastic deformation during cooling reduces the magnitude of tensile stresses.

3) During solidifying: When the molten layer solidifies, it will shrink more than the base materials. However, the colder base materials will resist against its contracting action during solidifying. Since the whole body tends to keep a continuous body, the tensile residual stress in the solidified layer will occur at room temperature.

If plastic deformation takes place laser processing of material will always produce residual stresses that are tensile in nature

As shown in Fig.4.30 and Fig.4.31 , the calculated melted depth and the calculated thermal gradient change very little with laser energy density for the higher energy densities. Therefore, an increase in the laser energy density will result in an increase in the residual stress to a certain amount, but, above a certain level the amount of the residual stresses will be approximately constant as shown in Fig.4.8.

Fig.4.9 shows that residual macro-stresses after irradiation are always tensile independently of the initial state of the specimen (viz. annealed or cold rolled). The magnitude of the stress is also very similar for the different substrates irradiated at  $4\text{J}/\text{cm}^2$ .

The high temperature to which the samples are submitted during irradiation promotes the annealing of the structure. The residual stresses due to cold working will be relaxed by annealing effect. Therefore, at the end of heating process, up to a certain depth from the surface the material property of the cold rolled specimen may be very similar to that of the annealed substrate. During subsequent cooling, the behavior of the cold rolled substrate to induce the residual stress will be the same as discussed above. At room temperature the amount of the residual stresses for annealed and cold rolled specimen will tend to be closer to each other in the near surface when considerably high energy density is used (Fig.4.9). If there is any difference in the residual

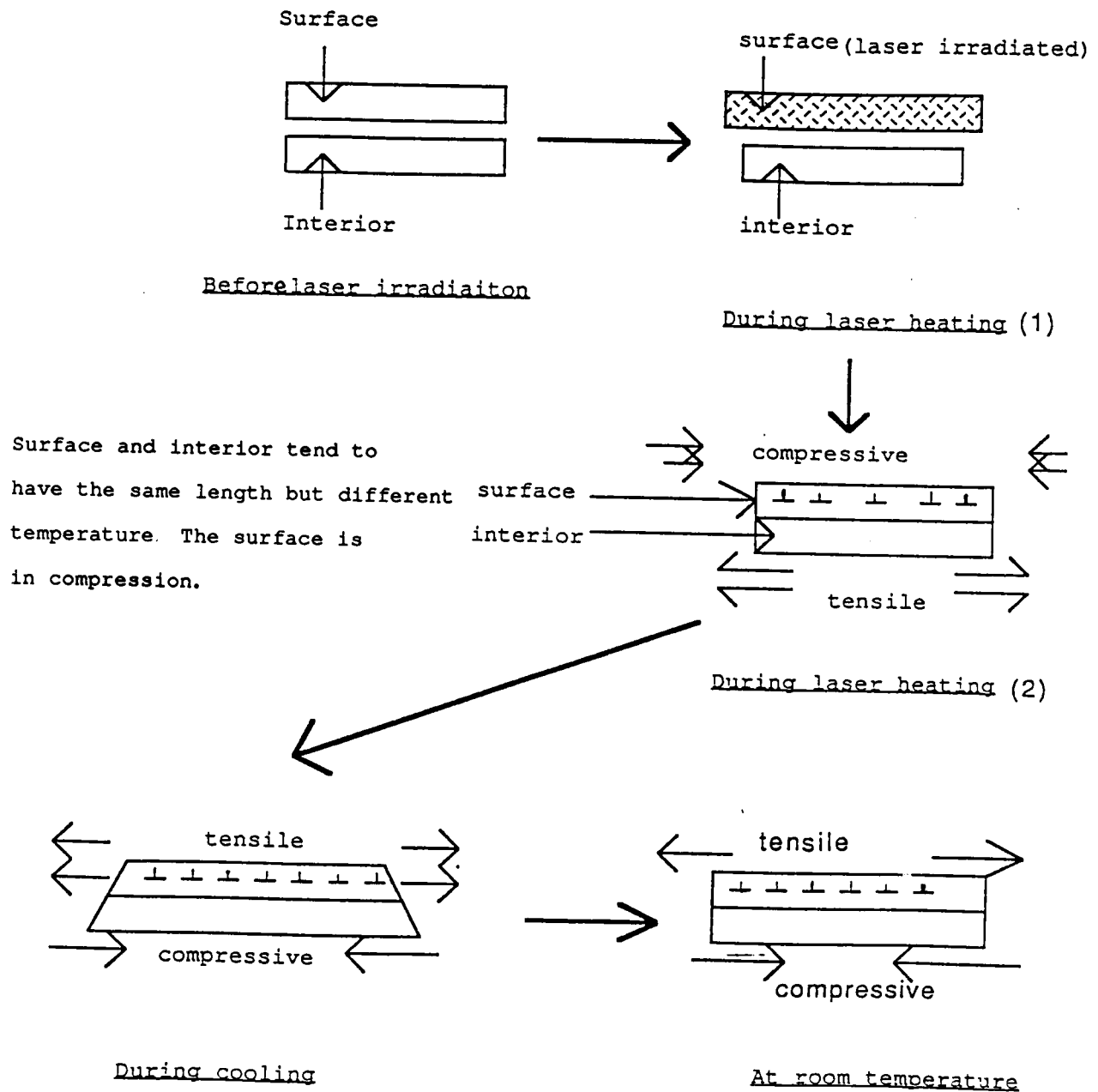


**Fig.5.3.** Schematic explanation of the process to induce the residual stress during laser processing.

stresses as a result of the X-ray measurement between different substrates (annealed or cold rolled), it will be when the diffracting volume covers up to the non-affected depth of the specimen as shown in Fig.5.4. 70 % integrated intensity for 420 reflection comes from near surface layer of  $2.8\mu\text{m}$ (Fig.5.1). From the computer simulation, the depth of the melted zone plus the laser affected zone of a specimen irradiated with an energy density of  $4\text{J}/\text{cm}^2$  energy density can be estimated as approximately  $1.35\mu\text{m}$  (Fig.4.30 and Fig.4.31). There is an unaffected region of  $1.45\mu\text{m}$  that contributes to the integrated intensity, but has not been affected by the laser irradiation. This contribution will reflect the initial conditions of the sample and is possibly the cause for the differences in residual stresses observed in the irradiated specimens.

The magnitudes of the residual stresses in the samples are somehow higher than the measured tensile stress of 316L stainless steel (TS ;  $620\text{MPa}$ , YS ;  $310\text{MPa}$  for cold finished specimen). This indicates that probably the mechanical properties of this thin layer are different from that of bulk specimens. In strong contrast with stainless steel no residual stresses were detected in the irradiated polycrystalline silicon specimen.

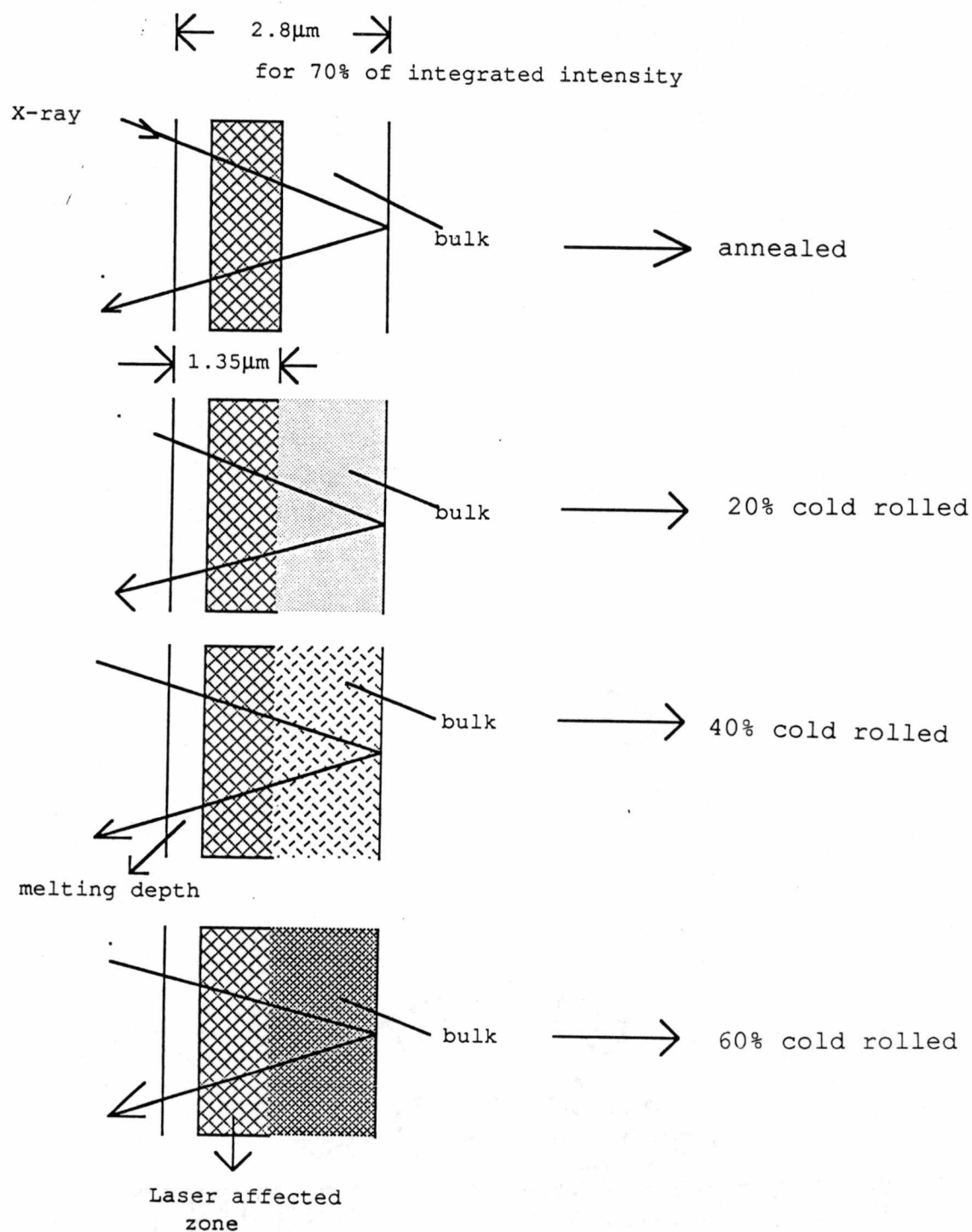


**Fig.5.4.** Schematic explanation of the stress deviations due to the different conditions of non-affected zone in the X-ray measurement .

## 5.2 Line broadening analysis

### 5.2.1 Variance and dislocation density

Line broadening of the X-ray reflections is due to the strain field of dislocations and dislocation arrays, and small domains with slight tilt each other (due to dislocation arrays, subgrain boundaries, etc.).

Laser irradiation of annealed samples produces significant broadening of the reflection lines (Table 4.2 and Fig.4.12). This is a clear indication that plastic deformation occurred during laser irradiation. On the other hand, a reduction in the line width taken place in cold rolled specimen indicates that an overall annealing took place during laser processing. Two main effects contribution to the broadening of the X-ray lines, the crystallite size and RMS strain. The RMS strains are in this case related mostly to the single distortion density. The crystallite size is determined by the clustering of dislocations in walls and the formation of the dislocation cells. To separate the two effects from the pure line broadening a Fourier analysis was performed on the line profile (Fig.4.16, table4.3 and table4.4). However, since the crystallite size ( $D_{eff}$ ) and the RMS strain have different units, we can not compare them directly to each other. A better way of comparing these two effects is by calculating the dislocation density that

promotes the RMS strain (lattice strain due to single dislocations) and the dislocation clustering in cells (crystallite size) (Fig.4.17 and Fig.4.18). The lattice strain is the main contribution to the line broadening in annealed substrates irradiated at low energy density ( $2\text{J}/\text{cm}^2$ ). For sample irradiated at an energy density of  $3\text{J}/\text{cm}^2$  and above the crystallite size effect was more important than the dislocation density on the line broadening (Fig.4.17 and Fig.4.18). At lower laser energy density single dislocations are generated. The dislocation density increases with increasing laser energy density but saturates at a given dislocation density (Fig.4.26).

For the cold rolled specimens, the dislocations from both small crystallite size and lattice strain contributed to the line broadening in equal portions except for the 40% cold rolled specimen (table 4.5). After laser processing, the lattice strain effect is more or less constant, while the size effect is much reduced (Fig.4.22). This means that the average crystallite sizes were increased due to laser processing in the case of the cold rolled specimens. Laser processing in cold rolled specimen also produced a reduction in the dislocation density (Fig.4.23).

For cold rolled specimens, the macro-stresses increased, while the dislocation density decreased after irradiation. Such an inconsistency can be explained in terms of X-ray penetration depth. For the 70% of integrated intensity the X-

ray penetration depths are approximately  $0.9\mu\text{m}$  for 111 reflection and  $2.1\mu\text{m}$  for 222 reflection, while for 420 reflection it is approximately  $2.8\mu\text{m}$  (Fig.5.1). This means that 111 and 222 reflection which were used in determining micro-residual stresses cover only up to the laser affected zone. On the other hand, as already shown, there is a contribution from the non-affected area to the integrated intensity of the 420 reflection. Assuming the melted layer is almost fully annealed, plastic deformation takes place around laser affected zone. 420 reflection covers up to the unaffected bulk. On the other hand 111 and 222 reflection cover only near the surface. Therefore, in 111 and 222 reflection, the annealing effect can be pronounced due to melted area.

In the cold rolled specimens higher laser energy density produced a more complete cell structure (Fig.4.25). This is consistent with the fact that dislocations tend to form cell structures in FCC materials.

#### 5.2.2. Cell structure effect

As shown in Table 4.5, the dislocation densities determined from the Deff and RMS strain are different from each other. That implies that there exists dislocation clustering, because dislocation clustering can be expected if the dislocation density values determined from Deff is

smaller than the values obtained from the RMS strains. If the dislocations are randomly arranged, these two should agree.

#### 5.2.3 Prior cold rolling effect on dislocation density change during laser processing

The more deformed specimen experienced more reduction in the dislocation density than the less deformed specimen as shown in Fig.4.24. In the case of the cold rolled specimen, the dislocation densities resulting from both the lattice strain and the crystallite sizes are not so different. This implies that the dislocations are somehow randomly arranged. However, after laser treatment, the dislocation density determined from the crystallite size is much smaller than that from the lattice strain. Therefore, it can be concluded that the annealing due to the laser heating caused the dislocations to cluster in the diffraction volume.

#### 5.2.4 Dislocations from the TEM

In agreement with the X-ray analysis, the thin films from annealed samples show a substantial increases in dislocation contents after laser irradiation ( Fig.4.28 and Fig.4.29). Therefore, TEM examination conforms that occurred in annealed specimen as a result of laser treatment.

### **5.3 The crystal structure of stainless steel thin film deposited on austenitic stainless steel**

According to the thermodynamic calculation the stable phase in austenitic stainless steel should be BCC rather than the usual FCC. The formation of a BCC phase in film deposited at room temperature is then consistent with those calculations. However, if the passive oxide layer is removed, the atom attached to the substrate on the crystal structure is the same as the substrate. These films have the FCC structure because, under this condition, nucleation of the BCC phase can not take place.

## CHAPTER 6

### CONCLUSIONS

1. Tensile residual macro-stresses were produced at the surface when austenitic stainless steel specimens were 20% cold rolled. In the 40% and 60% cold rolled samples the residual stresses were compressive in nature.

2. As a result of laser irradiation surface tensile residual stress developed for all the specimens independently of the initial specimen conditions (annealed or cold rolled).

3. The magnitude of the residual stresses in specimens irradiated with an energy density of  $4\text{J/cm}^2$  is very similar, independently of the initial conditions of the specimens.

4. The width of the X-ray reflection lines broadened when annealed samples are laser irradiated. This broadening is related to an increase in dislocation density.

5. Laser irradiation of cold rolled specimen produces a decrease in the line broadening indicating that an annealing process also takes place.

6. It can be concluded that the laser processing of austenitic stainless steel produces two effects: annealing

owing to the heating process and plastic deformation due to the extremely steep thermal gradient.

7. For irradiated annealed specimens, at lower energy density the line broadening was mainly caused by lattice strains, whereas for higher laser energy density the crystallite size effect was more predominant than the lattice strain effect on the line broadening.

8. In the cold rolled specimen case, lattice strain effect on the line broadening is almost constant before and after laser treatment, while the size effect on line broadening is reduced as a result of laser irradiation (except for 40% cold rolled specimen).

9. The line width of silicon did not change after laser processing indicating that very little or no plastic deformation took place.

10. In agreement with the previous results no macro-residual stresses were detected in the laser-irradiated silicon.

11. Stainless steel films deposited on austenitic stainless steel substrates have a BCC crystal structure.

However, if the passive oxide layer is removed the film grows as a homoepitaxial layer and has an FCC crystal structure.

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