

Metallographic Characterisation of Cold Worked Type 304 Austenitic Stainless Steels

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Executive Summary

The influence of cold work on the Stress Corrosion Cracking (SCC) susceptibility of Austenitic Stainless Steels (ASS) is an ongoing topic of research. While some workers have suggested that low levels of cold work are deleterious to SCC, others argue that high levels may be favourable to SCC under certain conditions.

The objective of this report is to quantify a number of key parameters associated with the influence of cold work on SCC susceptibility. The microstructures of ASS subjected to different deformation modes and different levels of cold work have been examined using a range of techniques: optical metallography was carried out to determine the grain size and elongation ratio; the X-ray diffraction technique was used to quantify the amount of strain-induced martensite and residual stress; and lastly, deformation texture was examined using Electron Back Scatter Diffraction (EBSD) technique. In addition, Vickers hardness was used to quantify the material hardness in each condition of cold work.

The outcome of the characterisation showed that the Vickers hardness and the volume fraction of strain induced martensite increases with both increasing levels of cold work and weight percent of carbon in the material composition. The stability of delta ferrite was also affected by the proportion of ferrite-stabilising elements (e.g. Si) in the composition. Bi-rolling (i.e. cross-rolling) significantly increased the hardness value and the amount of strain induced martensite compared with samples in which cold work was generated either by uniaxial rolling and pulling (i.e. tensile cycling). Cross-rolling also led to a more uniform distribution of delta-ferrite within the microstructure than either unidirectional rolling or pulling.

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1 Introduction

The performance of Austenitic Stainless Steels (ASS) in Pressurised Water Reactor (PWR) environment is considered satisfactory. However, there have been reported cases of Intergranular Stress Corrosion Cracking (IGSCC) under the laboratory test conditions as well as in the actual reactor plant. This has been attributed to factors including neutron irradiation even though the exact cause of SCC of irradiated materials in PWR is yet to be fully established. Reports have suggested that initiation of SCC in PWR coolant can be influenced by the degree of localised plastic deformation, including cold work under some specific conditions. And in recent time, studies on a simulated PWR coolant also suggested that cold work can significantly influence the propagation of IGSCC [1, 2, 3].

The objective of this report is to quantify a number of key parameters associated with the influence of cold work on SCC susceptibility. The microstructures of ASS subjected to different deformation modes and different levels of cold work have been examined using a range of techniques: optical metallography was carried out to determine the grain size and elongation ratio; the X-ray diffraction technique was used to quantify the amount of strain-induced martensite and residual stress; Vickers hardness was used to quantify the material hardness in each condition of cold work and lastly, deformation texture was examined using Electron Back Scatter Diffraction (EBSD) technique.

The report is structured into the following main chapters namely: introduction; experimentation; results of experiments; discussions of the results and finally; conclusion of the report.

2 Experimental

2.1 Materials

The specimens used for the characterisation were Compact Test (CT) specimens obtained from Serco Assurance, Risley. They were made from four different groups of ASS Type 304 materials namely: Heat G, Heat D, Heat B and Heat C. The chemical compositions of these materials are shown in Table 1.

Specimen Q032 was obtained from Heat G. There was no any prior knowledge of cold work specimen Q032 had been subjected to and it was therefore used as reference specimen. The second group comprised of three specimens obtained from Heat D namely: R592, R585 and R591. They have been cold worked by pulling to 5%, 10% and 30% reduction respectively. The third group of the specimen comprised of R583 and Q114 obtained from Heat B. They have been cold worked by bi-directional rolling to total reduction of 20% and 35% respectively. And lastly, Specimen Q016 and Q021 were obtained from Heat C. Q016 have been cold worked by bi-directional rolling to total reduction of 20%, while Q021 was cold worked by unidirectional rolling to 40% reduction.

Table 2 contains additional details which include: the specimen's orientation, the 3D dimensions (Longitudinal, L Transverse, T and Short transverse, S) of the CT specimens as received and the origin of the specimen. The orientations of the CT specimens used in the report include: Semi orientation, across orientation and along orientation, which are equivalent to L-T, L-S and S-L respectively in ASTM notation (Figure 1).

Table 1: Chemical Composition of ASS Type 304 Materials

Chemical Composition							
Material	C	Si	Mn	Cr	Ni	S	P
Heat G	0.059	0.34	1.47	18.28	8.56	0.003	0.023
Heat D	0.045	0.32	1.75	18.15	9.05	0.001	0.025
Heat B	0.043	0.43	1.35	18.15	8.54	0.004	0.022
Heat C	0.026	0.42	1.08	18.2	9.16	0.021	0.02

Table 2: Summary of the Specimens Details

Specimen Details						
Material	Specimen	%CW	Deformation Method	Orientation (ASTM)	Cut From Worked Master	Dimensions L x T x S (mm)
Heat G	Q032	0%	No Cold Work	N/A	N/A	30 x 60 x 25
Heat D	R592	5%	Cold Pulled	Along (S-L)	N/A Uniform bar	30 x 10 x 5
	R585	10%	Cold Pulled	Along (S-L)	N/A Uniform bar	30 x 10 x 5
	R591	30%	Cold Pulled	Along (S-L)	N/A Uniform bar	12 x 12 x 12
Heat B	R583	20%	Cold Bi-Rolled	Across (L-S)	Asymmetrical Offset	5 x 12 x 30
	Q114	35%	Cold Bi-Rolled	Semi (L-T)	Small Skim from master	5 x 30 x 12
Heat C	Q016	20%	Cold Bi-Rolled	Semi (L-T)	Asymmetrical Offset	22 x 60 x 12
	Q021	40%	Cold Uni-Rolled	Semi (L-T)	Small Skim from master	22 x 60 x 12

2.2 Test methods

Characterisation of the test materials were carried out using a range of experimental techniques which includes: Vickers Hardness Measurement, X-Ray Diffraction (XRD), Optical Metallography and Electron Back Scatter Diffraction (EBSD). The experimental procedure of each of these techniques is given as follow.

2.2.1 Vickers Hardness Measurement

Sample preparation was carried out following the basic metallographic wet grinding with silicon carbide paper in order of 320, 400, 600, 800, 1200 and 4000. They were subsequently

polished with diamond pastes in order of 3 μ m and 1 μ m. Vickers Hardness measurement was carried using a load of 30Kg along three parallel lines, which were spacious enough to minimise the effect of localised plastic strain, on the Short-transverse, S (through thickness) direction within each specimen. Measurements along each line were made at 1 mm intervals.

2.2.2 X-Ray Diffraction

The specimens for XRD were prepared by standard metallographic wet grinding as explained in subsection 2.2.1 and were subsequently electropolished in a solution containing 8% perchloric acid and 92% acetic acid for 2 minutes at 42 V according to ASTM Standard practice E 1558 – 99 [4]. X-ray diffraction and Data acquisition for the quantification of Strain Induced Martensite (SIM) were carried out with Philips X’Pert-1 Diffractometer using the Cobalt radiation tube. X-ray intensities were measured at Bragg angle 2θ between 40° and 130° and at step size of 0.05°. The austenite-ferrite ratio was evaluated from integrated intensities (area under the peaks) of the austenite and ferrite peaks according to ASTM Standard practice E 975 - 03 [5].

Residual stress in the specimens was also determined by $\sin^2\psi$ method using portable diffractometer with chromium radiation tube. Data acquisition and analysis were carried out by Croto Residual Stress Analyser. The longitudinal residual stress measurement was carried out on the S-direction direction (through thickness).

2.2.3 Optical Metallography

Metallography samples with dimensions 10 x 10 x 10 mm (L x T x S) were sectioned from the as received CT specimens for Three Dimensional (3D) metallography with the exception of specimens R583 and Q114 where because of their size limitations, only 4 x 10 x 10 mm was obtainable for sample preparation. The 3D planes were named as T-L, S-L and S-T plane using the 3 perpendicular axes of the plate shown in Figure 1.

The sample preparation was consistent with the usual wet grinding and polishing mentioned in subsection 2.2.1. Three rounds of metallographic studies were carried out using different etchants as shown in Table 3. The grain boundary was electrolytically etched with 10% oxalic acid solution for the purpose of determination of grain size and elongation ratio. Determination of average grain size was done following the procedure of Hilliard single cycle intercept outlined in ASTM Standard E 112-96⁶¹ [6]. Electrolytic etching to reveal delta (δ -ferrite) ferrite was also done with sodium hydroxide solution and strain induced martensite was etched with picral by complete immersion. 3D optical micrographs of the specimens were taken after each of the etchants used.

Table 3: Table of Etchant [7]

Etchant	Concentration	Etching Condition	Observation
Oxalic Acid	10g Oxalic acid + 90ml distil water	Electrolytic etching for 1-1.5minute at 6V	Revealed grain boundaries and twinning
Sodium Hydroxide	40g Sodium Hydroxide + 100ml distil water	Electrolytic etching for 10s at 5V	δ -ferrite coloured light brown or bluish-grey. σ -phase coloured orange-brown
Picral	1g Picric acid + 5ml Hydrochloric acid + 100ml distil water	Complete Immersion for 4-5 minutes	Martensite appears in dark patches and austenite appears lightened

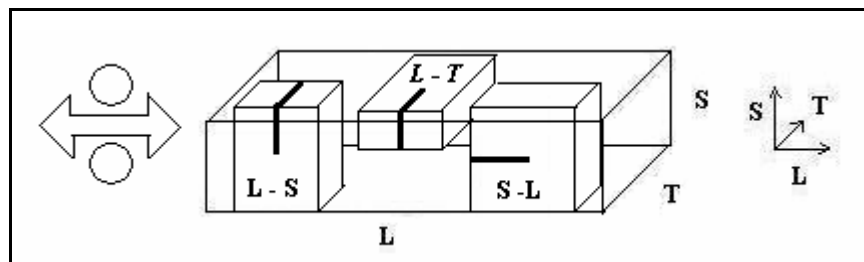


Figure 1: ASTM Notation for Specimen Extracted from Rolled Plate and Forgings [8]

2.2.4 Electron Back Scatter Diffraction

The samples used for Electron Back Scatter Diffraction (EBSD) technique was similar in dimension to those used for optical metallography (Subsection 2.2.3). Sample preparation included wet grinding and electropolishing as explained in Subsection 2.2.2. CamScan Field Emission Gun Scanning Electron Microscope (FEGSEM) in conjunction with Channel 5

Flamenco data acquisition software was used for EBSD imaging and data acquisition. Data were acquired for texture at variable step sizes between 4 and 8 μm . The samples were indexed as FCC and BCC Iron. However, some heavily cold worked specimens were poorly indexed probably because of the large plastic strain within the grain structure. Specimen Q114 could not be indexed at all.

Texture data obtained from Flamenco were subsequently processed into pole figures with Mambo software in Channel 5. The orientation information in unit reference sphere is represented in equatorial 2D planes by equal area projection where poles are projected from reference sphere into pole figures. Two complete pole figures were obtained from austenite phase on S-L and S-T planes of the specimen (Figure 1). The two pole figures were obtained using crystallographic planes $\{110\}$ and $\{100\}$.

3 Results

The results of experiments used for the characterisation of the cold worked specimens are categorised into four groups namely: Heat G, Heat D, Heat B and Heat C. And, the sequence of the results followed the same order of experimental techniques in Section 2 of the report.

3.1 Characterisation of Heat G

Heat G material contained the highest carbon contents of 0.059wt% and relatively low Si content of 0.34wt% as shown in Table 1. Only specimen Q032 was made from Heat G. The results of experimental techniques carried out on the characterisation of Heat G are as follow.

3.1.1 Vickers Hardness

3.1.1.1 Specimen Q032

Hardness values obtained from the 3 parallel lines along the through thickness (S) direction is shown as HV30 at line 1, 2 and 3 (Table 4). The mean value of the 3 results is shown in Mean HV30 column. The hardness values slightly dropped close to the edges with the margin of about 10HV30. The average hardness value across the through thickness was 222HV30. The plot of Vickers hardness results against the through thickness is shown in Figure 2.

Table 4: Vickers Hardness of Specimen Q 032 with 0% CW

Through Thickness Distance (mm)	HV30 @ 1	HV30 @ 2	HV30 @ 3	Mean HV30
1	213	199	211	208
2	232	216	211	220
3	234	214	223	224
4	233	219	213	222
5	235	225	212	224
6	238	224	207	223
7	237	224	215	225
8	241	222	230	231
9	235	212	223	223
10	229	207	230	222

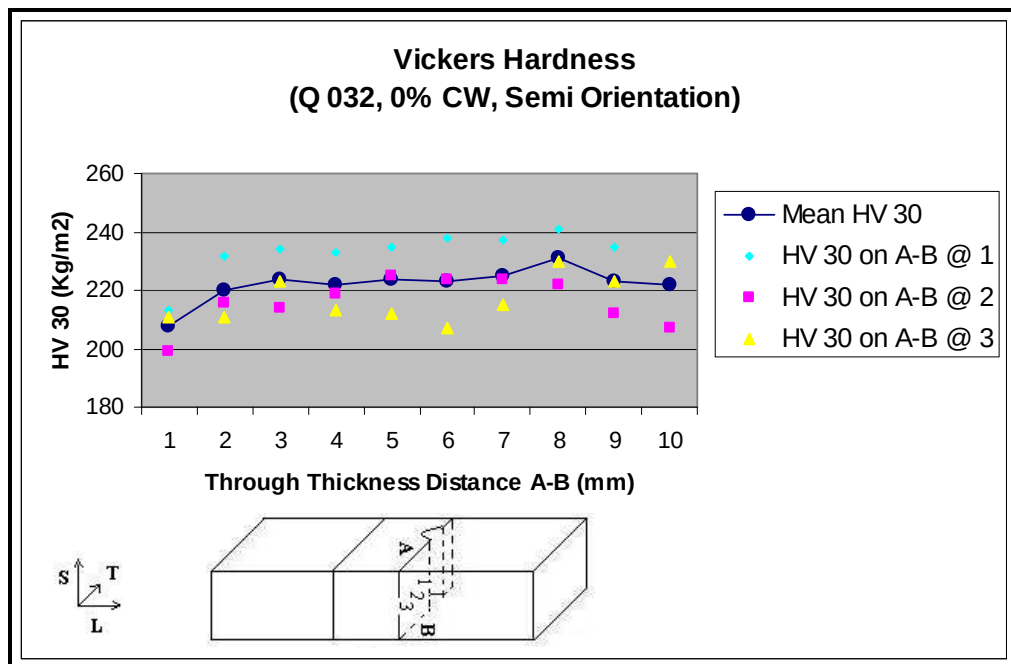


Figure 2: Vickers Hardness of Specimen Q 032 with 0% CW

3.1.2 X-Ray Diffraction (Strain Induced Martensite)

3.1.2.1 Specimen Q032

The result of quantification of Strain Induced Martensite (SIM) is shown in Table 5. Table 5 shows the integrated intensities obtained from the austenite and ferrite crystallographic planes. There was no knowledge of any previous cold work (CW) specimen Q032 had been subjected to and it was therefore assigned 0% CW. However, SIM of about 1% was still found in specimen Q032. The volume fraction of austenite, V_γ in the specimen was about 0.9 (equivalent to 90%).

Table 5: Quantification of Strain Induced Martensite in Specimen Q032 with 0% CW

Sample s	Cold Work (%)	Integrated Intensity, <i>I</i>							V_γ	Martensit e (%)
		γ -200	α -200	γ -220	α -211	γ -311	γ -222	α -220		
Q032	0	2383 2	143	1515 6	190	1562 1	4094	166	0.985 5	1

3.1.3 X-Ray Diffraction (Residual Stress)

3.1.3.1 Specimen Q032

The results of longitudinal residual stress measured along the through thickness of specimen Q032 is shown in Table 6 and Figure 3. Residual stress values varied across the through thickness from compressive residual stress value of -83.54MPa to tensile residual stress of 53.07MPa. The error values were below the acceptable value of 50MPa with the exception of both edges which correspond to Number 1 and 5 in Table 6.

Table 6: Residual Stress of Specimen Q032 with 0% CW

No	X-Position (mm)	Y-Position (mm)	Phi-Position (deg.)	Stress (MPa)	Error
1	0	0	0	-83.547	53.202
2	0	2	0	-1.19	29.057
3	0	4	0	-20.314	38.803
4	0	6	0	53.071	29.063
5	0	8	0	48.057	56.152

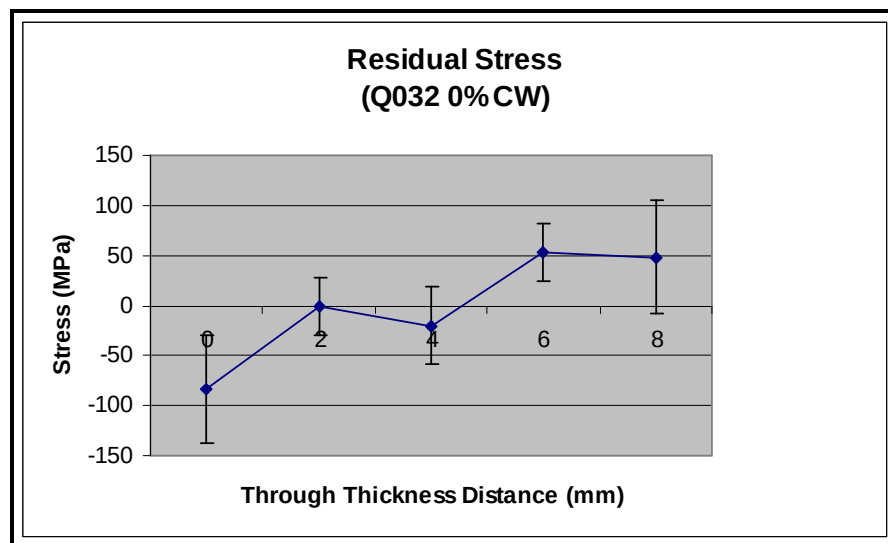


Figure 3: Residual Stress of Specimen Q032 with 0% CW

3.1.4 Optical Metallography (Grain Boundaries Etching)

3.1.4.1 Specimen Q032

The results of grain boundaries etching of specimen Q032 with 10% Oxalic acid solution is shown in Figure 4A-D.

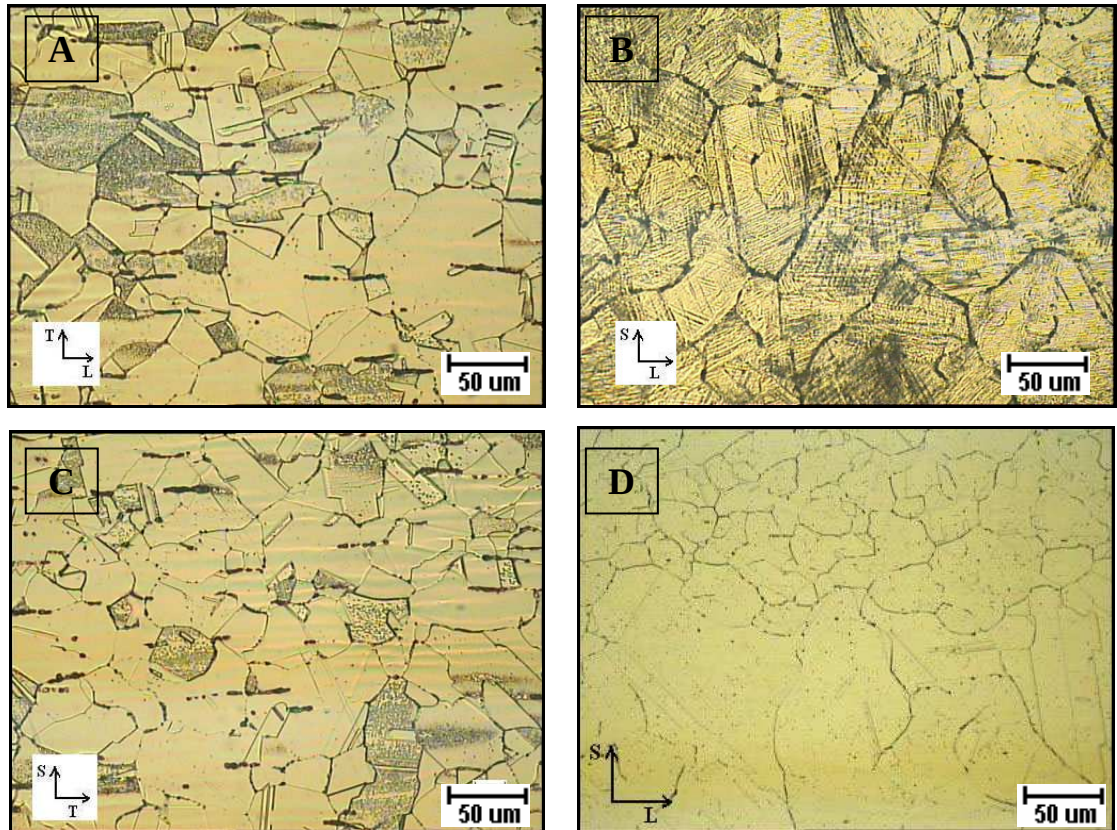


Figure 4: 3D Optical Micrographs of Specimen Q032 (0%CW) Etched with Oxalic Acid

Figure 4 shows the 3D optical micrographs of specimen Q032. Figure 4A, B and C were taken from T-L, S-L and S-T planes of the specimen respectively. The results of grain size and elongation ratio determined from the 3D micrographs are shown in Table 7. Figure 4A and C are fairly equiaxed with the grain size of $31\mu\text{m}$ but Figure 4B from S-L plane contained relatively large grains and has the grain size of about $34\mu\text{m}$. Figure 4D was taken closed to the edge on S-L plane and showed a sharp contrasting image of larger grain sizes at the extreme end and the adjacent smaller grain sizes. The average grain size across the 3 planes was $32\mu\text{m}$.

The elongation ratios on each of the 3 planes are also shown in the Table 7. The indicated that grains on the S-L plane appeared to be more elongated that the S-T and T-L planes.

Table 7: Specimen Q032 (0% CW) Grain Size and Elongation Ratio

specimen	Grain Size μm				Elongation Ratio		
	T-L plane	S-L plane	S-T plane	Average Grain Size	T-L plane	S-L plane	S-T plane
Q 032 (0% CW)	31	34	31	32	1.08	1.38	1.26

3.1.5 Optical Metallography (Delta Ferrite Etching)

3.1.5.1 Specimen Q032

The results of delta ferrite etching of specimen Q032 with NaOH is shown in Figure 5A-C. There was no any trace of ferrite seen on the T-L plane (Figure 5A) but limited amount of elongated ferrite streaks were found in the S-L and S-T planes (Figure 5B and C respectively).

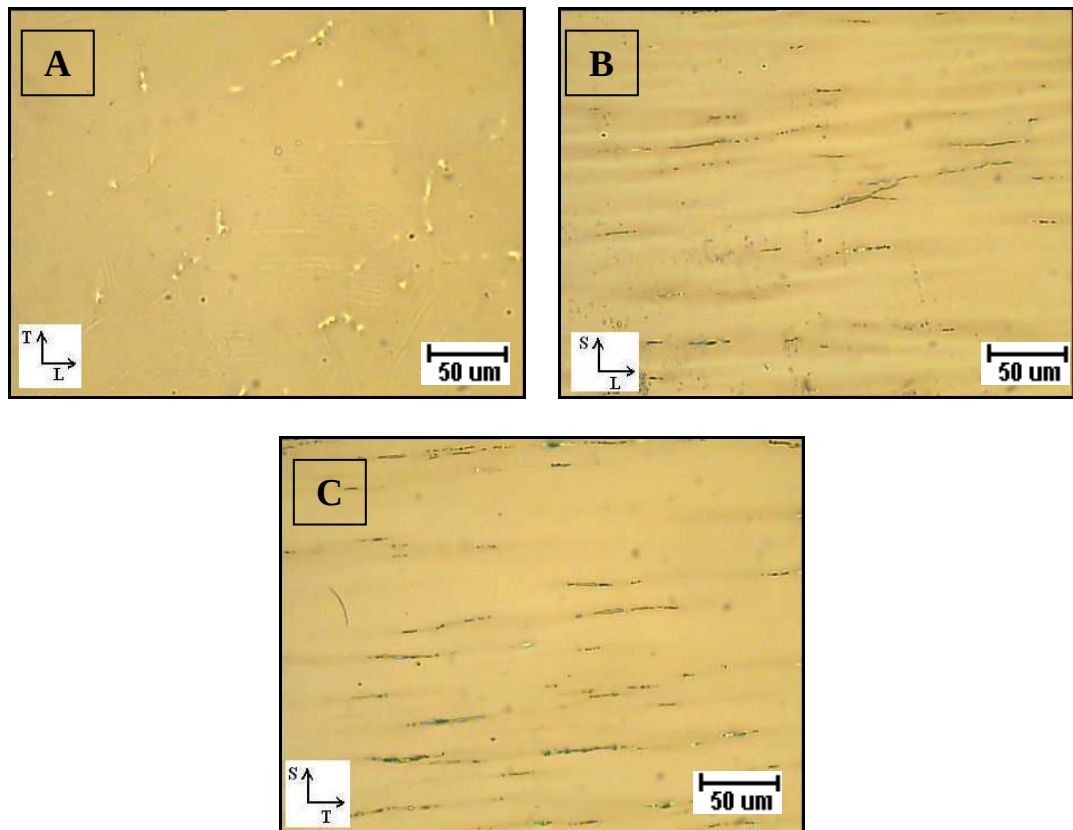


Figure 5: 3D Optical Micrographs of Specimen Q032 (0%CW) Etched with NaOH

3.1.6 Optical Metallography (Martensite Etching)

3.1.6.1 Specimen Q032

Etching of specimen Q032 with picral reveal traces of martensite on the T-L plane (Figure 6A) but no any trace of martensite was found in S-L (Figure 6B) and S-T plane (Figure 6C).

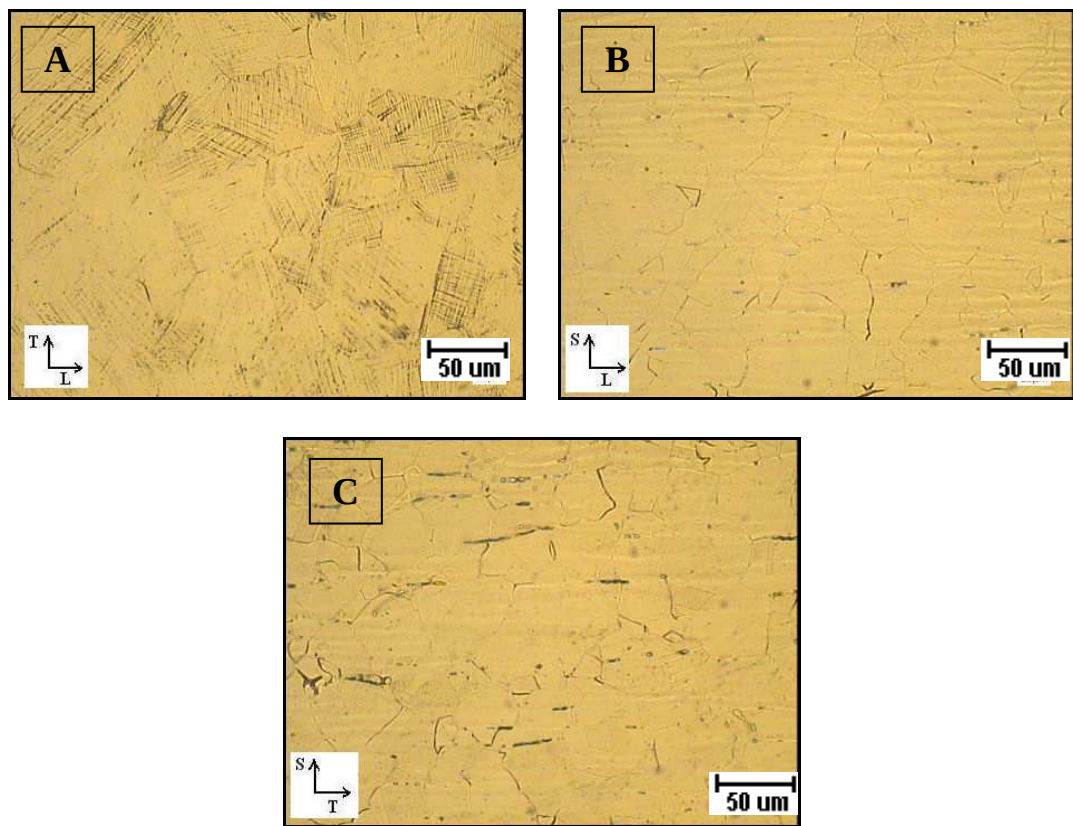


Figure 6: 3D Optical Micrographs of Specimen Q032 (0%CW) Etched with Picral

3.1.7 Electron Back Scatter Diffraction

3.1.7.1 Specimen Q032

The results of texture obtained from EBSD on planes S-L and S-T of specimen Q032 are shown as pole figures in Figure 7A and B.

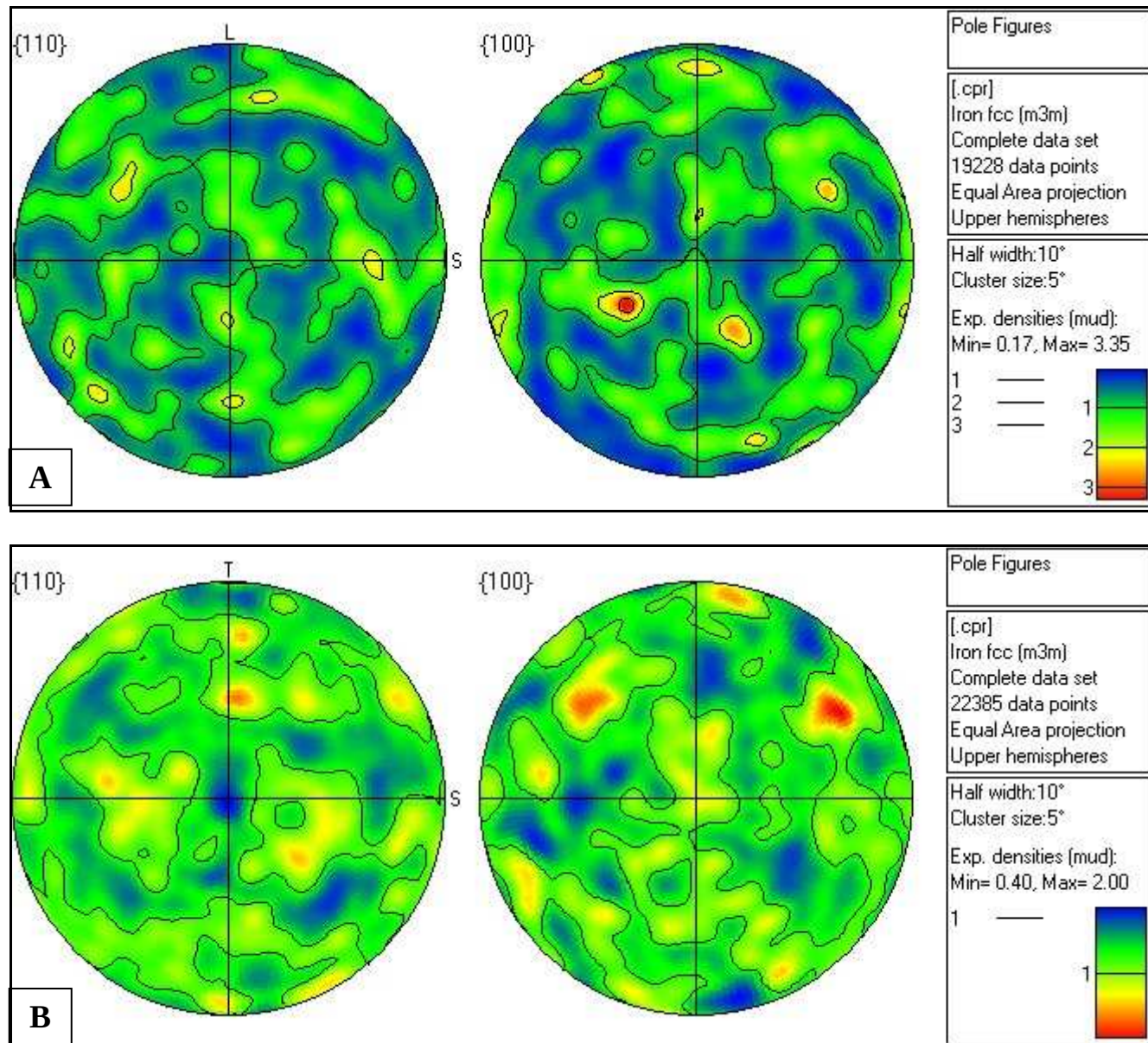


Figure 7: Pole Figures of Specimen Q032 (0% CW) on S-L Plane (A) and S-T Plane (B)

The texture in specimen Q032 was fairly random as shown in Figure 7A and B, however, in both S-L and S-T planes, crystallographic planes {100} appeared to be slightly textured than {110}. In contrast, the texture in S-L plane is relatively stronger than that of S-T plane as indicated by the Multiples of Uniform Density (MUD).

3.2 Characterisation of Heat D

The carbon content in Heat D material was 0.045wt% slightly lower than Heat G. It has the lowest Si content of 0.32wt% and the highest amount of Mn amongst other materials in Table 1. The specimens made from Heat D have been subjected to various degree of cold work. Specimens R592, R585 and R591 have been cold worked by pulling to 5%, 10% and 30% reduction respectively. The 3 specimens were obtained from CT specimen with along orientation ((S-L orientation in Figure 1). The results of their characterisation are as follow.

3.2.1 Vickers Hardness

3.2.1.1 Specimen R592 (5% Pulled)

The mean Vickers hardness of specimen R592 with 5% cold reduction fluctuated between 183 and 189 HV30 (Table 8 and Figure 8). The mean hardness value across the through thickness was 185 HV30 (Table 28). The hardness values across the through thickness were fairly uniform with little deviation from the mean value.

Table 8: Vickers Hardness of Specimen R 592, 5% Pulled (Along Orientation)

Through Thickness Distance (mm)	HV30 @ 1	HV30 @ 2	HV30 @ 3	Mean HV30
1	190	189	188	189
2	189	187	184	187
3	188	187	186	187
4	188	185	184	186
5	185	184	181	183

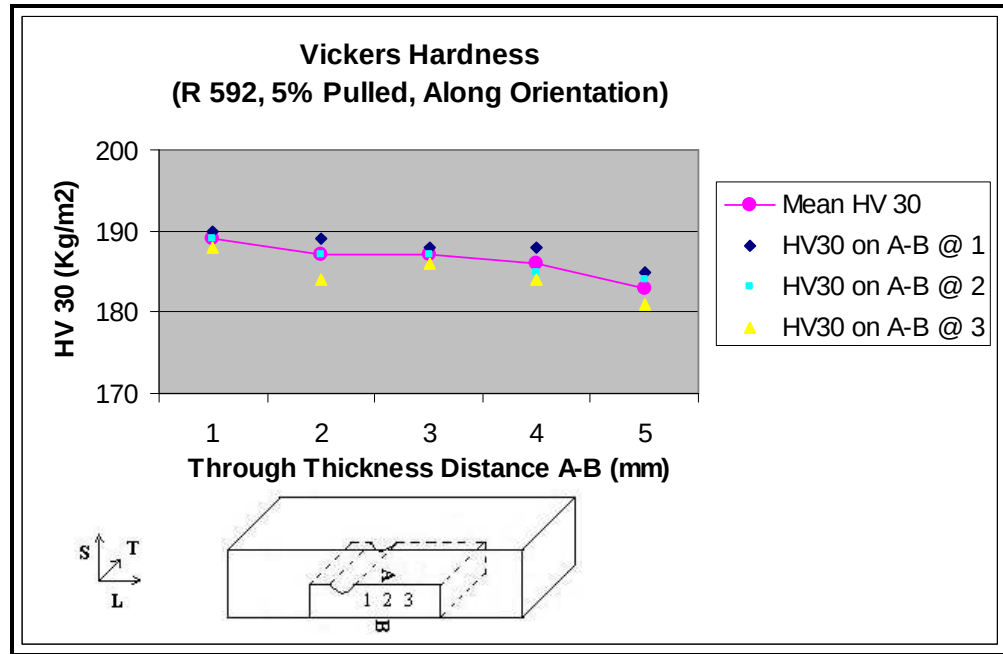


Figure 8: Vickers Hardness of Specimen R 592, 5% Pulled

3.2.1.2 Specimen R585 (10% Pulled)

The Vickers hardness values obtained from specimen R585, cold worked to 10% reduction was slightly higher and relatively more uniform than specimen R592. Table 9 and Figure 9 showed the results of Vickers hardness of Specimen R585. The average hardness value on specimen R585 across the through thickness was 223 HV30 (Table 28).

Table 9: Vickers Hardness of Specimen R 585 (10% Pulled)

Through Thickness Distance (mm)	HV30 @ 1	HV30 @ 2	HV30 @ 3	Mean HV30
1	224	223	225	224
2	229	225	222	225
3	227	223	222	224
4	228	219	220	222
5	224	216	223	221

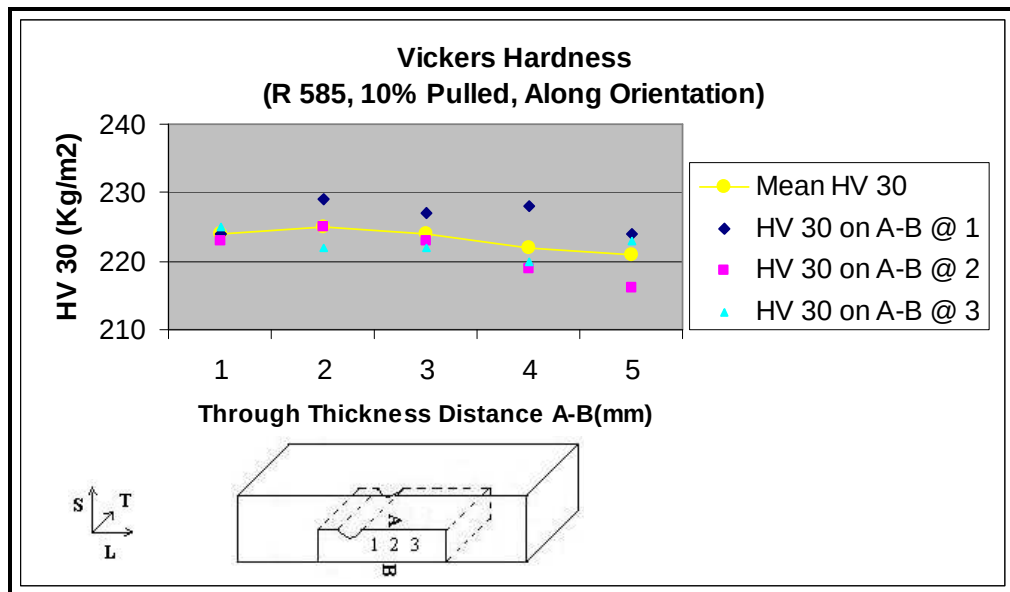


Figure 9: Vickers Hardness of Specimen R 585 (10% Pulled)

3.2.1.3 Specimen R591 (30% Pulled)

Specimen R591 has the highest cold work of 30% reduction amongst Heat D specimens. The Vickers hardness results of specimen R591 (Table 10 and Figure 10) were significantly higher than specimens R592 and R585. The mean hardness value across the through thickness was 344HV30 (Table 28).

Table 10: Vickers Hardness of Specimen R 591, 30% Pulled

Through Thickness Distance (mm)	HV30 @ 1	HV30 @ 2	HV30 @ 3	Mean HV30
1	337	344	341	341
2	339	341	331	337
3	336	342	339	339
4	352	346	343	347
5	338	347	333	339
6	339	341	336	339
7	349	343	346	346
8	346	350	357	351
9	343	353	357	351
10	350	346	350	349

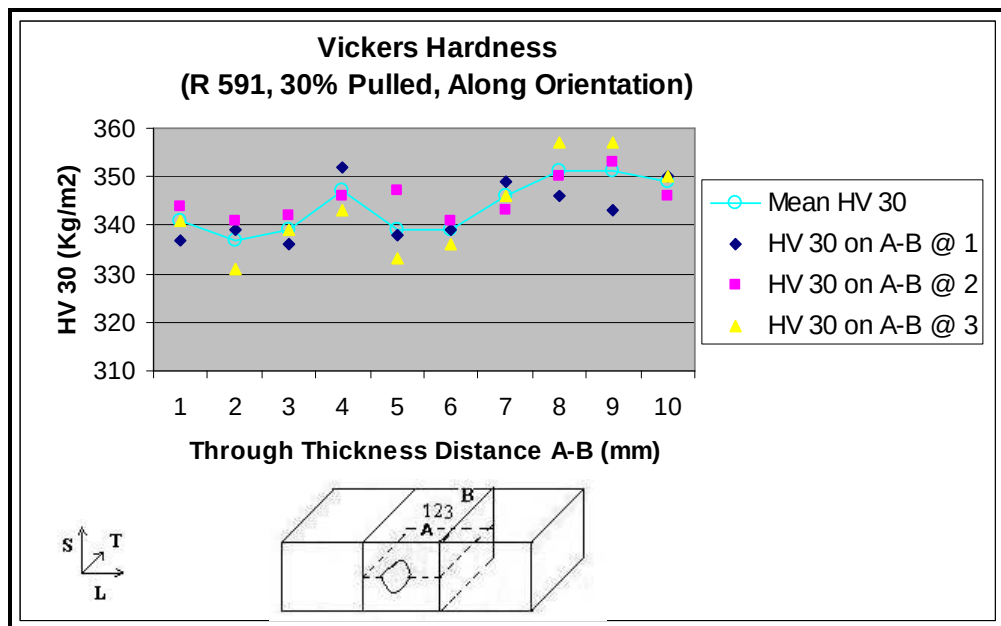


Figure 10: Vickers Hardness of Specimen R 591, 30% Pulled

Figure 11 showed the summary of the mean hardness profiles of the three specimens made from Heat D materials. The hardness value appeared to be sensitive to the amount of cold work. Specimen R591 with 30% reduction has the highest hardness and specimen R592 cold worked to 5% reduction was the lowest slightly below R585 with 10% reduction.

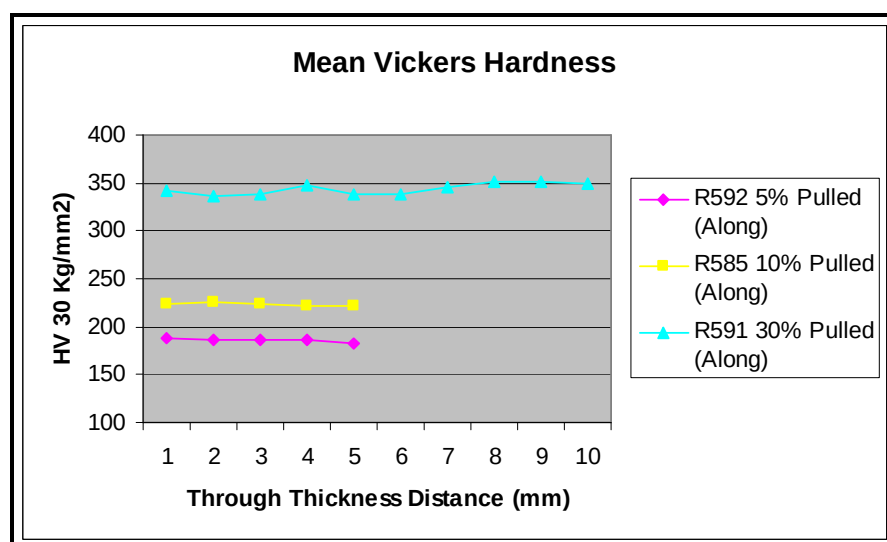


Figure 11: Mean Vickers Hardness of Heat D Specimens

3.2.2 X-Ray Diffraction (Strain Induced Martensite)

3.2.2.1 Specimen R592 (5% Pulled)

The XRD undertaken with respect to specimen R592 (5% CW) showed a complete absence of martensite (Table 11). Specimen R592 contained predominantly austenite phase after 5% cold reduction.

3.2.2.2 Specimen R585 (10% Pulled)

The results of SIM measurement on specimen R585 cold worked to 10% reduction was not different from that of R592 (Table 11). This indicated that the level of cold work in specimen R585 was still not sufficient to produce detectable amount of SIM. The amount of austenite phase present after 10% cold work was found to be 100%.

3.2.2.3 Specimen R591 (30% Pulled)

The high cold reduction in specimen R591 (30%) generated significant amount of SIM (Table 11). The amount of SIM in specimen R591 was about 4%, quite significant amounts compare to complete absence in specimens R592 and R585. Figure 12 shows the graphical comparison of SIM in Heat D materials with their different cold reductions.

Table 11: Strain Induced Martensite of Heat D Specimens

Samples	C W (%)	Integrated Intensity, <i>I</i>							V _γ	Martensite (%)
		γ-200	α-200	γ-220	α-211	γ-311	γ-222	α-220		
R592	5	11165	0	15816	0	8190	2021	0	1	0
R585	10	23396	0	3714	0	9100	4157	0	1	0
R591	30	24160	300	795	509	4143	7978	258	0.9594	4

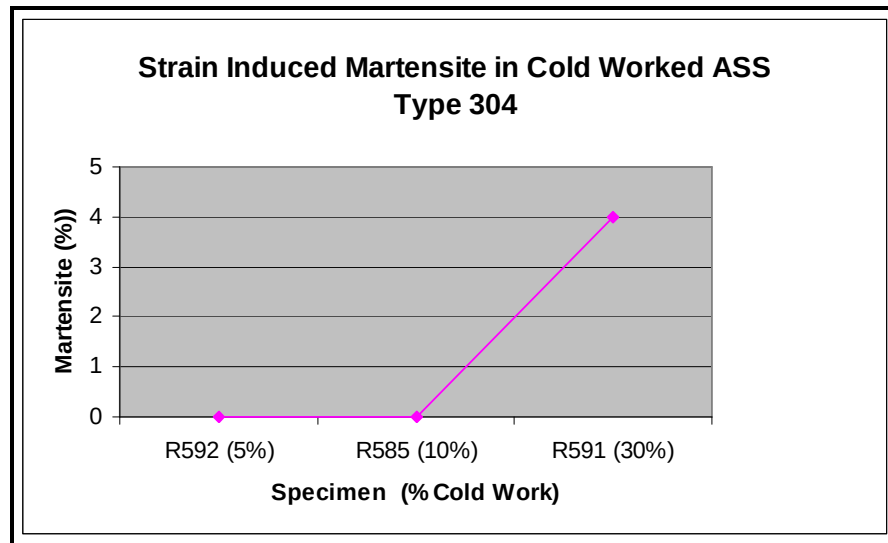


Figure 12: Strain Induced Martensite of Heat G Specimens

3.2.3 X-Ray Diffraction (Residual Stress)

3.2.3.1 Specimen R592 (5% Pulled)

The longitudinal residual stress measurement in specimen R592 cold worked to 5% reduction was completely compressive across the through thickness (Table 12 and Figure 13). The compressive residual stress in specimen R592 was quite large and its magnitude varied between -111.8 and -118.5 MPa.

Table 12: Residual Stress in Specimen R592 (5% Pulled)

No	X-Position (mm)	Y-Position (mm)	Phi-Position (deg.)	Stress (MPa)	Error
1	0	0	0	-111.811	22.736
2	0	1.5	0	-126.111	20.24
3	0	3	0	-118.514	25.235

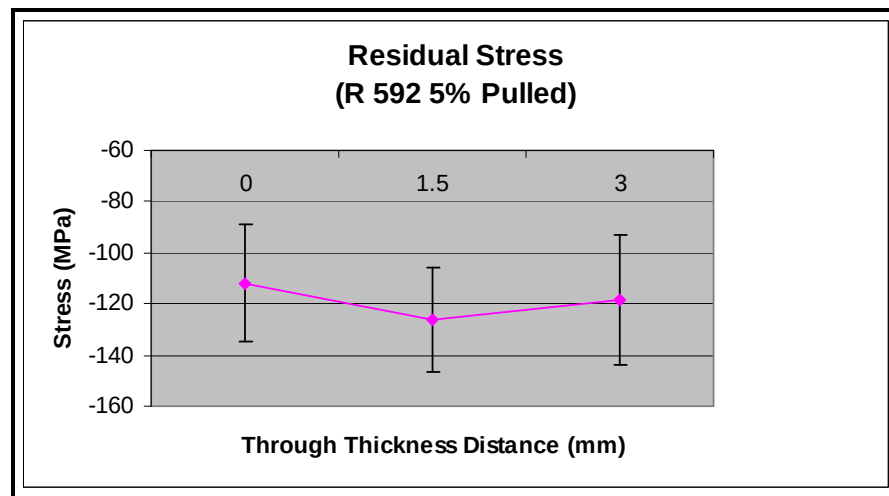


Figure 13: Residual Stress in Specimen R592 (5% Pulled)

3.2.3.2 Specimen R585 (10% Pulled)

There was significant reduction in compressive residual stress found in specimen R585 cold worked to 10% reduction when compared with specimen R592. Table 13 and Figure 14 show the results of residual stress measurement on specimen R585. The extreme points 1 and 3 have stress values of about -18 and -28 MPa with an acceptable error margin < 50 MPa. The stress value at point 2 was relatively low (-2 MPa) with error value greater than acceptable margin.

Table 13: Residual Stress in Specimen R585 (10% Pulled)

No	X-Position (mm)	Y-Position (mm)	Phi-Position (deg.)	Stress (MPa)	Error
1	0	0	0	-17.993	44.567
2	0	1.5	0	-2.022	72
3	0	3	0	-28.286	46.816

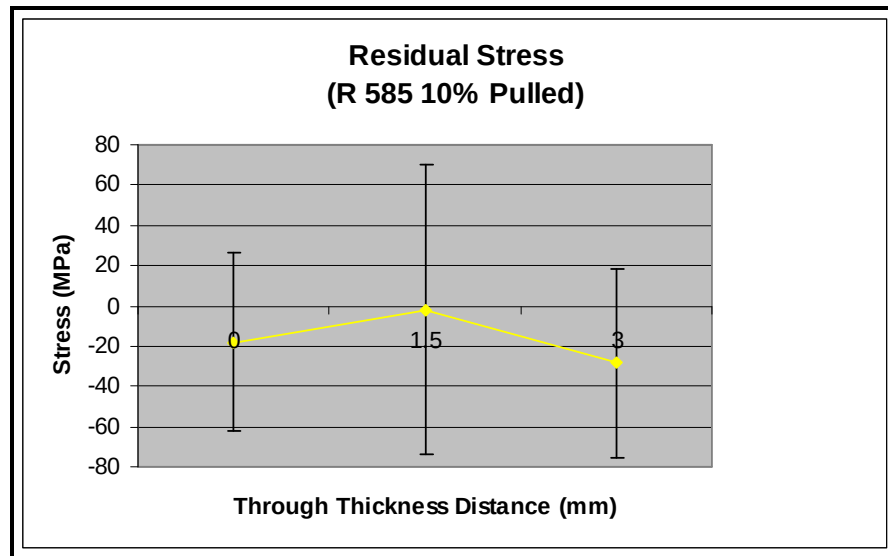


Figure 14: Residual Stress in Specimen R585 (10% Pulled)

3.2.3.3 Specimen R591 (30% Pulled)

The residual stress in specimen R591 cold worked to 30% reduction was mainly compressive with some tensile stresses at point number 2 and 4 (Table 14). All the error values were below the acceptable limit of 50MPa. Figure 15 showed the plot of residual stress against the Y-positions. The two tensile components of the residual stresses correspond to Y-positions 2 and 6.

Table 14: Residual Stress in Specimen R591 (30% Pulled)

No	X-Position (mm)	Y-Position (mm)	Phi-Position (deg.)	Stress (MPa)	Error
1	0	0	0	-73.887	30.15
2	0	2	0	27.051	30.61
3	0	4	0	-36.29	22.159
4	0	6	0	4.307	27.305
5	0	8	0	-33.597	26.871

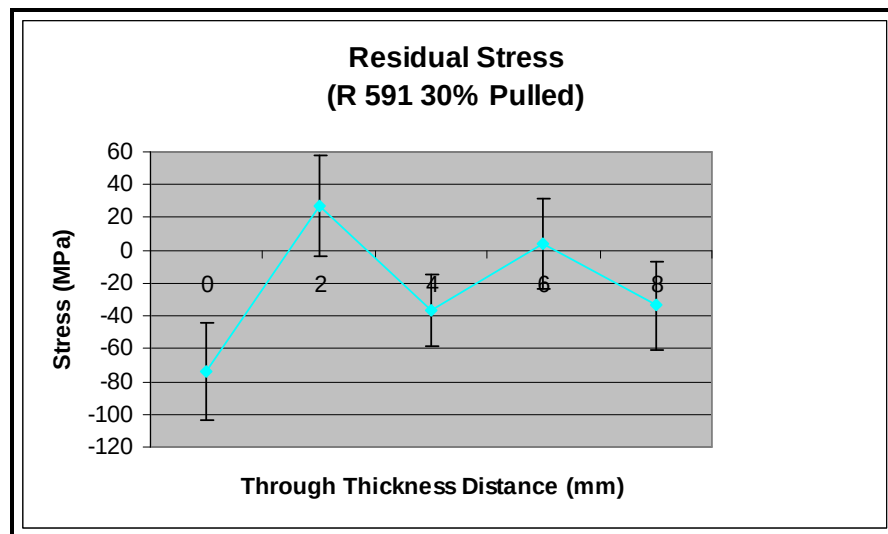


Figure 15: Residual Stress in Specimen R591 (30% Pulled)

Figure 16 compares and contrasts the longitudinal residual stress measured in Heat D specimens R592, R585 and R591 cold worked to 5%, 10% and 30% respectively. R592 was compressive. The stress pattern in R585 was quite similar to R591, except that there were relatively more tensile stress components in R591 whilst the residual stress in R585 was completely compressive.

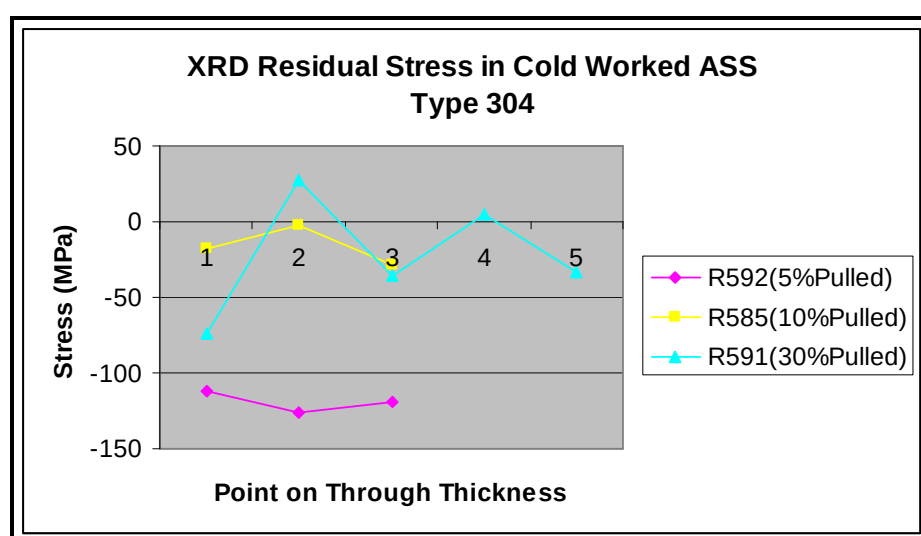


Figure 16: Residual Stress in Heat D Specimens

3.2.4 Optical Metallography (Grain Boundary Etching)

3.2.4.1 Specimen R592 (5% Pulled)

The results of oxalic acid etching of specimen R592 is shown in Figure 17. The micrographs revealed the grain boundaries and twinning in all the 3 planes namely: T-L, S-L and S-T planes. There were more of abnormal large grain sizes in both S-L and S-T plane than T-L plane.

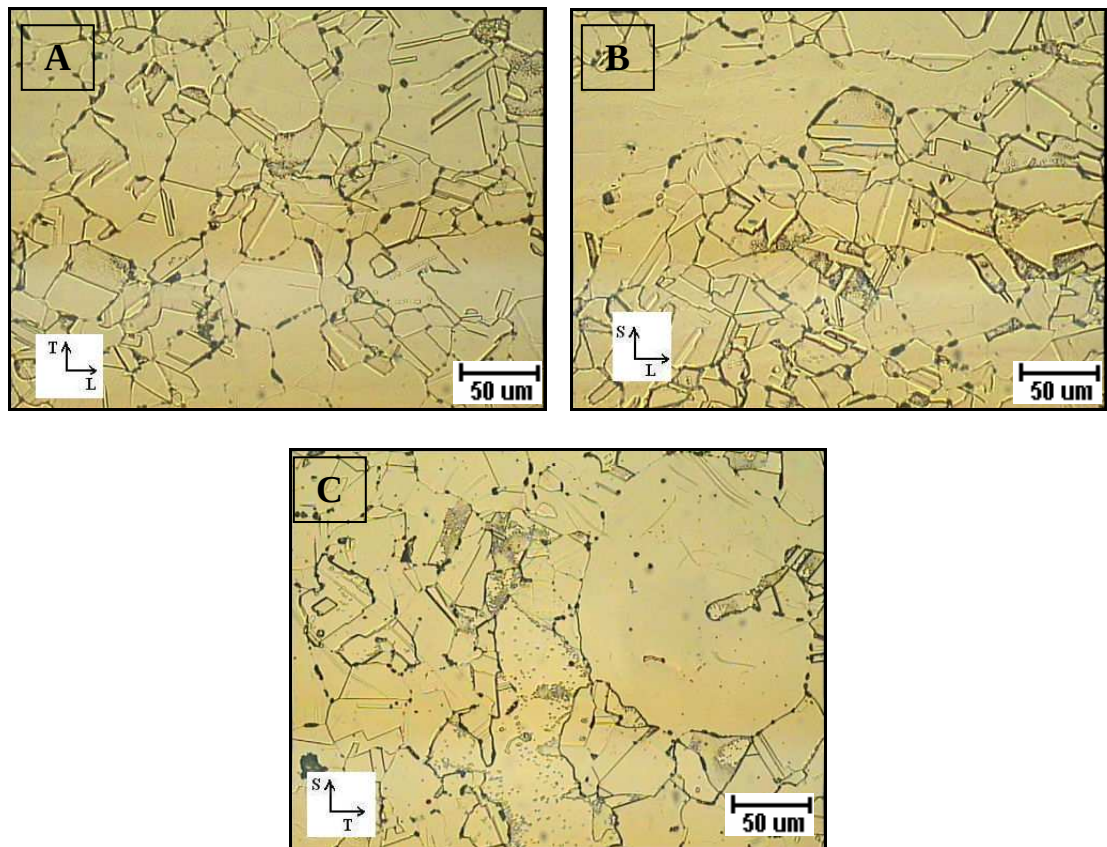


Figure 17: 3D Optical Micrographs of Specimen R592 (5% Pulled) Etched Oxalic Acid

Results of grain size and elongation ratio determined from Figure 17 showed slight variation in grain size and elongation ratio across the 3 planes (Table 15). S-L plane has the largest grain size of 41μm and the lowest grain size occurred in the T-L plane. The average grain size across

the 3 planes measured at $36\mu\text{m}$. The grains in the T-L plane were more elongated with ratio of about 1.6 than S-L and S-T planes which have elongation ratios of 1.4 and 1.2 respectively.

3.2.4.2 Specimen R585 (10% Pulled)

There were no abnormal large grain sizes found in specimen R585 micrographs (Figure 18). The grain size in T-L plane was $27\mu\text{m}$. It is relatively smaller than S-L and S-T plane which have almost the same grain size of 30 and $29\mu\text{m}$ respectively. The average grain size across the 3 planes was $29\mu\text{m}$. Results show that grains were more elongated in S-L plane than other planes. The elongation ratio on S-L plane was about 0.3 and 0.4 more than those of T-L and S-T respectively (Table 15).

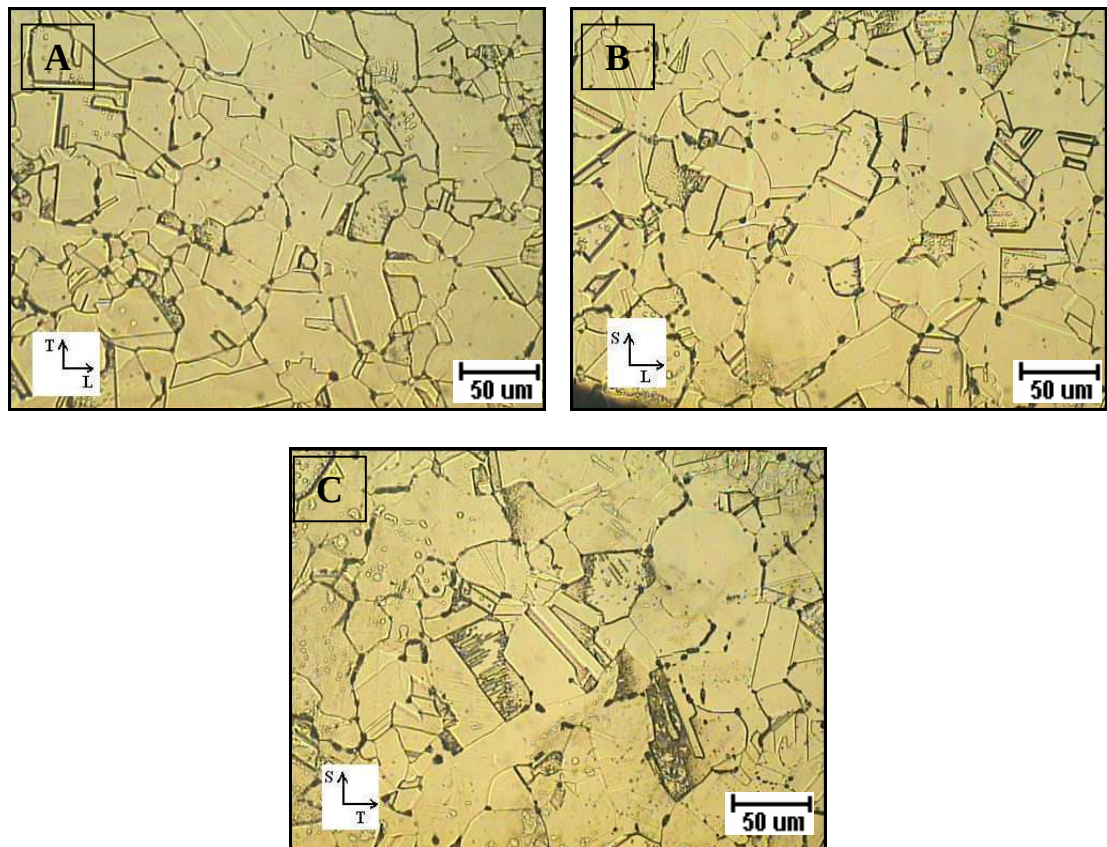


Figure 18: 3D Optical Micrographs of Specimen R585 (10% Pulled) Etched Oxalic Acid

3.2.4.3 Specimen R591 (30% Pulled)

The 3D optical micrographs of oxalic acid etching of specimen R591 is shown in Figure 19. The grain sizes in S-L and S-T planes were about 3 - 4 μm larger than T-L plane (Table15). Average grain size across the 3 planes of specimen R591 was about 32 μm . The elongation ratio in T-L plane was almost twice, significantly higher than S-L and S-T planes which have almost equal ratio of about 1.6

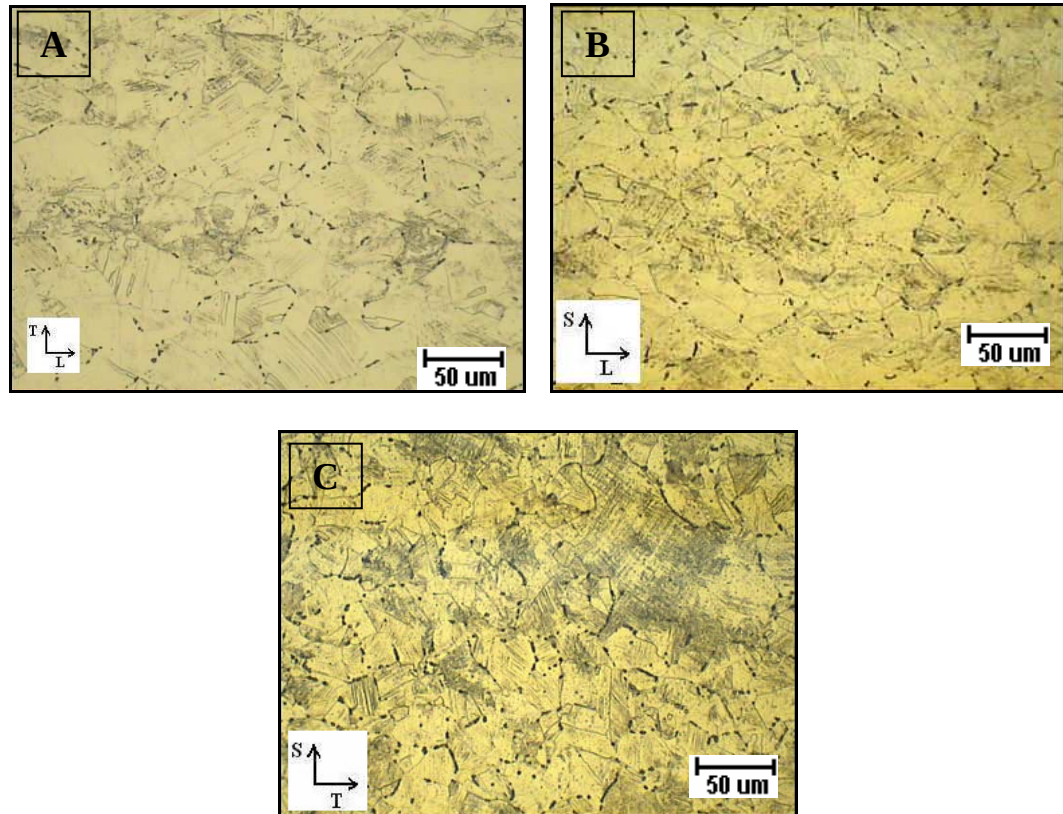


Figure 19: 3D Optical Micrographs of Specimen R591 (30% Pulled) Etched Oxalic Acid

Table 15: Grain Size and Elongation Ratio of Heat D Specimens

Specimen (%CW)	Grain Size μm				Elongation Ratio		
	T-L plane	S-L plane	S-T plane	Average Grain Size	T-L plane	S-L plane	S-T plane
R 592 (5%)	32	41	36	36	1.63	1.41	1.22
R 585 (10%)	27	30	29	29	1.14	1.44	1.04
R 591 (30%)	30	34	33	32	2.06	1.61	1.63

3.2.5 Optical Metallography (Delta Ferrite Etching)

3.2.5.1 Specimen R592 (5% Pulled)

The results of delta ferrite etching of specimen R592 with NaOH revealed no any trace of ferrite as evident in Figure 20. In none of the 3 planes were delta ferrite found.

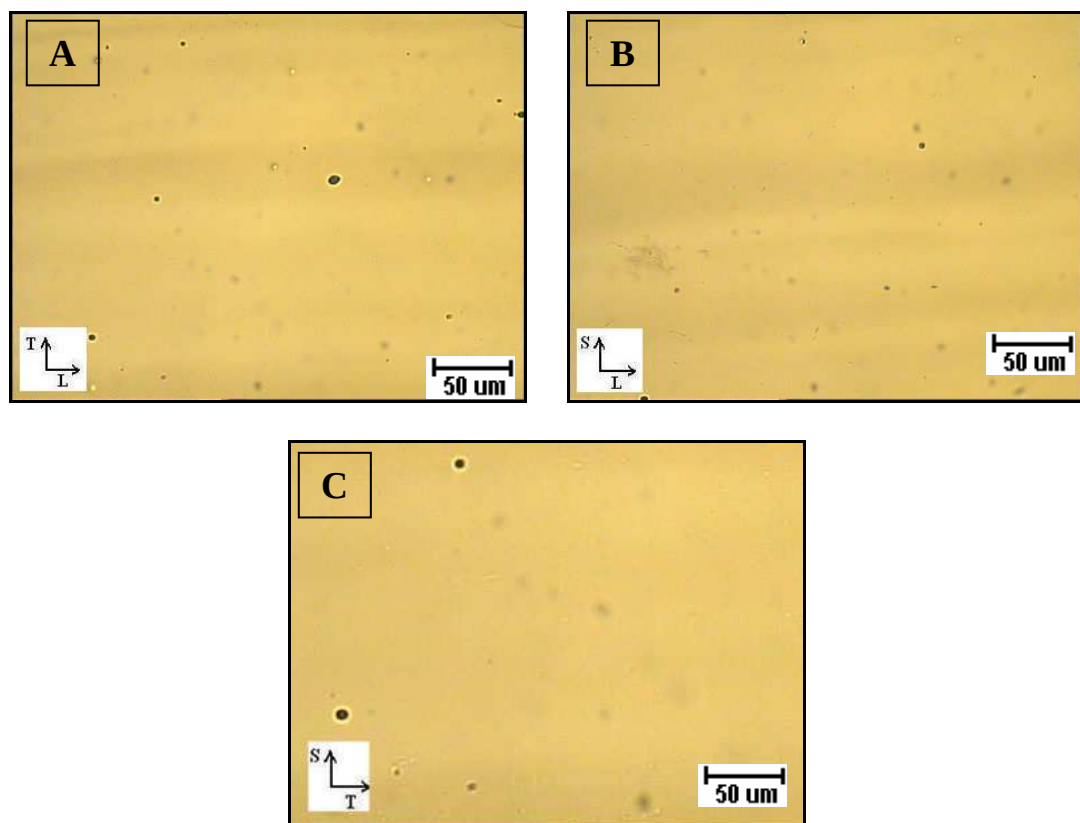


Figure 20: 3D Optical Micrographs of Specimen R592 (5% Pulled) Etched NaOH

3.2.5.2 Specimen R585 (10% Pulled)

The result of delta ferrite etching of specimen R585 (Figure 21) was no different from of specimen R592. No delta ferrite was found in all the 3 planes.

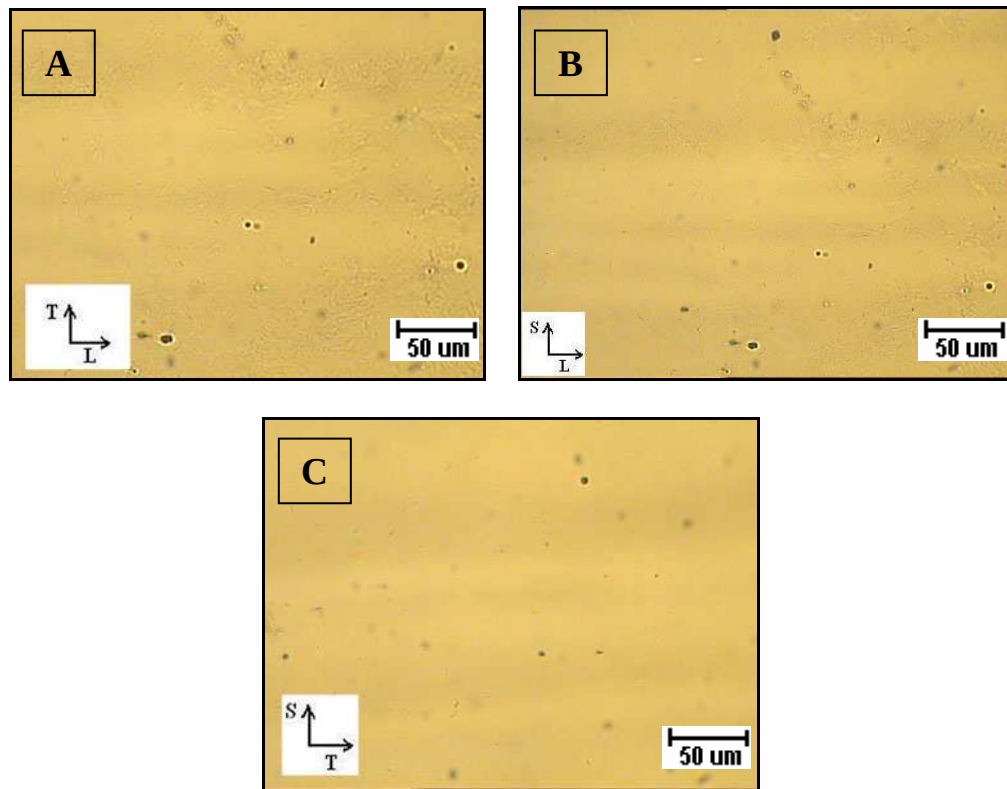


Figure 21: 3D Optical Micrographs of Specimen R585 (10% Pulled) Etched NaOH

3.2.5.3 Specimen R591 (30% Pulled)

Figure 22 shows the 3D micrographs of specimen R591 etched with NaOH to reveal delta ferrite. Just as the other two specimens from Heat D material, there was no any trace of ferrite found in all the 3 planes of the specimen R591.

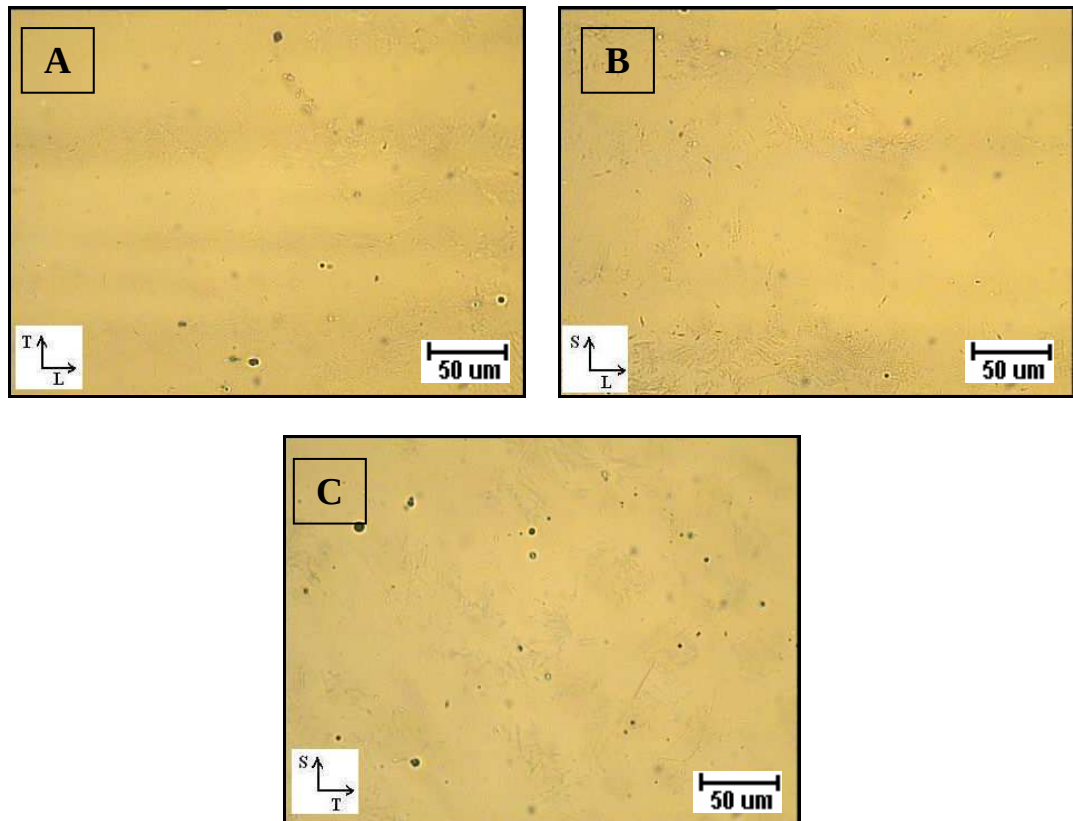
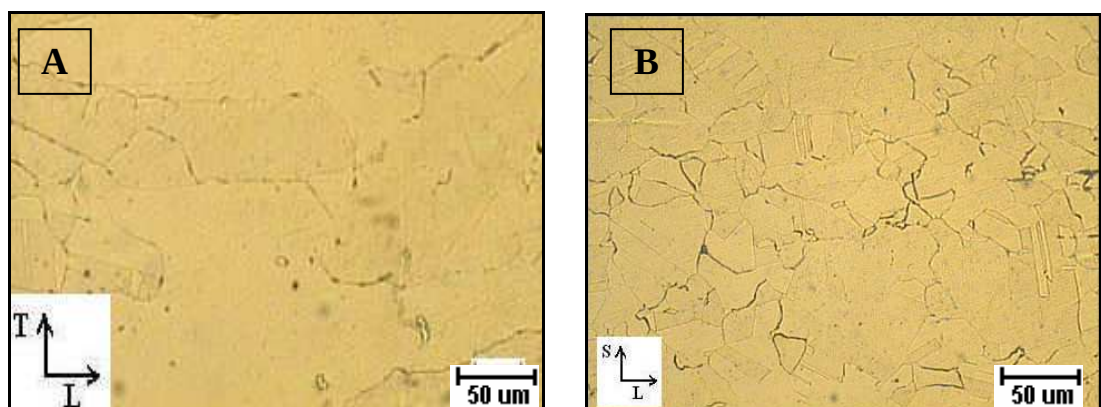


Figure 22: 3D Optical Micrographs of Specimen R591 (30% Pulled) Etched NaOH

3.2.6 Optical Metallography (Martensite Etching)

3.2.6.1 Specimen R592 (5% Pulled)

Figure 23 shows the 3D micrographs obtained from specimen R592 etched with picral to reveal the presence of SIM. No trace of SIM was found in all the 3 planes.



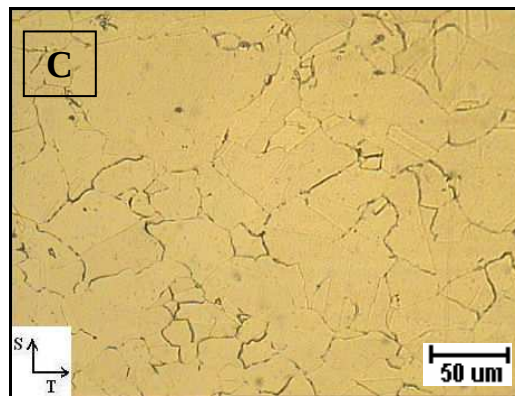


Figure 23: 3D Optical Micrographs of Specimen R592 (5% Pulled) Etched Picral

3.2.6.2 Specimen R585 (10% Pulled)

The result of SIM etching in specimen R585 shown in Figure 24 was not different from that of R592 result. No SIM was found in all the 3 planes of the specimen R585.

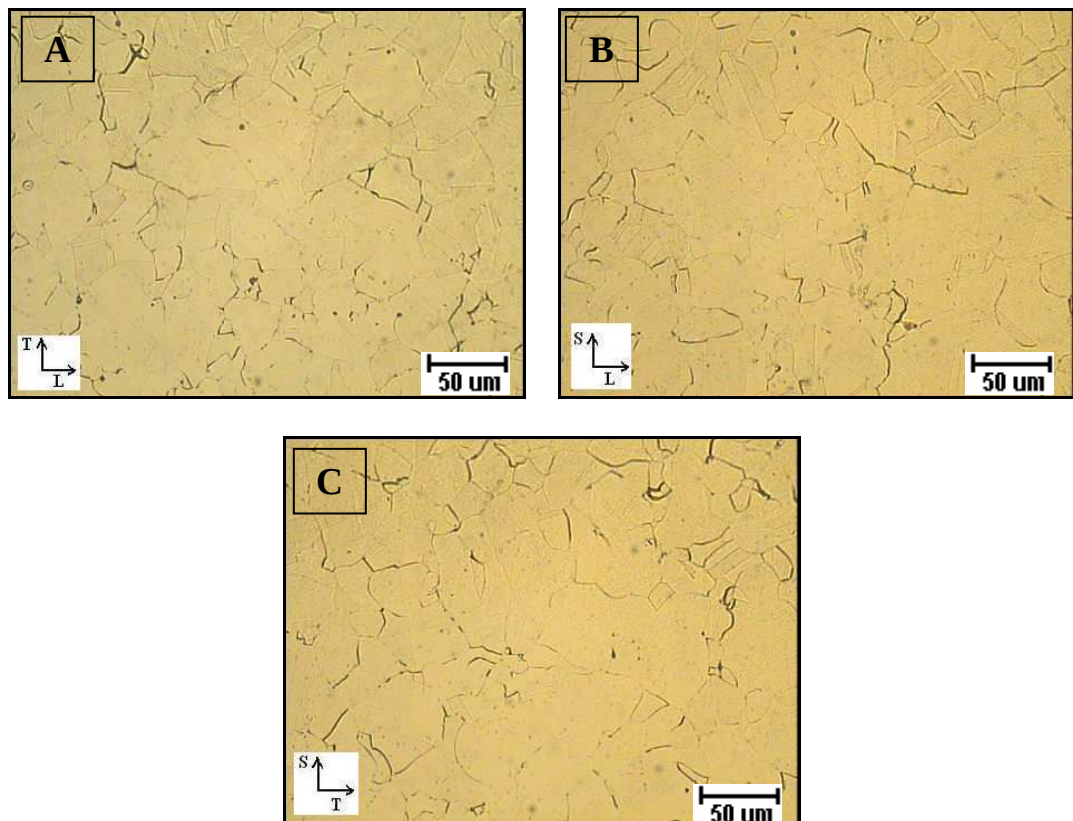


Figure 24: 3D Optical Micrographs of Specimen R585 (10% Pulled) Etched Picral

3.2.6.3 Specimen R591 (30% Pulled)

The T-L plane of Specimen R591 shows some traces of SIM (Figure 25A) and significant amount of SIM was found in S-T plane (Figure 25C). However, S-L plane did not reveal any visible presence of martensite.

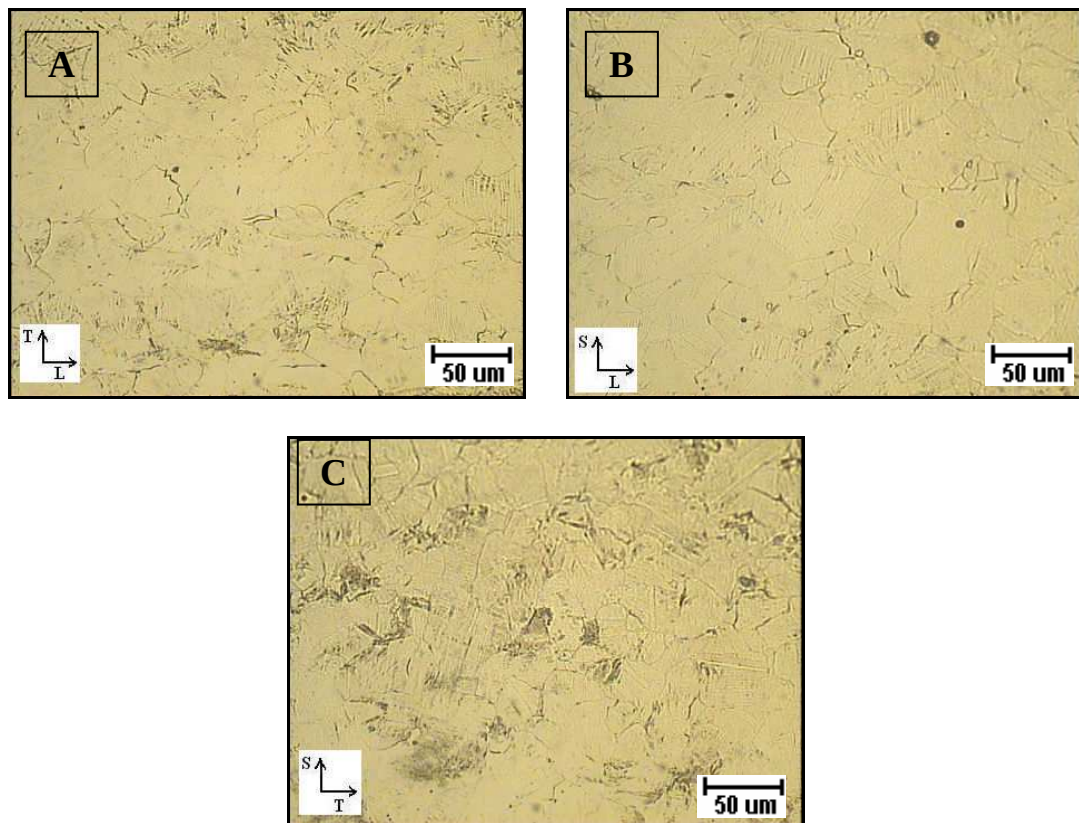


Figure 25: 3D Optical Micrographs of Specimen R591 (30% Pulled) Etched Picral

3.2.7 Electron Back Scatter Diffraction

3.2.7.1 Specimen R592 (5% Pulled)

The results of texture in pole figures obtained from S-L and S-T planes of specimen R592 is shown in Figure 26A and B respectively. S-L and S-T planes show random texture in both $\{110\}$ and $\{100\}$ crystallographic planes. Contrast between S-L and S-T planes shows that, S-L

plane is strongly textured relative to S-T plane as indicated by the Multiples of Uniform Density (MUD). The maximum MUD in S-L plane was about 3, and in S-T plane, the MUD was just about 2.

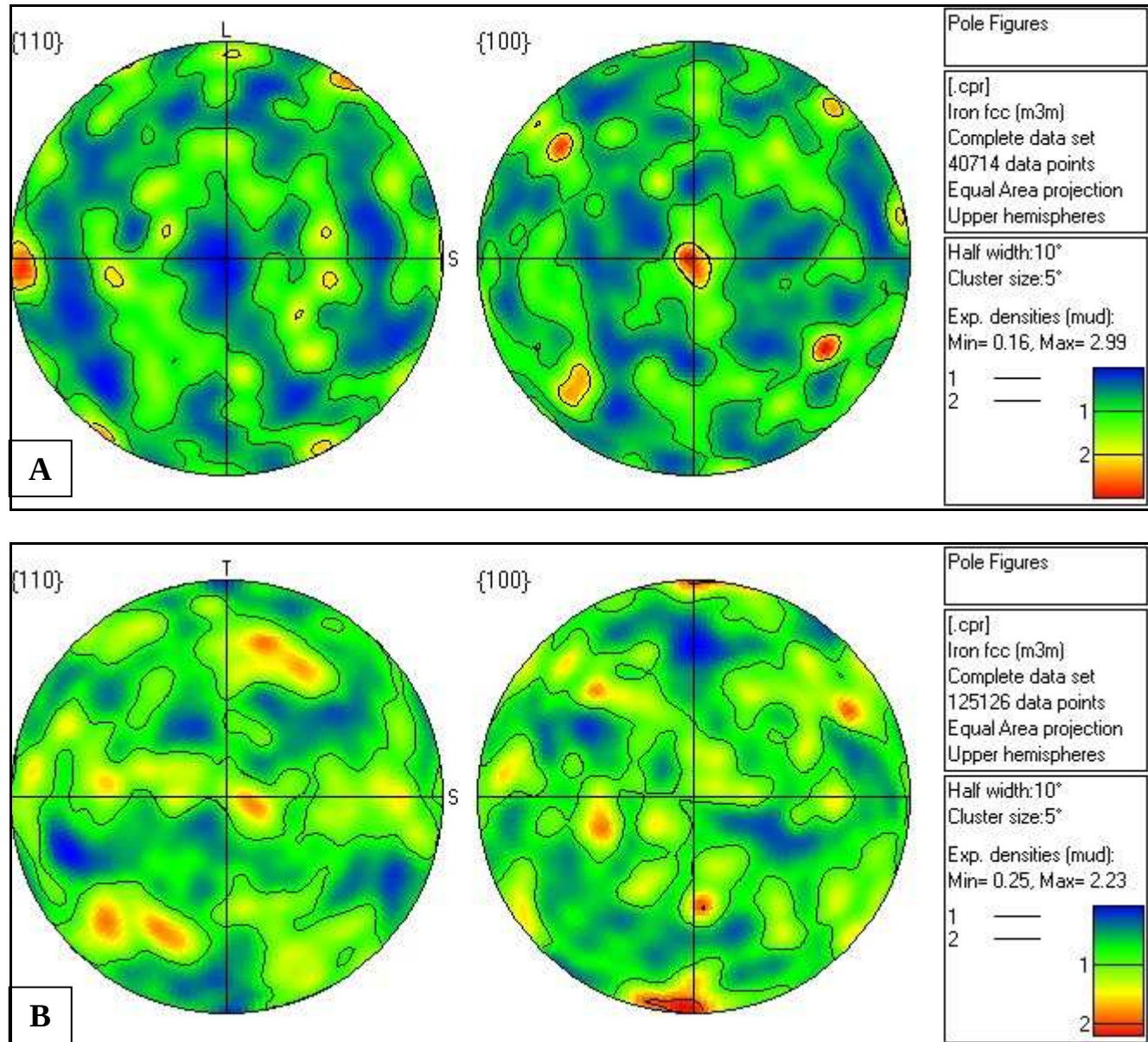


Figure 26: Pole Figures of Specimen R592 (5% Pulled) on S-L Plane (A) and S-T Plane (B)

3.2.7.2 Specimen R585 (10% Pulled)

The pole figures of specimen R585 also show random texture in both S-L and S-T planes (Figure 27) but with slightly stronger texture relative specimen R592. The maximum MUD in

S-L plane was about 3.6 while S-T plane has maximum MUD of about 3. In both cases, crystallographic planes $\{100\}$ were more textured than $\{110\}$ planes.

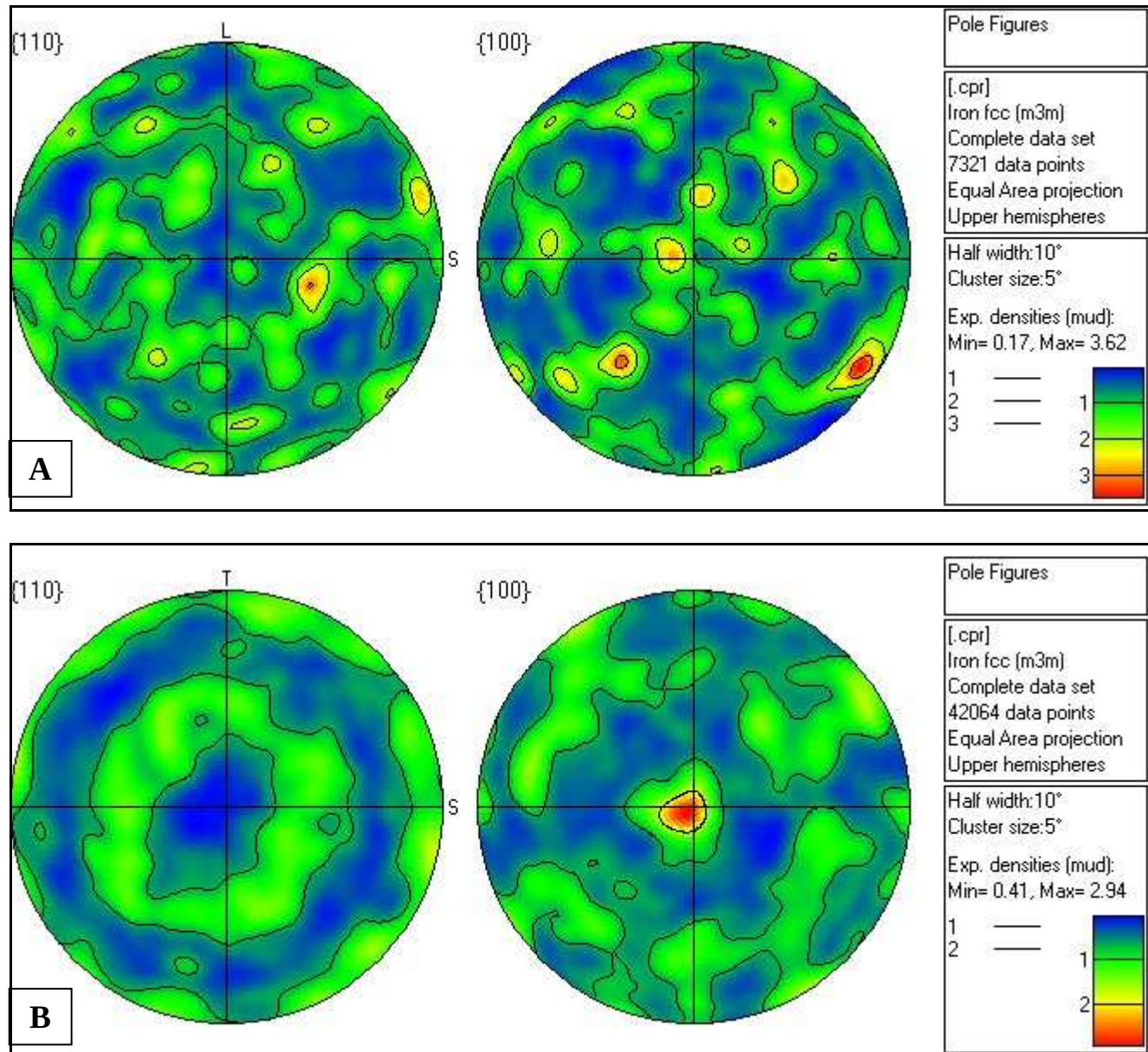


Figure 27: Pole Figures of Specimen R585 (10% Pulled) on S-L Plane (A) and S-T Plane (B)

3.2.7.3 Specimen R591 (30% Pulled)

The pole figures in Figures 28A and B show the results of texture on S-L and S-T planes of specimen R591. The textures in both planes were less random compare to specimen R592 and R585. However, S-L plane showed strong texture while the texture in S-T plane was relatively

weak. The MUD in S-L plane was about 3.5 while on S-T plane it was about 2.7. Crystallographic planes $\{100\}$ were strongly textured than planes $\{110\}$ in both cases.

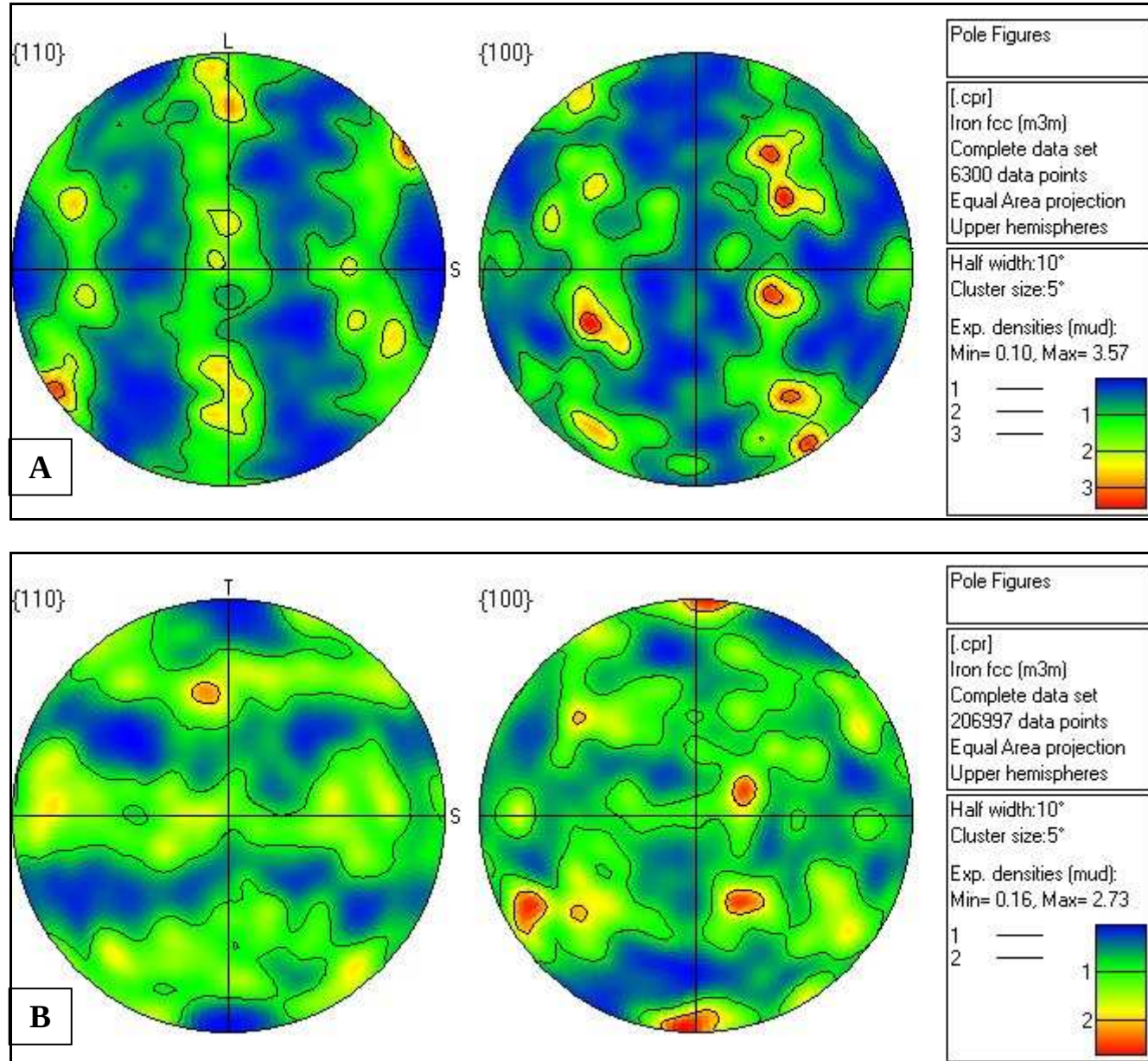


Figure 28: Pole Figures of Specimen R591 (30% Pulled) on S-L Plane (A) and S-T Plane (B)

3.3 Characterisation of Heat B

The carbon content in Heat B material was 0.043wt%, slightly lower than Heat D. Heat D contained highest amount of Si (0.43wt %) and significantly low Mn content (1.35 wt %)

(Table 1). Specimens R583 and Q114 cold worked by bi-directional rolling to 20% and 35% reduction respectively were made from Heat B material. Both were obtained from CT specimen but with different orientation. The former was obtained from CT specimen with across orientation (L-S orientation on ASTM notation (Figure 1)) and the latter was obtained from CT Specimen with semi orientation (L-T orientation in ASTM notation). The results of their characterisation are as follow.

3.3.1 Vickers Hardness

3.3.1.1 Specimen R583 (20% Bi-Rolled)

The results of Vickers hardness on specimen R583 cold worked to 20% reduction by rolling in two directions is shown in Table16 and Figure 29. The mean hardness varies between the highest value of 396HV30 closed to the surface and the lowest value of 307HV30 at the middle of the specimen. The average hardness across the specimen was 336HV30 (Table 28). The hardness profile in Figure 29 shows that the edges of the specimen adjacent to the rolls have higher hardness value than the central region of the specimen.

Table 16: Vickers Hardness of Specimen R 583 (20% Bi-Rolled)

Through Thickness Distance (mm)	HV30 @ 1	HV30 @ 2	HV30 @ 3	Mean HV30
1	385	397	405	396
2	370	382	368	373
3	349	351	349	350
4	317	322	315	318
5	312	307	312	310
6	317	328	323	323
7	307	342	309	319
8	307	310	303	307
9	320	321	318	320
10	327	336	348	337
11	343	350	342	345

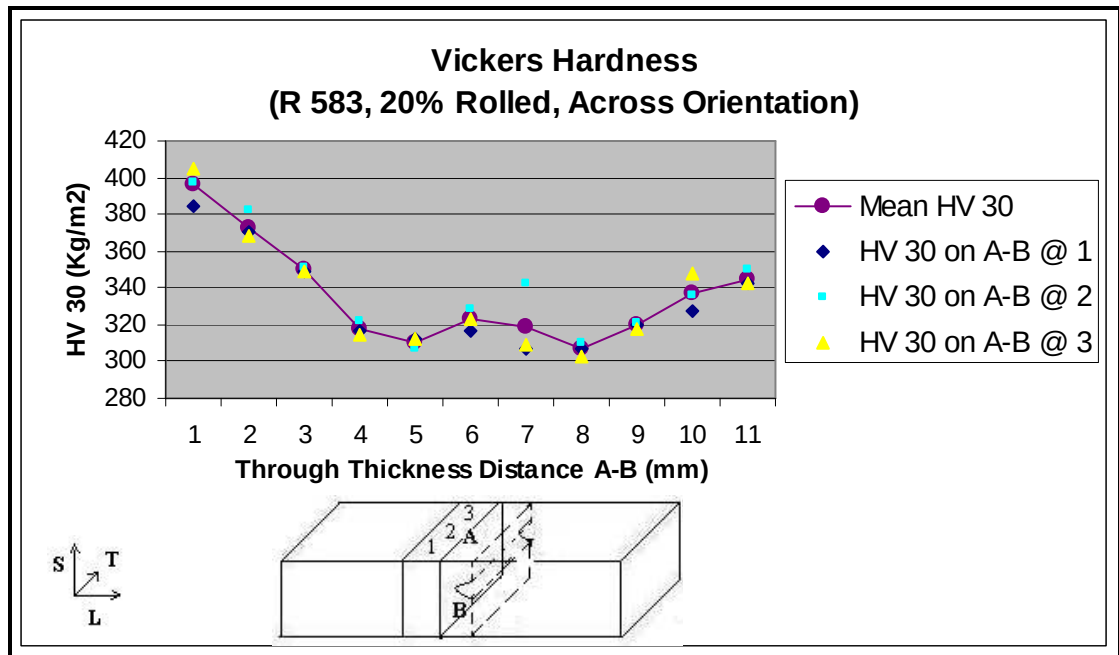


Figure 29: Vickers Hardness of Specimen R583 (20% Bi-Rolled)

3.3.1.2 Specimen Q114 (35% Bi-Rolled)

The mean Vickers hardness in specimen Q114 cold worked to 35% reduction (Table 17 and Figure 30) increased steadily from 377 to 438HV30 across the through thickness of the specimen. The average hardness across the through thickness measured at 409HV30 (Table 28) was comparatively higher than specimen R583.

Table 17: Vickers Hardness of Specimen Q114 (35% Bi-Rolled)

Through Thickness Distance (mm))	HV30 @ 1	HV30 @ 2	HV30 @ 3	Mean HV30
1	379	362	389	377
2	381	388	376	382
3	381	405	384	390
4	389	416	396	400
5	389	411	399	400
6	393	411	411	405
7	416	412	418	415
8	425	429	433	429
9	427	422	434	428
10	428	429	438	432
11	441	436	438	438

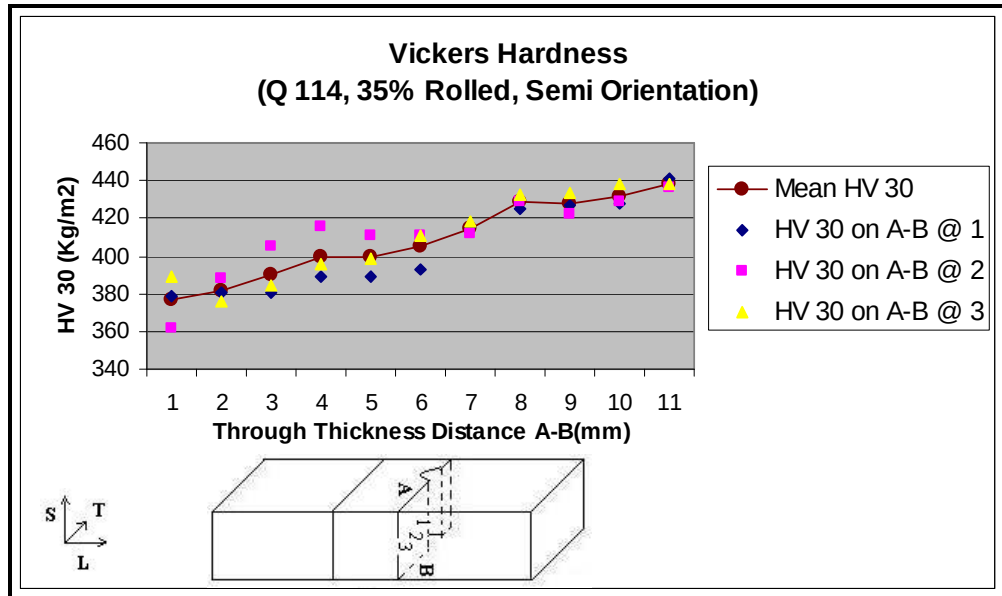


Figure 30: Vickers Hardness of Specimen Q114 (35% Bi-Rolled)

Figure 31 compares and contrasts the mean hardness profiles of specimen R583 and Q114 both of which were made from Heat B material. Specimen Q114 showed steady increase in hardness across the through thickness. While in the case of specimen R583, the hardness values close to the surface of the specimen adjacent to the rolls were significantly higher than the central portion of the specimen. The mean hardness in Q114 with higher cold reduction of 35% was significantly higher than R583 with 20% cold reduction with a margin of about 70HV30. Both of the specimens were rolled in two directions.

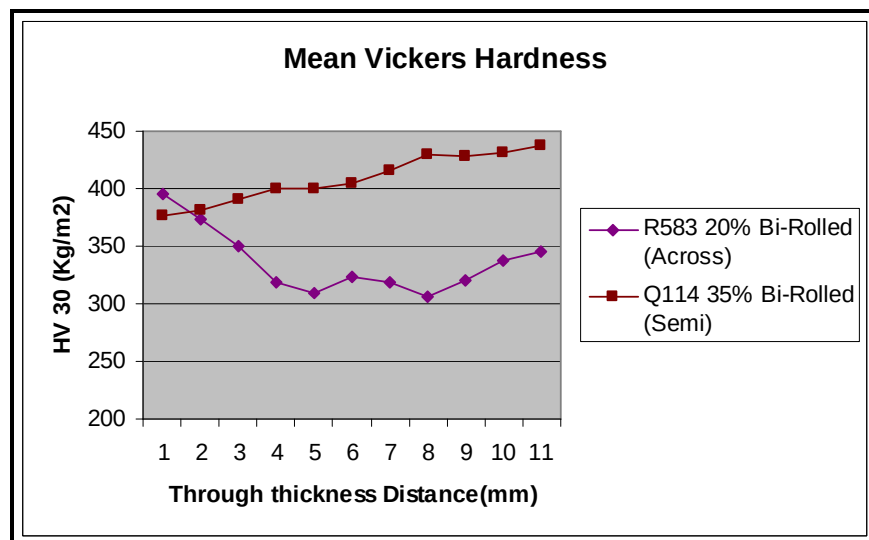


Figure 31: Mean Vickers Hardness of Heat B Specimens

3.3.2 X-Ray Diffraction (Strain Induced Martensite)

3.3.2.1 Specimen R583 (20% Bi-Rolled)

The results of quantification of SIM in specimen R583 by XRD is shown in Table 18 and Figure 32. 17% of SIM was found in specimen R583 cold worked to 20% reduction. The amount of SIM in R583 was relatively low compared to specimen Q114.

3.3.2.2 Specimen Q114 (35% Bi-Rolled)

The amount of SIM found in specimen Q114 measured up to 31% (Table 18), comparatively higher than the amount found in specimen R583 and indeed the highest amongst the rest of specimen tested. The increased amount of SIM in specimen Q114 is consistent with the degree and the method of cold reduction.

Table 18: Strain Induced Martensite in Heat B Specimens

Samples	C W (%)	Integrated Intensity, <i>I</i>							<i>V_γ</i>	Martensite (%)
		γ -200	α -200	γ -220	α -211	γ -311	γ -222	α -220		
R583	20	3139	601	2830	441	3286	907	165	0.8304	17
Q114	35	1985	918	2514	1204	2515	1186	399	0.6938	31

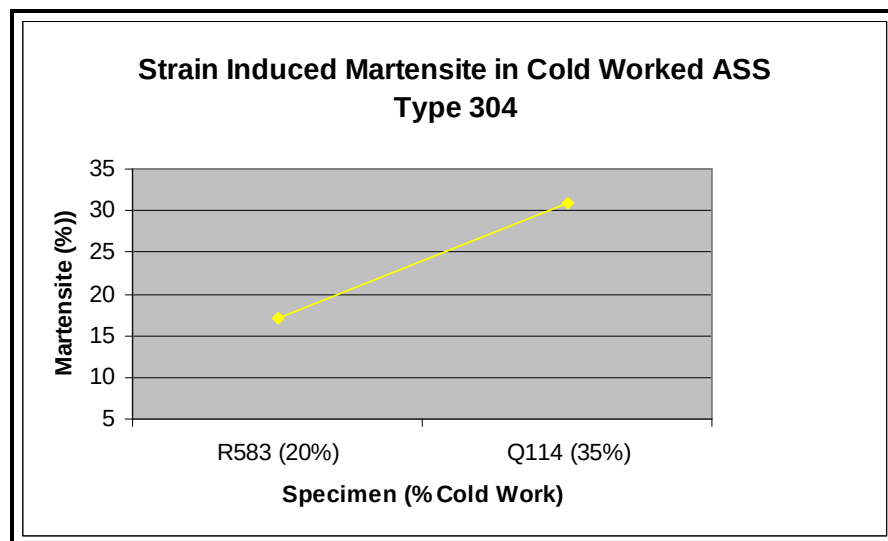


Figure 32: Strain Induced Martensite in Heat B Specimens

3.3.3 X-Ray Diffraction (Residual Stress)

3.3.3.1 Specimen R583 (20% Bi-Rolled)

The longitudinal residual stress measured in specimen R583 was largely compressive close to the surface (about -76MPa) and increased steadily across the through thickness to stress value of -24MPa (Table 19 and Figure 33). All the error values were within the acceptable limit of <50MPa.

Table 19: Residual Stress in Specimen R583 (20% Bi-Rolled)

No	X-Position (mm)	Y-Position (mm)	Phi-Position (deg.)	Stress (MPa)	Error
1	0	0	0	-76.402	24.267
2	0	3	0	-54.294	24.081
3	0	6	0	-46.692	27.75
4	0	9	0	-42.012	25.457
5	0	12	0	-29.948	15.211
6	0	15	0	-24.315	31.06
7	0	18	0	-29.248	41.613

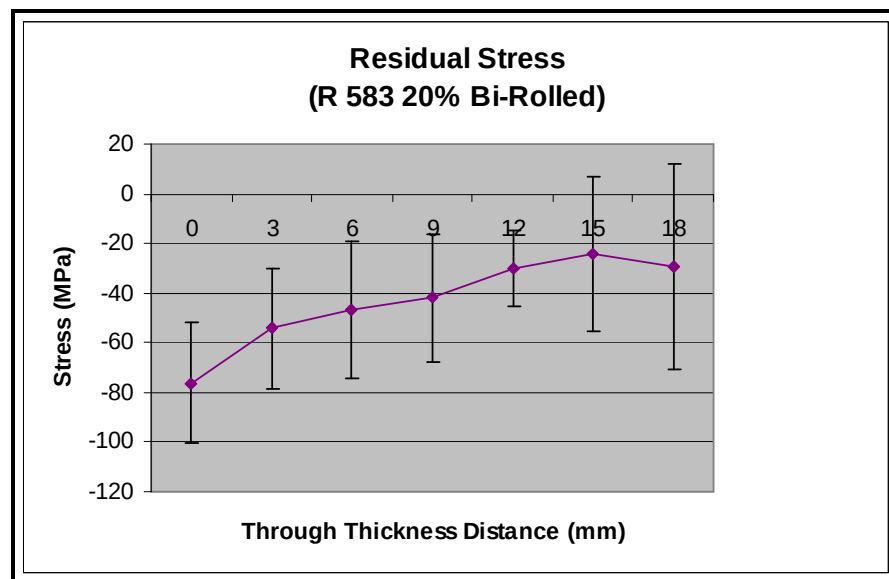


Figure 33: Residual Stress in Specimen R583 (20% Bi-Rolled)

3.3.3.2 Specimen Q114 (35% Bi-Rolled)

The longitudinal residual stress measured in specimen Q114 cold worked to 35% reduction is shown in Table 20 and Figure 34. The residual stress measured in specimen Q 114 was entire compressive but the magnitude of the stress was relatively lower compared to specimen R583. The stress level fluctuated between about -49 and -23 MPa across the through thickness of the specimen Q114.

Table 20: Residual Stress in Specimen Q114 (35% Bi-Rolled)

No	X-Position (mm)	Y-Position (mm)	Phi-Position (deg.)	Stress (MPa)	Error
1	0	0	0	-25.451	23.998
2	0	2.5	0	-42.183	13.059
3	0	5	0	-34.173	29.396
4	0	7.5	0	-22.986	12.882
5	0	10	0	-48.964	30.072

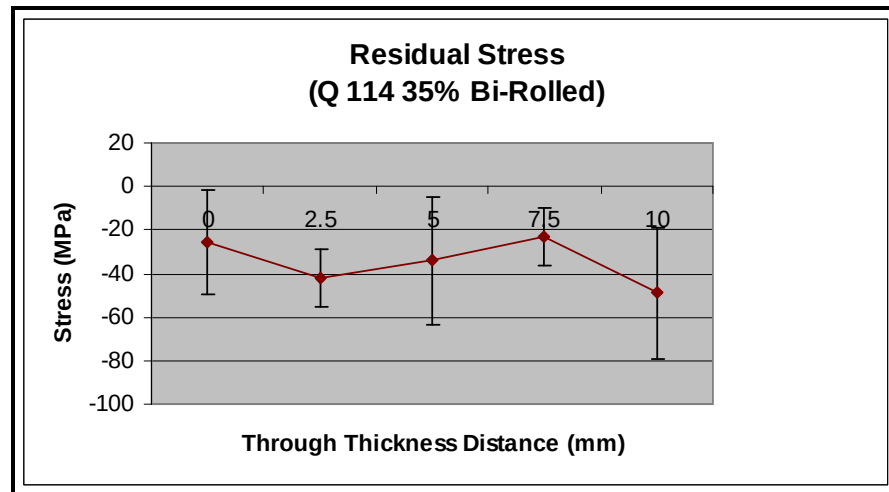


Figure 34: Residual Stress in Specimen Q114 (35% Bi-Rolled)

Figure 35 shows the graphical comparison between residual stress profiles of specimen R583 and Q114 cold worked to 20% and 35% reduction respectively. Specimen R583 residual stress profile showed a steady increase and was largely compressive compare to specimen Q114 with slight fluctuation and less compressive in nature.

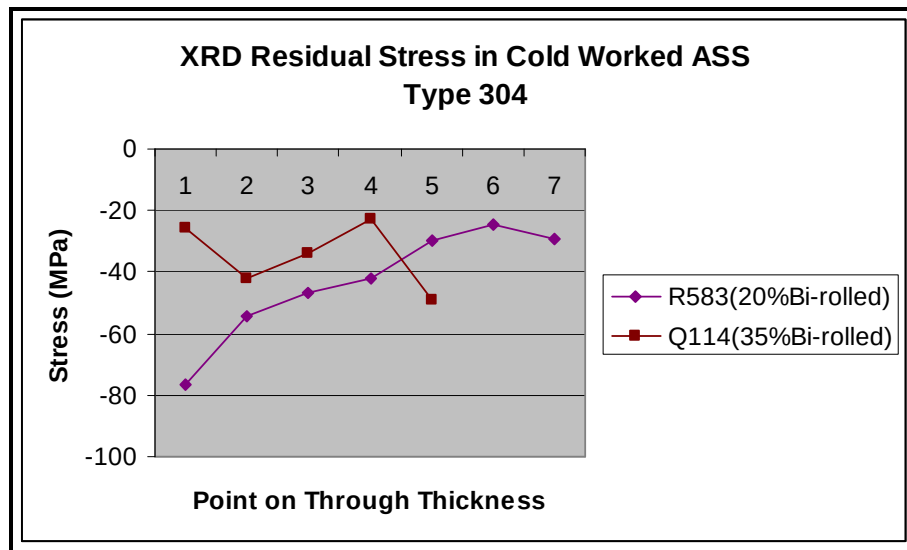


Figure 35: Residual Stress in Heat B Materials

3.3.4 Optical Metallography (Grain Boundaries Etching)

3.3.4.1 Specimen R583 (20% Bi-Rolled)

The result of optical metallography of specimen R583 etched with oxalic acid is shown in Figure 36. The S-L and S-T plane micrographs showed characteristic signs of grain elongation and slip bands while in T-L plane micrograph, there were little indications of slip bands and the grains were less elongated. On ordinary term, S-L and S-T plane revealed one dimensional grains elongation and appeared “sandwich-like” (Figure 36B and C), while in the T-L plane, the grains have suffered two dimensional stretches due to cross rolling and rather maintained a “pancake-like” shape (Figure 36A).

The result of grain size and grain elongation determined from Figure 36 is shown in Table 21. T-L has the largest grain size of 53 μ m. The grain sizes in S-L and S-T planes were the same at 35 μ m. The average grain size across the 3 planes was 40 μ m. The grains on S-L plane were more elongated than S-T plane and the least elongation occurred in T-L plane. The mean elongation ratio was about 1.5

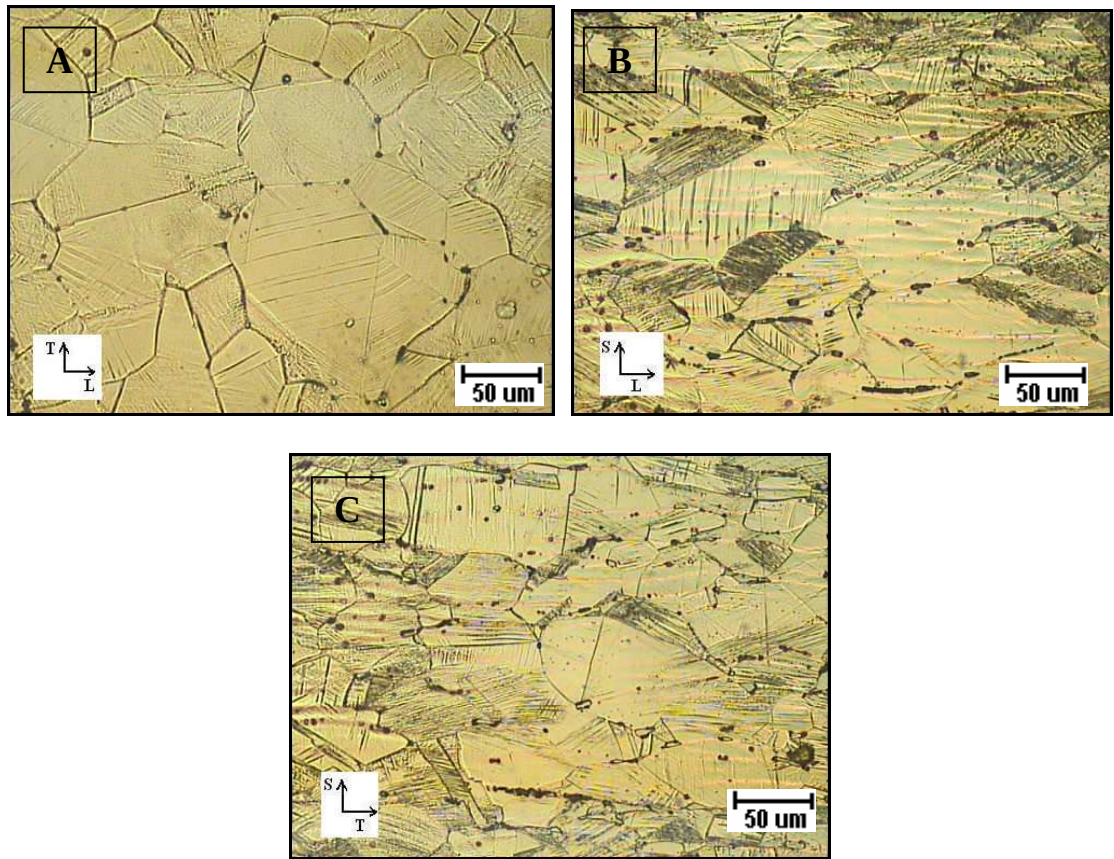


Figure 36: 3D Optical Micrographs of Specimen R583 (20% Pulled) Etched with Oxalic Acid

3.3.4.2 Specimen Q114 (35% Bi-Rolled)

The results of optical metallography of specimen Q114 etched with oxalic acid (Figure 37) were no much different from that of R583 except that the grains were more elongated and the slip bands were relatively more in intensity. The grains in T-L plane were larger and pancake like probably because of stretches in two directions. On the other hand, the grains in S-L and S-T planes were more elongated, narrow and sandwich like.

The grain size on T-L plane was the largest at about $85\mu\text{m}$. while on S-L and S-T planes, the grain sizes were $26\mu\text{m}$ and $31\mu\text{m}$ respectively. The average grain size in specimen Q114 was $41\mu\text{m}$. The elongation ratio on S-T plane was more than twice the ratio on S-L and T-L planes (Table 21). The average grain elongation across the specimen was about 2.

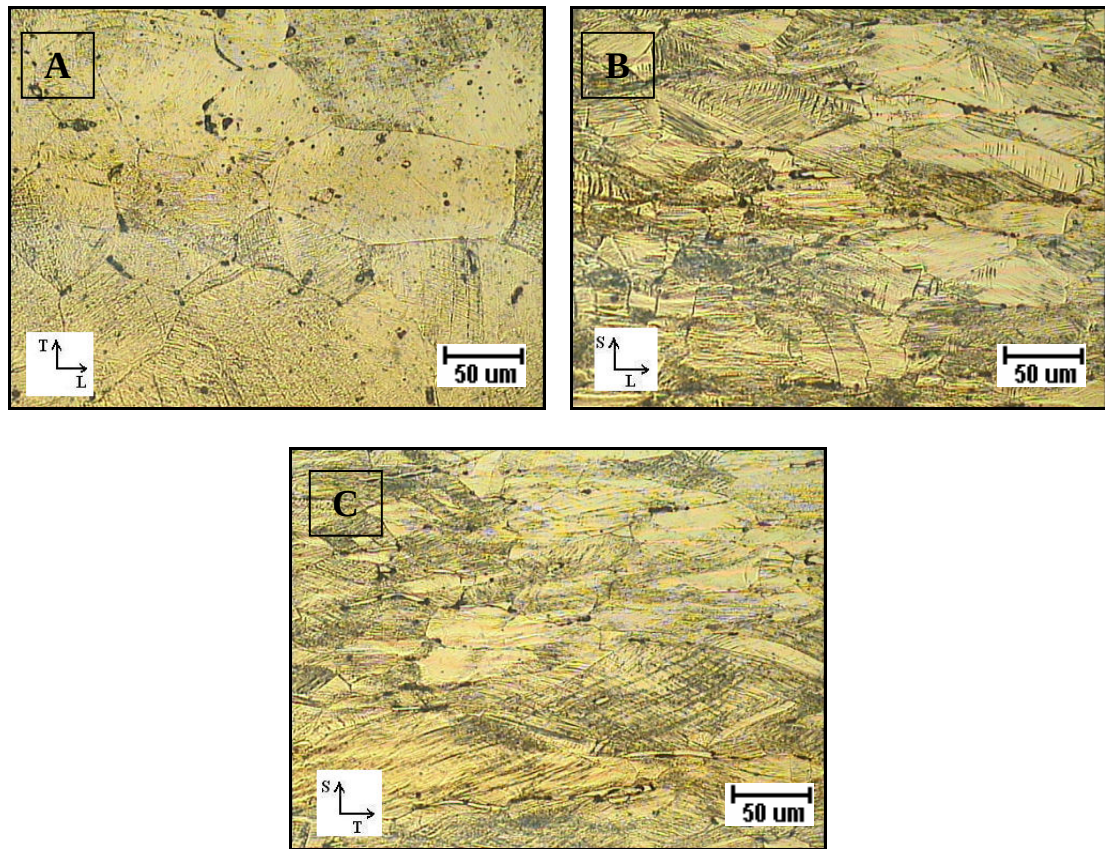


Figure 37: 3D Optical Micrographs of Specimen Q114 (35% Bi-Rolled) Etched with Oxalic Acid

Table 21: Grain Size and Elongation Ratio of Heat B Specimens

Specimen (%CW)	Grain Size μm				Elongation Ratio		
	T-L plane	S-L plane	S-T plane	Average Grain Size	T-L plane	S-L plane	S-T plane
R 583 (20%)	53	35	35	40	1.42	1.77	1.53
Q 114 (35%)	85	26	31	41	1.29	1.53	3.27

3.3.5 Optical Metallography (Delta Ferrite Etching)

3.3.5.1 Specimen R583 (20% Bi-Rolled)

The 3D optical micrographs of specimen R583 etched with NaOH are shown in Figure 38. Large amount of delta ferrite found were not quite surprising because of high content of ferrite

stabilising and relatively lower amount austenite stabilising alloying elements in the chemical composition of Heat B. Ferrite appeared flaky rather than elongated and evenly distributed in T-L plane. However, in S-L and S-T planes, they appeared more elongated in their localised concentration within the austenite matrix.

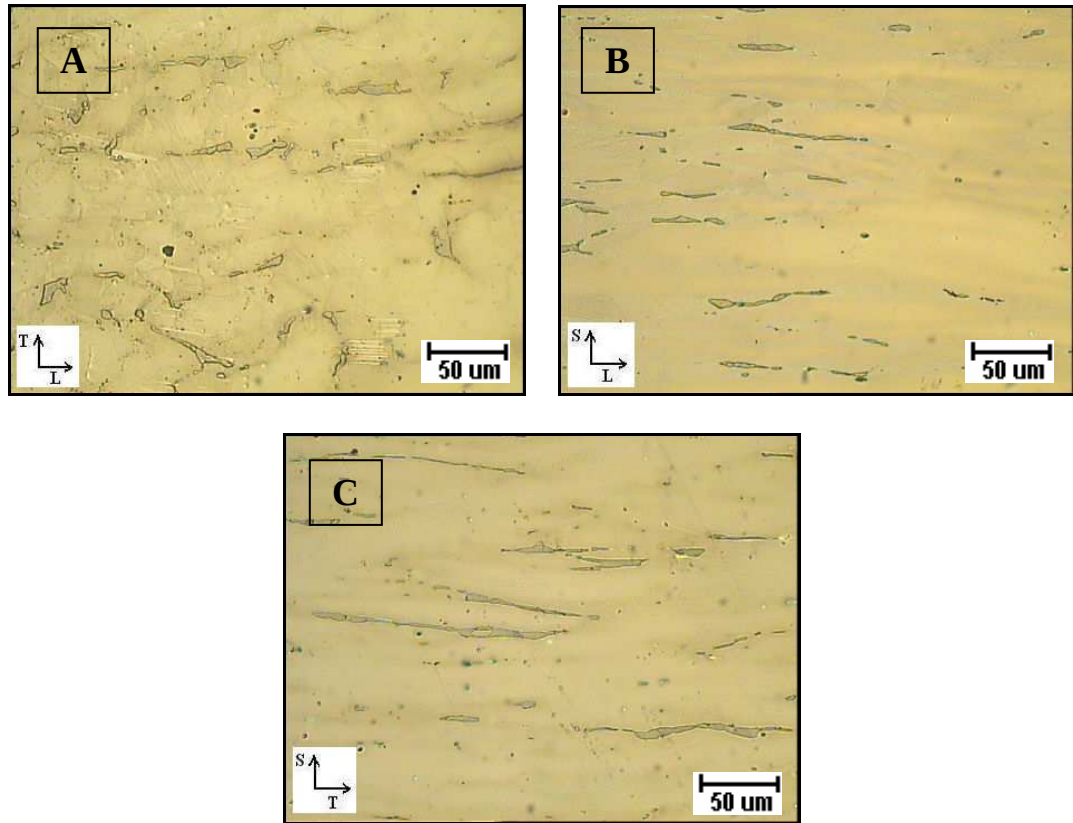


Figure 38: 3D Optical Micrographs of Specimen R583 (20% Bi-Rolled) Etched with NaOH Acid

3.3.5.2 Specimen Q114 (35% Bi-Rolled)

Delta ferrite in specimen Q114 appeared like particles evenly scattered in T-L plane (Figure 39). But in S-L and S-T planes, they appeared as continuous broken streaks sandwiched between austenite grains.

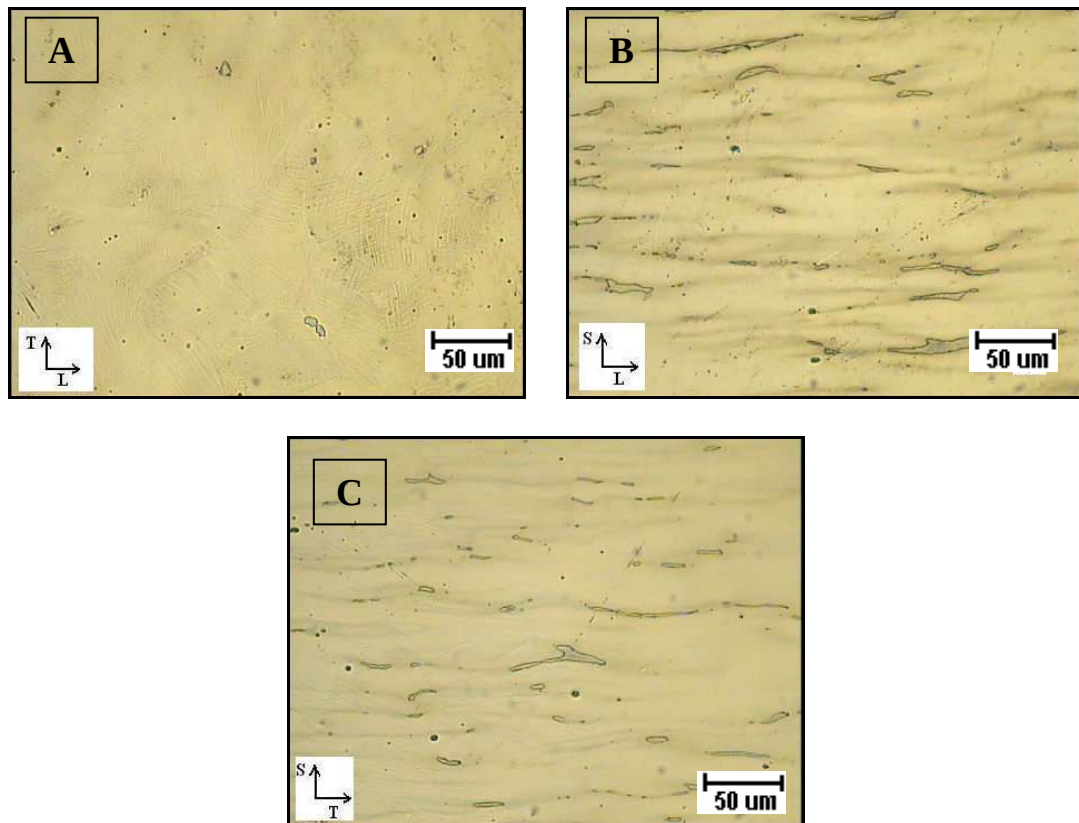


Figure 39: 3D Optical Micrographs of Specimen Q114 (35% Bi-Rolled) Etched with NaOH

3.3.6 Optical Metallography (Martensite Etching)

3.3.6.1 Specimen R583 (20% Bi-Rolled)

The results of 3D metallography of specimen R583 etched with picral to reveal SIM is shown in Figure 40. Martensite occurs as laths which are aligned parallel to one another. Laths of martensite may also group together to form larger structural entities inform of rectangular or trapezoidal blocks or packet within the prior austenite grain boundary [12]. In Figure 40, the S-L plane contained more packets of SIM than found in S-T plane. The amount of SIM found in T-L plane was relatively low compared to S-L and S-T planes.

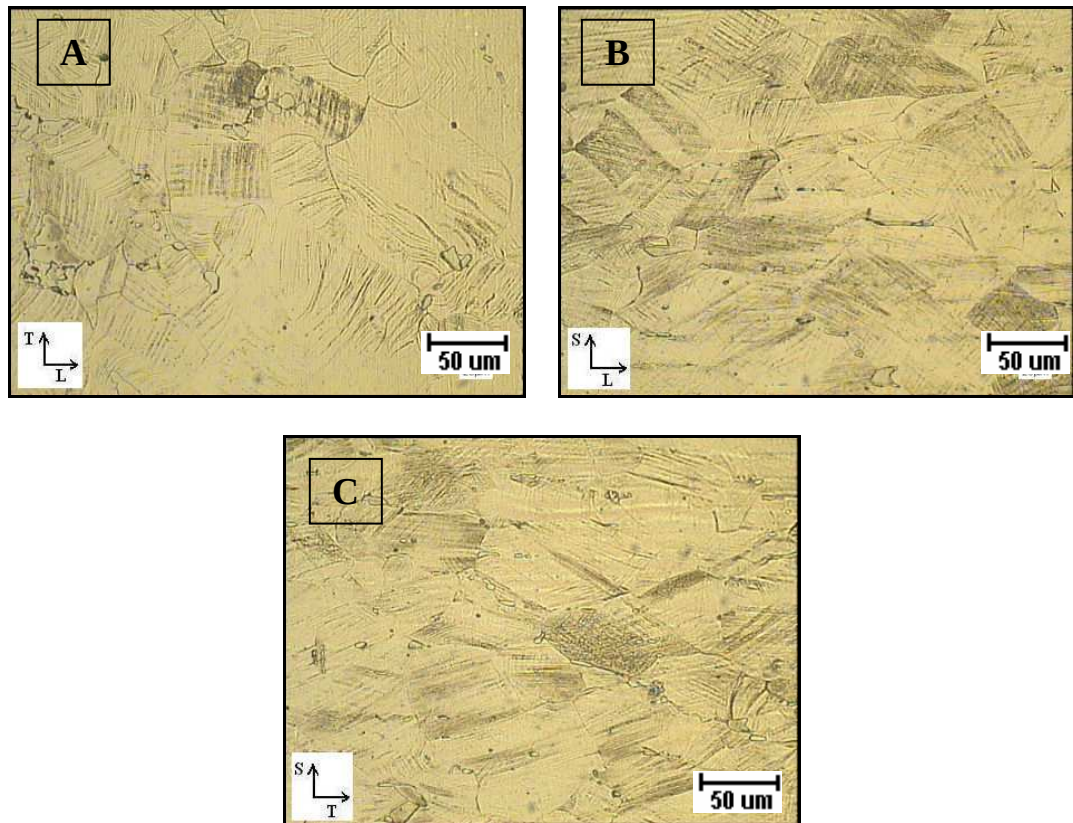
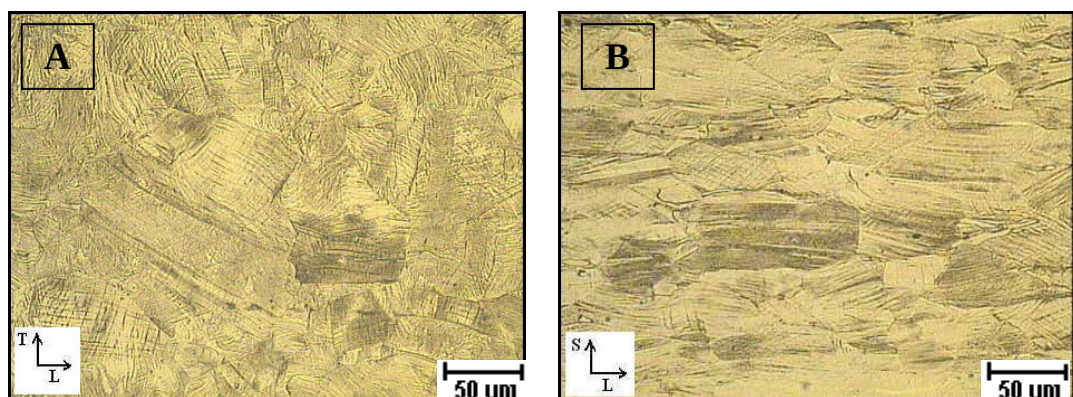


Figure 40: 3D Optical Micrographs of Specimen R583 (20% Bi-Rolled) Etched with Picral

3.3.6.2 Specimen Q114 (35% Bi-Rolled)

The amount of SIM found specimen Q114 (Figure 41) as indicated by the intensity of the packets was relatively greater than was found in specimen R583. The higher amount of SIM is consistent with the higher cold reduction in specimen Q114. S-L and S-T plane showed greater amount of SIM than T-L plane.



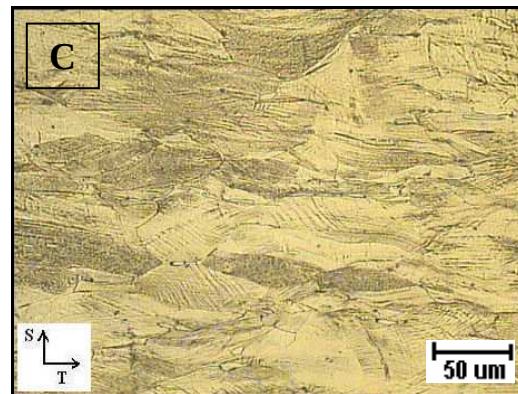
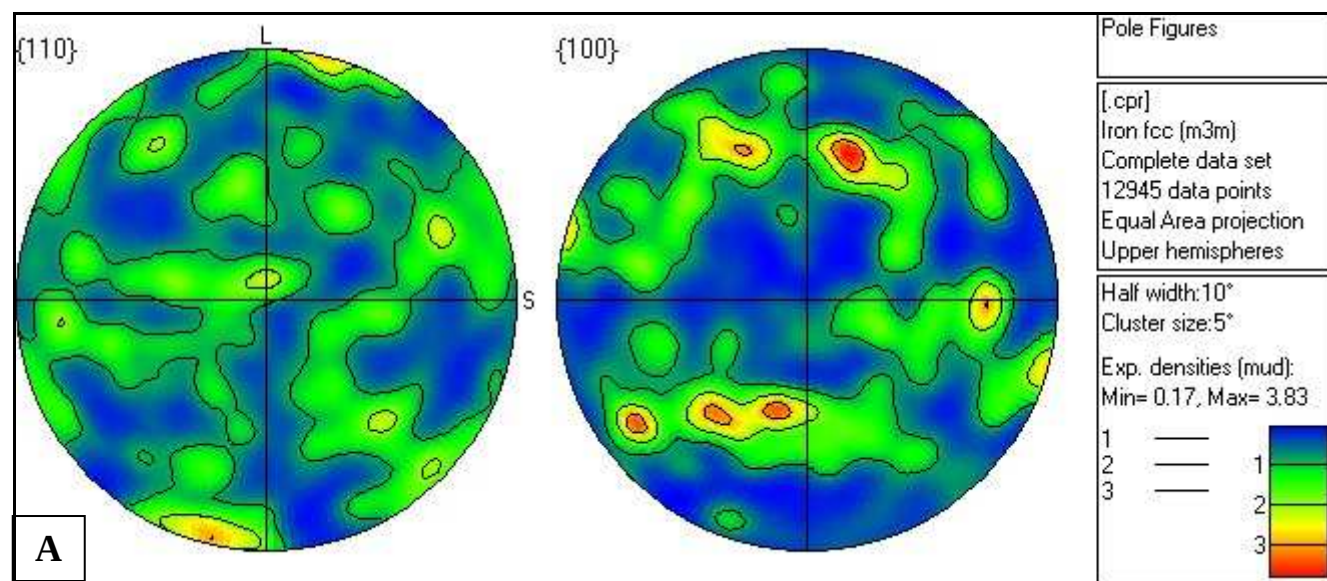


Figure 41: 3D Optical Micrographs of Specimen Q114 (35% Bi-Rolled) Etched with Picral

3.3.7 Electron Back Scatter Diffraction

3.3.7.1 Specimen R583 (20% Bi-Rolled)

The results of texture on S-L and S-T planes of specimen R583 are shown as pole figures in Figure 42A and B. The texture in both S-L and S-T planes were slightly random but stronger in crystallographic planes $\{100\}$ than $\{110\}$ planes. S-T plane showed relatively stronger texture and was less random than S-L plane as indicated by maximum value of the MUD.



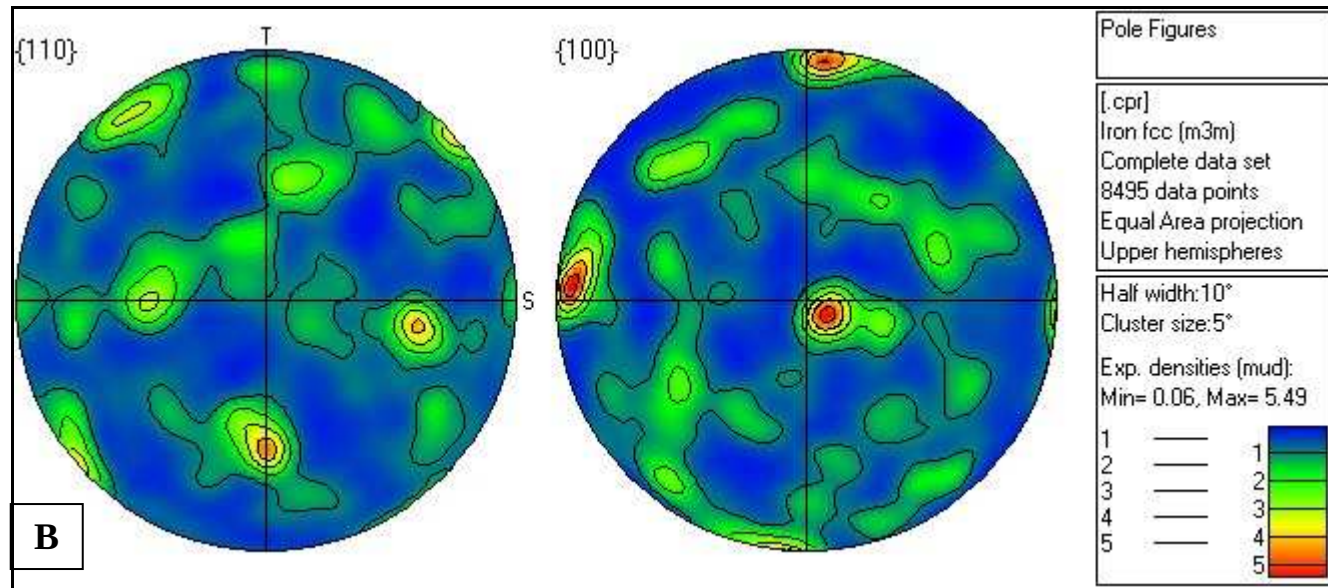


Figure 42: Pole Figures of Specimen R583 (20% Bi-Rolled) on S-L Plane (A) and S-T Plane (B)

3.3.7.2 Specimen Q114 (35% Bi-Rolled)

There was no texture data available for specimen Q114. Specimen Q114 could not be indexed during the EBSD. The reason was probably due to large amount of plastic strain in the grains caused by heavy cold work during the bi-directional rolling.

3.4 Characterisation of Heat C

Heat C material is characterised by low carbon and Manganese contents. On the other hand, Heat C also contained significantly higher amount of Silicon and sulphur contents in its chemical composition (Table 1). The net effect of these alloying elements may improve the stability of ferrite phase in Heat C than other materials in the test. The CT specimens Q016 cold worked to 20% reduction by bi-directional rolling and Q021 with 40% reduction by uni-directional rolling were obtained from Heat C material. The orientation of the CT specimens was defined as semi orientation (or L-T orientation in ASTM notation) (Figure 1). The results of characterisation of Heat C materials are as follow.

3.4.1 Vickers Hardness

3.4.1.1 Specimen Q016 (20% Bi-Rolled)

The results of Vickers hardness measured on specimen Q016 is shown in Table 22 and Figure 43. The mean hardness values at the edges of the specimen adjacent to the rolls were higher than the central region of the specimen. The mean hardness fluctuated between 290HV30 at the central region of the specimen and 325HV30 at the edge. The average hardness across the specimen was 300HV30.

Table 22: Vickers Hardness of Specimen Q 016 (20% Bi-Rolled)

Through Thickness Distance (mm)	HV30 @ 1	HV30 @ 2	HV30 @ 3	Mean HV30
1	308	315	324	316
2	299	297	314	303
3	283	295	293	290
4	283	293	305	294
5	291	295	297	294
6	299	299	309	302
7	315	304	307	309
8	328	307	320	318
9	318	311	331	320
10	314	314	326	318
11	315	321	339	325

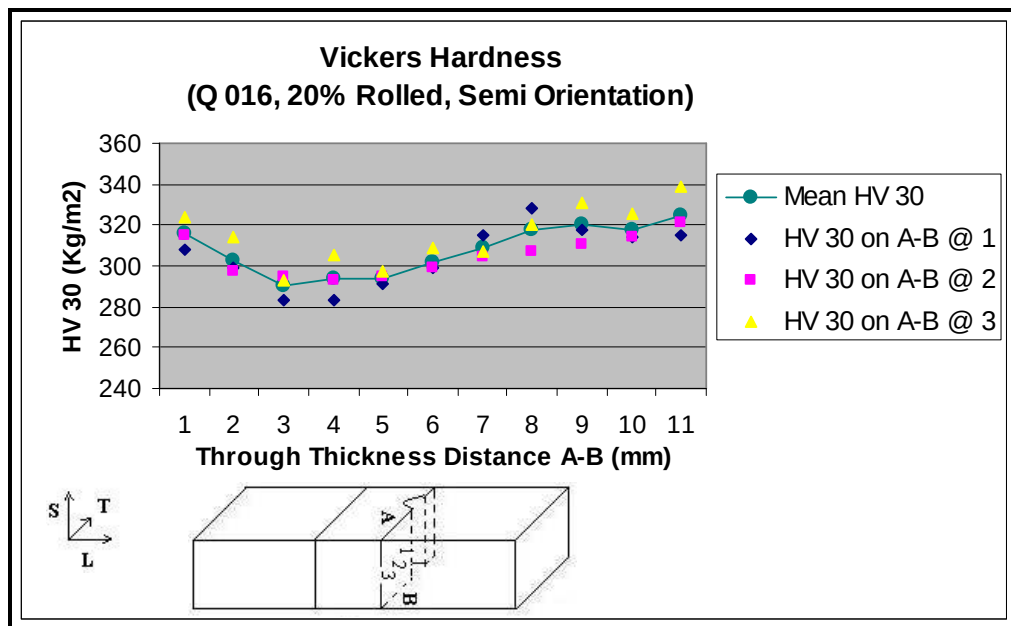


Figure 43: Vickers Hardness of Specimen Q 016 (20% Bi-Rolled)

3.4.1.2 Specimen Q021 (40% Uni-Rolled)

The Vickers hardness results of specimen Q021 (Table 23 and Figure 44) show consistent increase in hardness with percent cold reduction. The mean hardness values at the edges were slightly higher than central region of the specimen. The mean hardness varied between 391HV30 and 414HV30, and the average hardness across the through thickness measured at 400HV30.

Table 23: Vickers Hardness of Specimen Q 021 (40% Uni-Rolled)

Through Thickness Distance (mm)	HV30 @ 1	HV30 @ 2	HV30 @ 3	Mean HV30
1	396	413	413	407
2	403	432	406	414
3	412	423	386	407
4	377	413	399	396
5	385	403	405	398
6	387	389	392	389
7	389	381	405	392
8	386	381	406	391
9	399	405	407	404
10	403	414	409	409
11	375	407	409	397

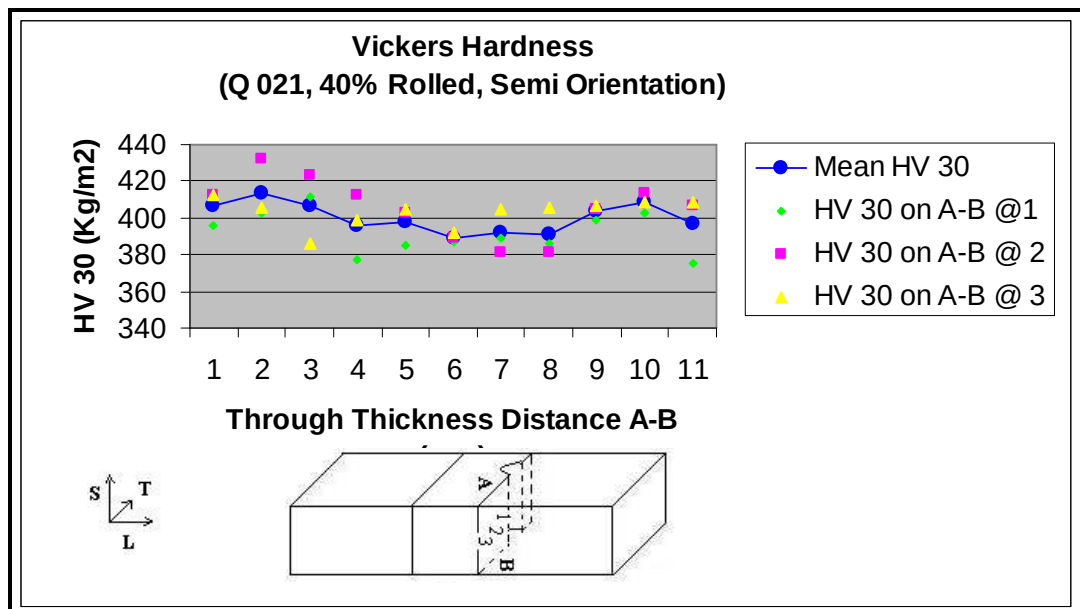


Figure 44: Vickers Hardness of Specimen Q 021 (40% Uni-Rolled)

Figure 45 compares and contrasts the hardness profiles in specimens Q016 and Q021. Specimen Q021 hardness values were more uniform and significantly higher than specimen Q016 with a margin of about 100HV30.

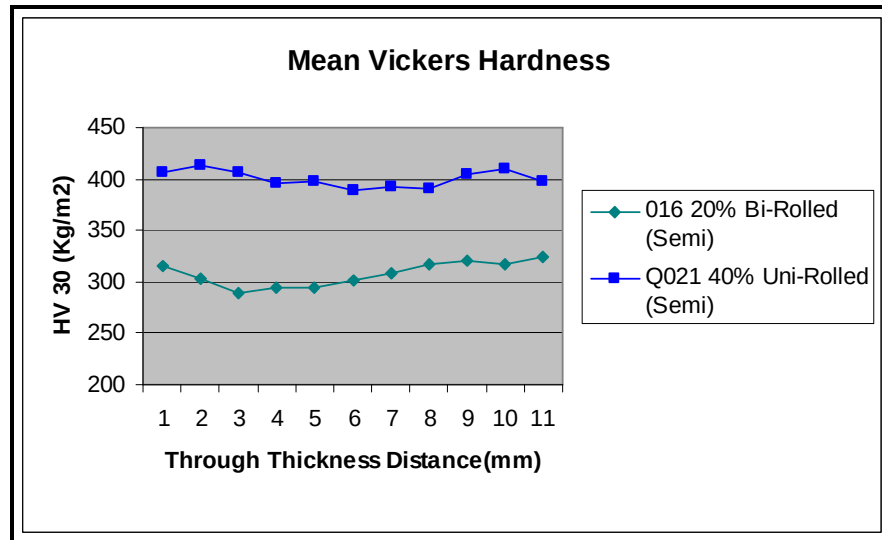


Figure 45: Mean Vickers Hardness of Heat C Specimens

3.4.2 X-Ray Diffraction (Strain Induced Martensite)

3.4.2.1 Specimen Q016 (20% Bi-Rolled)

The results of quantification of SIM in specimen Q016 by XRD is shown in Table 24 and Figure 46. The amount of SIM found in specimen Q016 was 6% and the rest was presumed to be austenite. The amount of SIM in specimen Q016 was quite low compare to specimen Q021.

3.4.2.2 Specimen Q021 (40% Uni-Rolled)

The amount of SIM found in specimen Q021 was relatively high and measured up to 21%. The remaining balance was considered to be austenite (Table 24). The high amount of SIM in specimen Q021 is not unexpected because it's high level of cold reduction.

Table 24: Strain Induced Martensite in Heat C Specimens

Samples	C W (%)	Integrated Intensity, <i>I</i>							V _V	Martensite (%)
		γ-200	α-200	γ-220	α-211	γ-311	γ-222	α-220		
Q016	20	8090	307	2657	256	4760	2661	95	0.9435	6
Q021	40	4414	644	1412	728	2960	1940	451	0.792	21

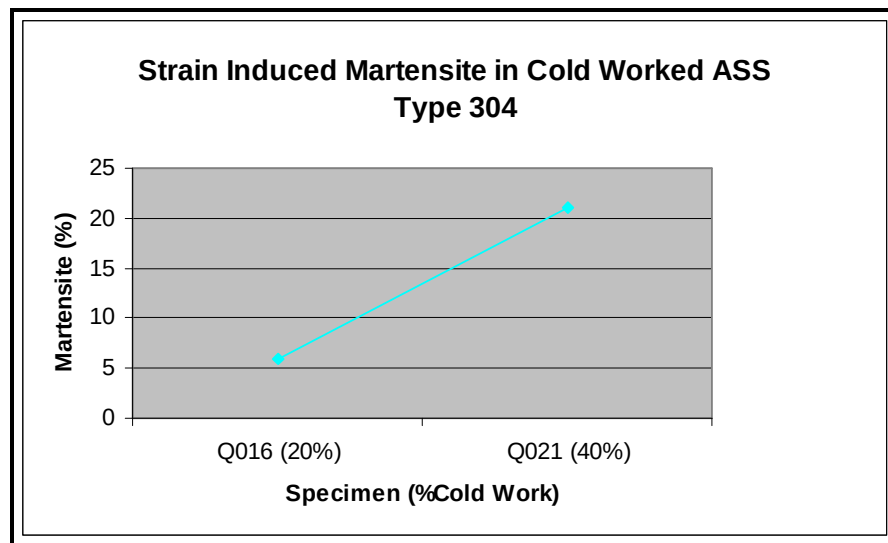


Figure 46: Strain Induced Martensite in Heat C Specimens

3.4.3 X-Ray Diffraction (Residual Stress)

3.4.3.1 Specimen Q016 (20% Bi-Rolled)

The longitudinal residual stress measured in specimen Q016 was largely compressive. The stress value varied between about -150MPa and tensile stress value of about 15MPa (Table 25 and Figure 47). The error values were within the acceptable limit.

Table 25: Residual Stress in Specimen Q016 (20% Bi-Rolled)

No	X-Position (mm)	Y-Position (mm)	Phi-Position (deg.)	Stress (MPa)	Error
1	0	0	0	-50.992	25.507
2	0	2.5	0	-149.809	25.27
3	0	5	0	-115.735	22.005
4	0	7.5	0	-78.069	22.835
5	0	10	0	14.883	23.243

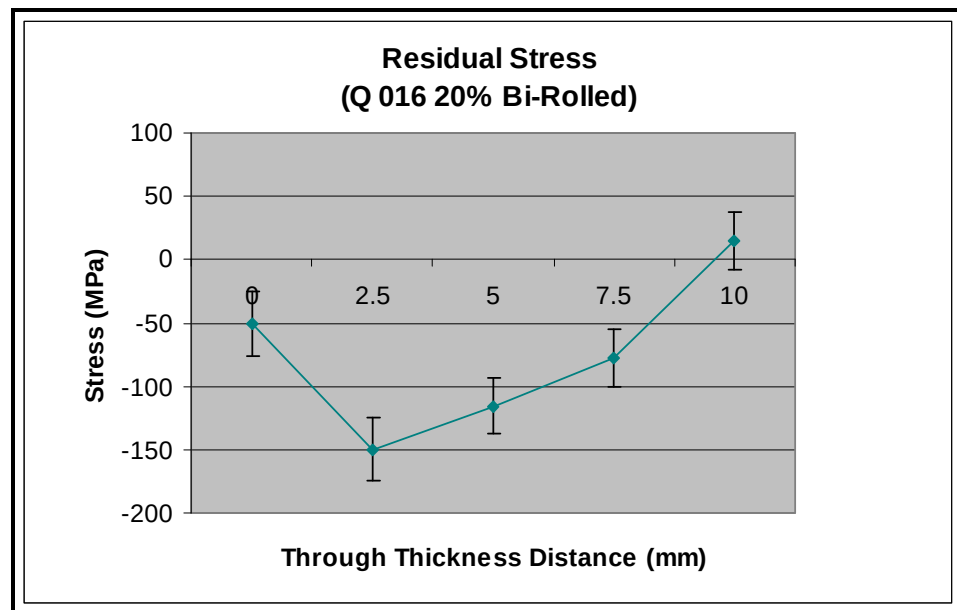


Figure 47: Residual Stress in Specimen Q016 (20% Bi-Rolled)

3.4.3.2 Specimen Q021 (40% Uni-Rolled)

The results of residual stress measured in specimen Q021 (Table 26 and Figure 48) were highly contrasting to values obtained from specimen Q016. The entire stress values were tensile residual stresses, quite contrasting to largely compressive residual stress obtained in specimen Q016. The tensile residual stress reduced down from 477MPa to 286MPa across the through thickness.

Figure 49 shows the residual profiles of specimen Q016 and Q021 across the through thickness. Specimen Q021 was completely tensile while the stresses in Q016 were predominantly compressive residual stress.

Table 26: Residual Stress in Specimen Q021 (40% Uni-Rolled)

No	X-Position (mm)	Y-Position (mm)	Phi-Position (deg.)	Stress (MPa)	Error
1	0	0	0	477.604	40.576
2	0	2.25	0	413.336	32.26
3	0	4.5	0	365.267	36.511
4	0	6.75	0	301.576	36.919
5	0	9	0	286.175	33.207

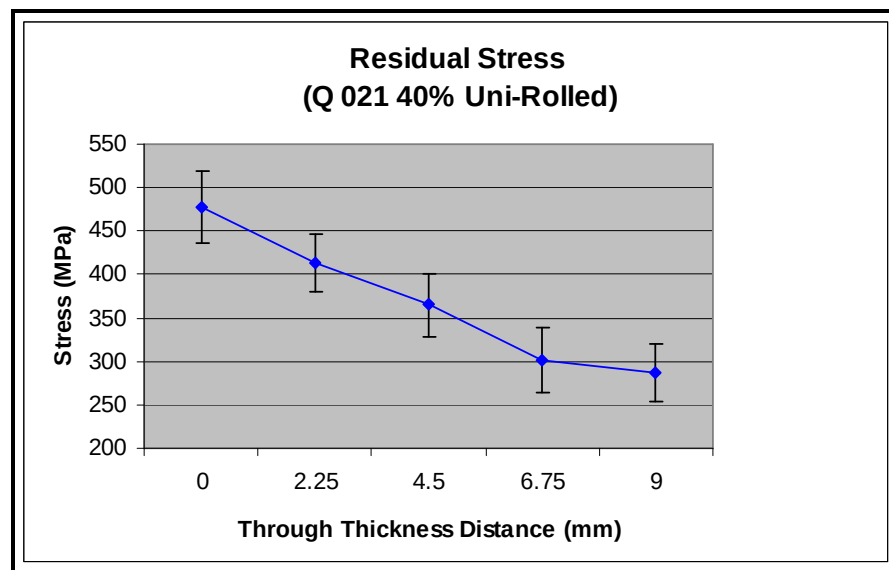


Figure 48: Residual Stress in Specimen Q021 (40% uni-Rolled)

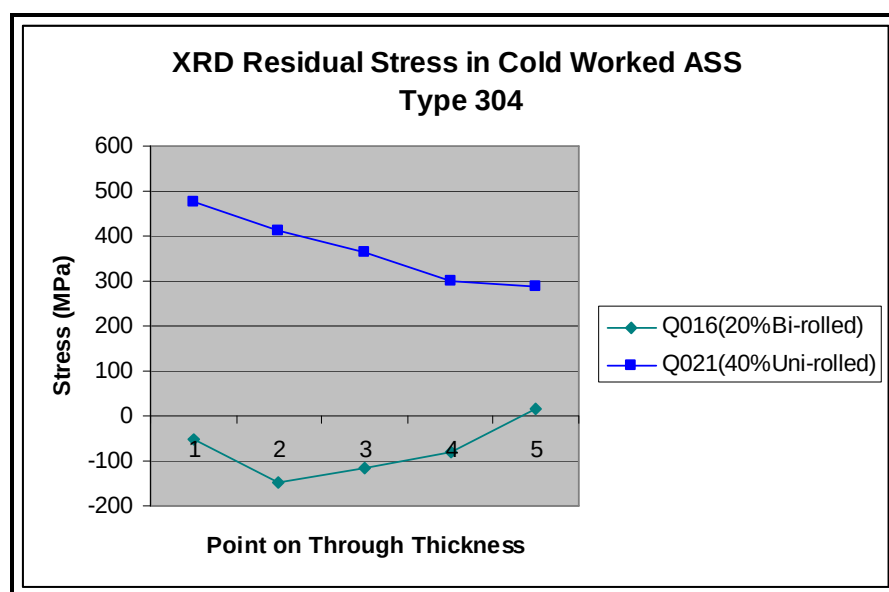


Figure 49: Residual Stress in Heat C Materials

3.4.4 Optical Metallography (Grain Boundaries Etching)

3.4.4.1 Specimen Q016 (20% Bi-Rolled)

The 3D micrographs of specimen Q016 etched with oxalic acid are shown in Figure 50. The micrographs revealed the slip bands and the grain boundaries. The grains on the T-L plane appeared like pancakes as usual because of two dimensional strains caused by cross rolling. However, the grains in S-L and S-T planes were slightly elongated in L and T directions.

Grain size determination from Figure 50 shows that grains on S-L plane have the largest grain size of $35\mu\text{m}$ and the grains on the S-T and T-L planes were relatively closed at $29\mu\text{m}$ and $31\mu\text{m}$ respectively (Table 27). The average grain size across the 3 planes was $32\mu\text{m}$. The highest elongation ratio of 1.83 occurred on S-L plane. Grains were slightly more elongated on T-L plane than on S-T plane. The average elongation ratio across the 3D was about 1.5

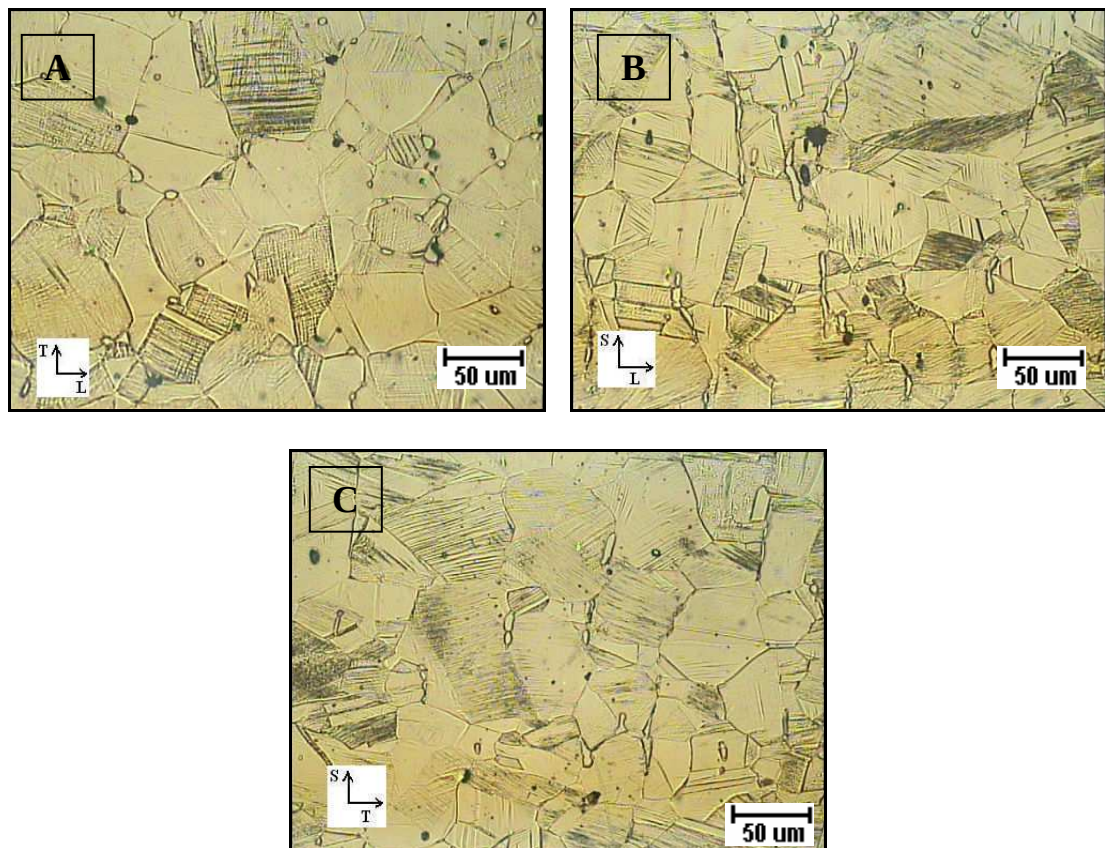


Figure 50: 3D Optical Micrographs of Specimen Q016 (20% Bi-Rolled) Etched with Oxalic Acid

3.4.4.2 Specimen Q021 (40% Uni-Rolled)

The grains in 3D micrographs of specimen Q021 etched with oxalic acid show sign of strain in one direction (Figure 51). The result of grain size determination in Table 27 shows that T-L plane has the largest grain size of $33\mu\text{m}$, while S-L and S-T plane have $31\mu\text{m}$ and $30\mu\text{m}$ respectively. The highest elongation ratio of 2.13 occurred on S-T plane. Elongation ratio on S-L plane was slightly less than S-T plane at the value of 2. Elongation ratio on T-L plane measured at about 1.5 and the average elongation ratio across the 3D was almost twice.

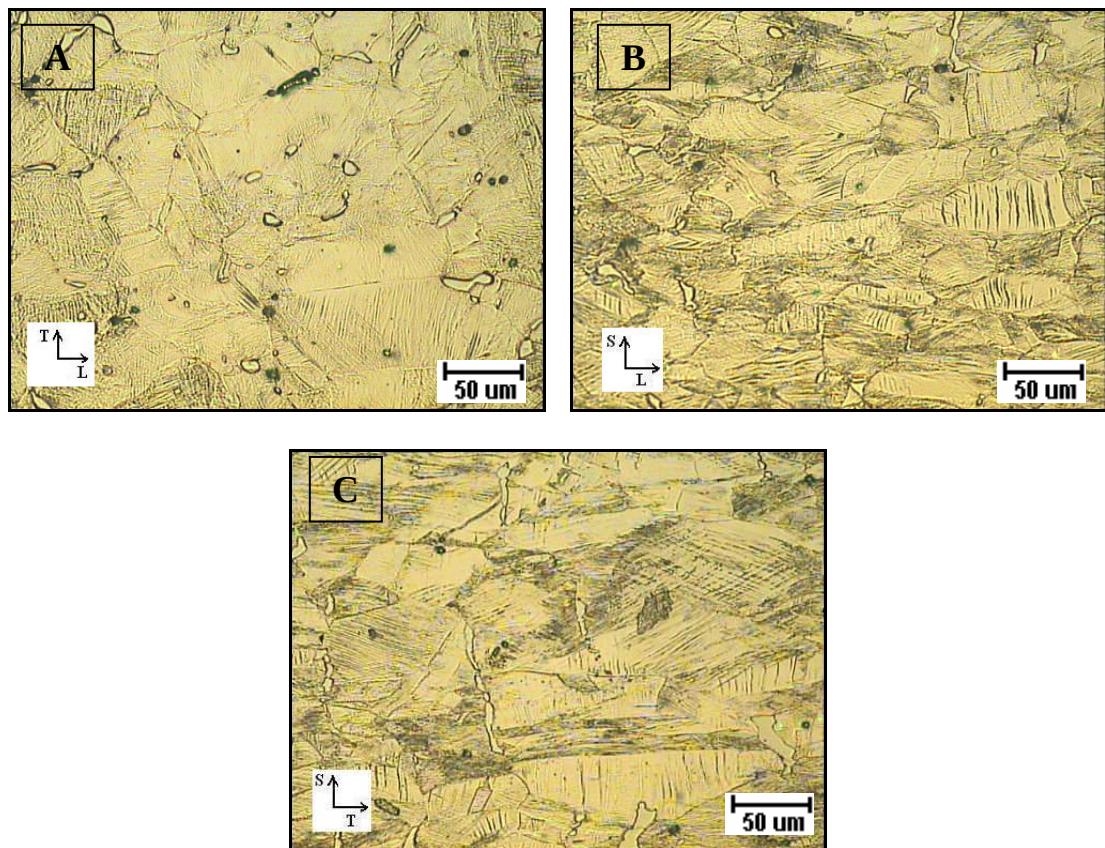


Figure 51: 3D Optical Micrographs of Specimen Q021 (40% Uni-Rolled) Etched with Oxalic Acid

Table 27: Grain Size and Elongation Ratio of Heat C Specimens

Specimen (%CW)	Grain Size μm				Elongation Ratio		
	T-L plane	S-L plane	S-T plane	Average Grain Size	T-L plane	S-L plane	S-T plane
Q 016 (20%)	31	35	29	32	1.38	1.83	1.26
Q 021 (40%)	33	31	30	31	1.54	2	2.13

3.4.5 Optical Metallography (Delta Ferrite Etching)

3.4.5.1 Specimen Q016 (20% Bi-Rolled)

The 3D micrographs of delta ferrite etching of specimen Q016 with NaOH is shown in Figure 52. The amount delta ferrite found was quite significant compared to the previously tested materials. S-L and S-T planes revealed the ferrite elongated within the austenite matrix. But in T-L plane, ferrite appeared rather flaky and scattered within the austenite matrix.

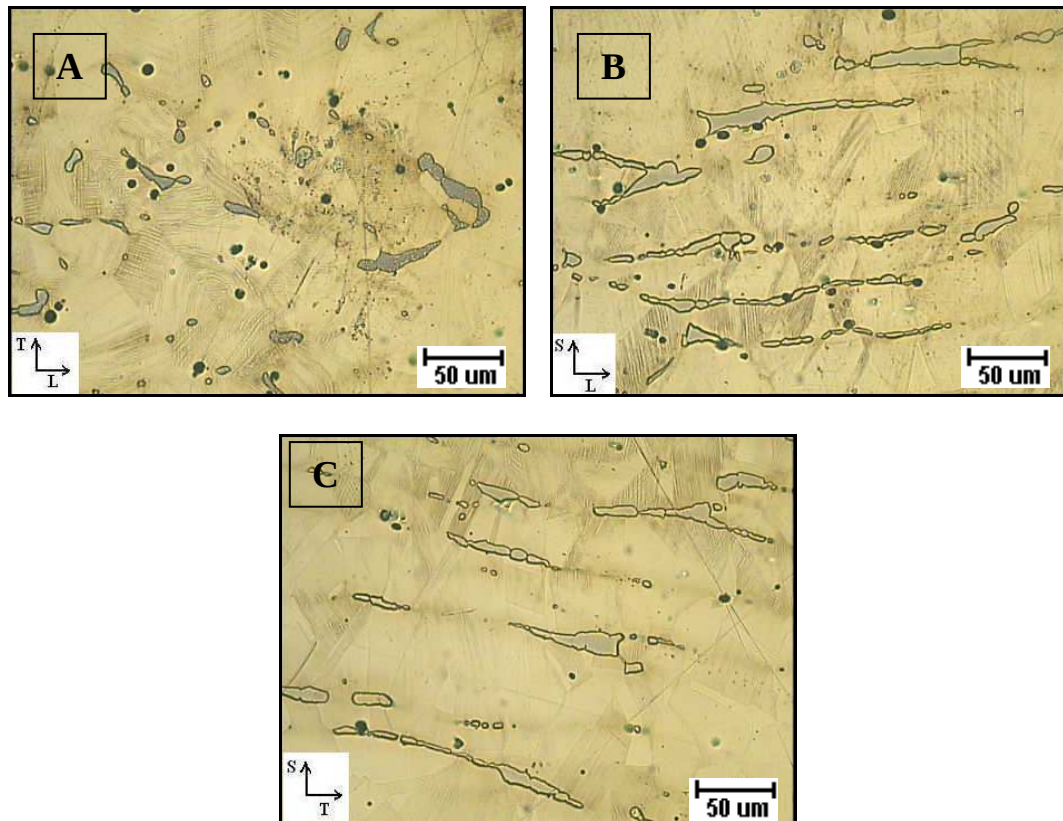


Figure 52: 3D Optical Micrographs of Specimen Q016 (20% Bi-Rolled) Etched with NaOH

3.4.5.2 Specimen Q021 (40% Uni-Rolled)

Figure 53 shows delta ferrite in specimen Q021. The delta ferrite in S-L and S-T plane appeared slightly elongated but broken up into pieces. But, in T-L plane, they appeared like flakes without any sign of elongation and scattered within the austenite matrix.

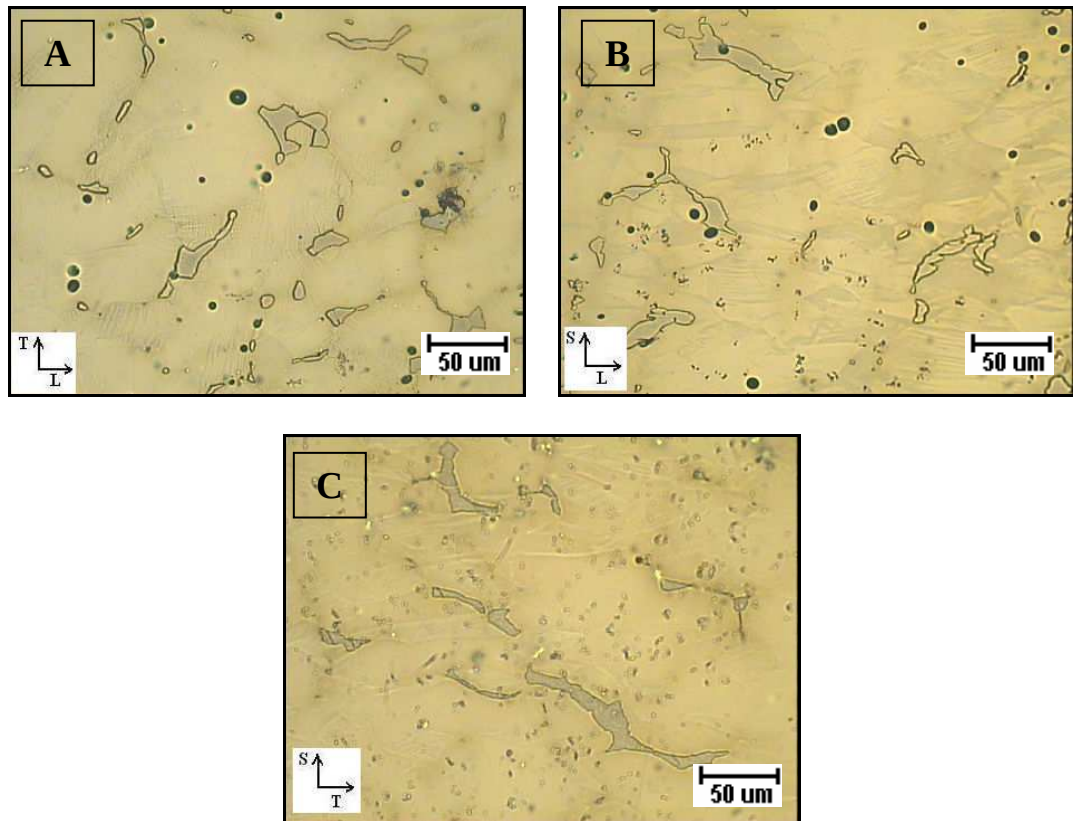


Figure 53: 3D Optical Micrographs of Specimen Q021 (40% Uni-Rolled) Etched with NaOH

3.4.6 Optical Metallography (Martensite Etching)

3.4.6.1 Specimen Q016 (20% Bi-Rolled)

The amount of SIM found in specimen Q016 was relatively small compare with specimen Q021. Figure 54 shows the 3D optical micrographs of specimen Q016 etched with picral. The packet of martensite appeared faded within the austenite grain boundaries. The amount of martensite in S-T plane was relatively low compared to S-L and T-L planes. The low amount of martensite revealed in Figure 54 is consistent with the XRD result in Table 24.

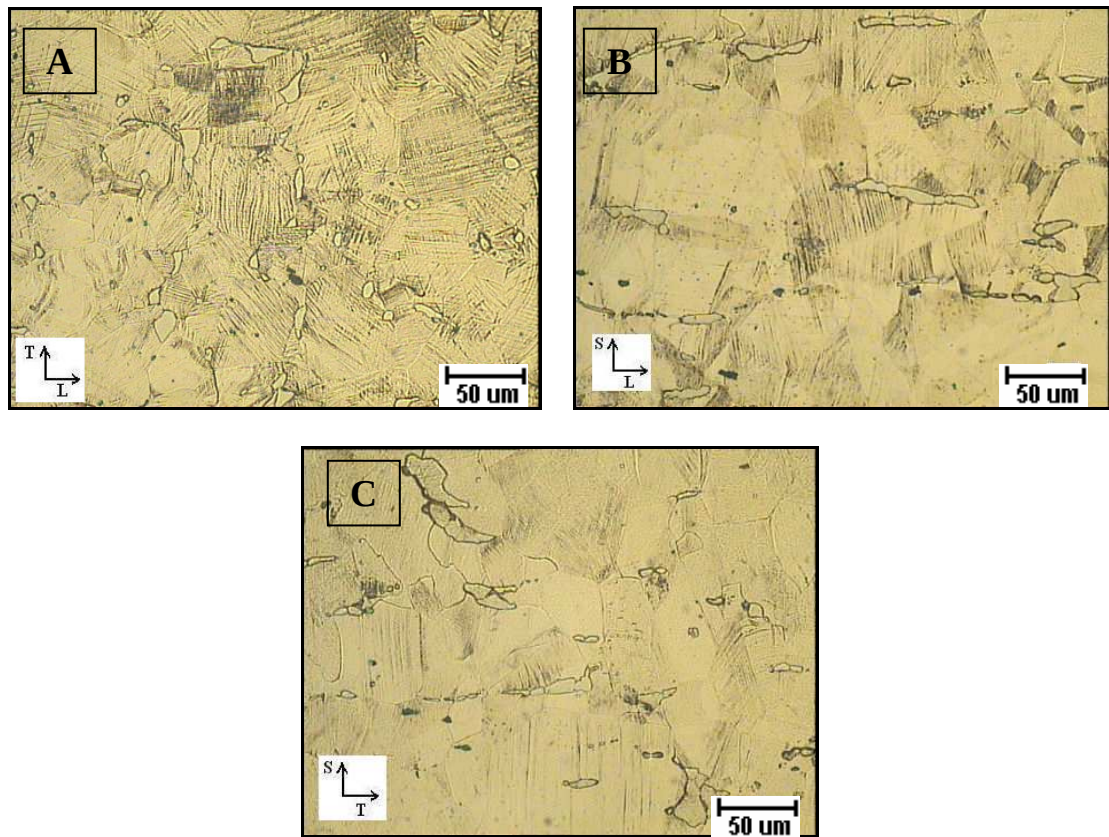


Figure 54: 3D Optical Micrographs of Specimen Q016 (20% Bi-Rolled) Etched with Picral

3.4.6.2 Specimen Q021 (40% Uni-Rolled)

The amount of SIM in specimen Q021 as shown by the optical micrographs in Figure 55 was relatively higher compare to specimen Q016. The S-T plane micrograph indicated higher amount of SIM by contrast to S-L and T-L planes. The high amount of SIM in specimen Q021 is consistent with heavy cold reduction of about 40%.

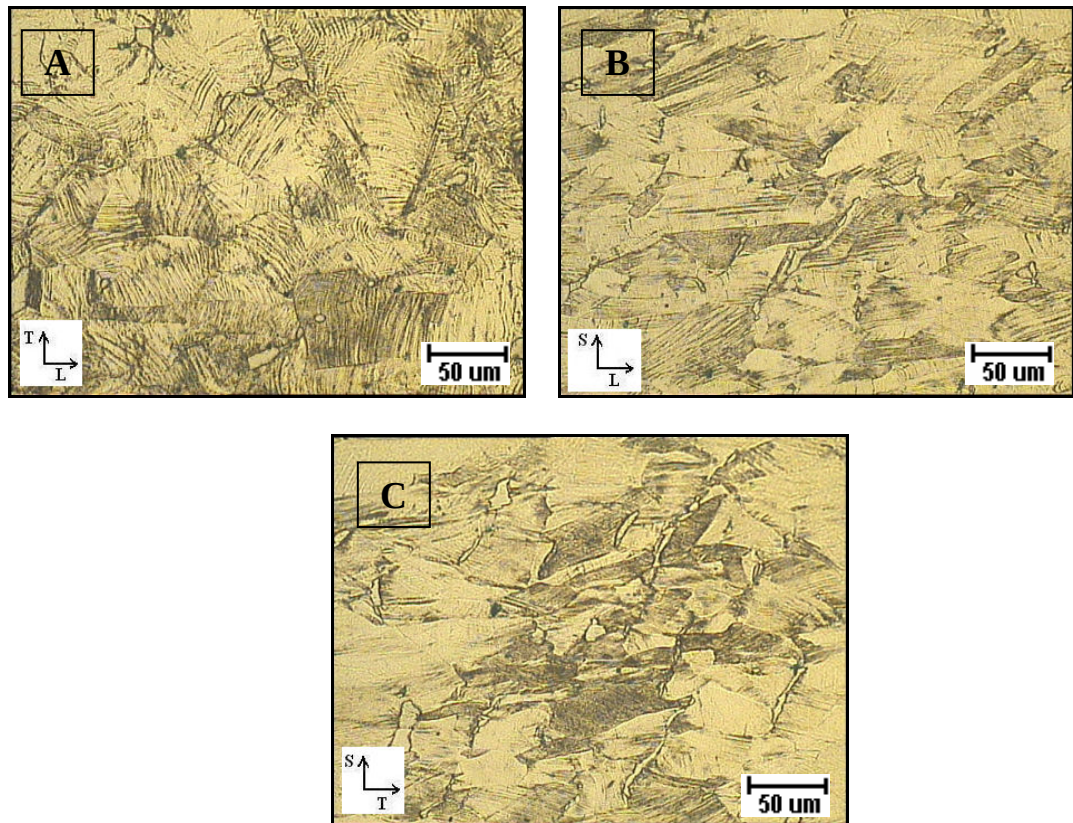


Figure 55: 3D Optical Micrographs of Specimen Q021 (40% Uni-Rolled) Etched with Picral

3.4.7 Electron Back Scatter Diffraction

3.4.7.1 Specimen Q016 (20% Bi-Rolled)

Figure 56 shows the $\{110\}$ and $\{100\}$ pole figures obtained from S-L and S-T planes of specimen Q016. Crystallographic planes $\{100\}$ show stronger texture than $\{110\}$ planes. The texture on both S-L and S-T planes were slightly random. However, stronger texture occurred on S-T plane than S-L plane as indicated by the MUD. The maximum MUD in S-T and S-L planes were 2.86 and 2.56 respectively.

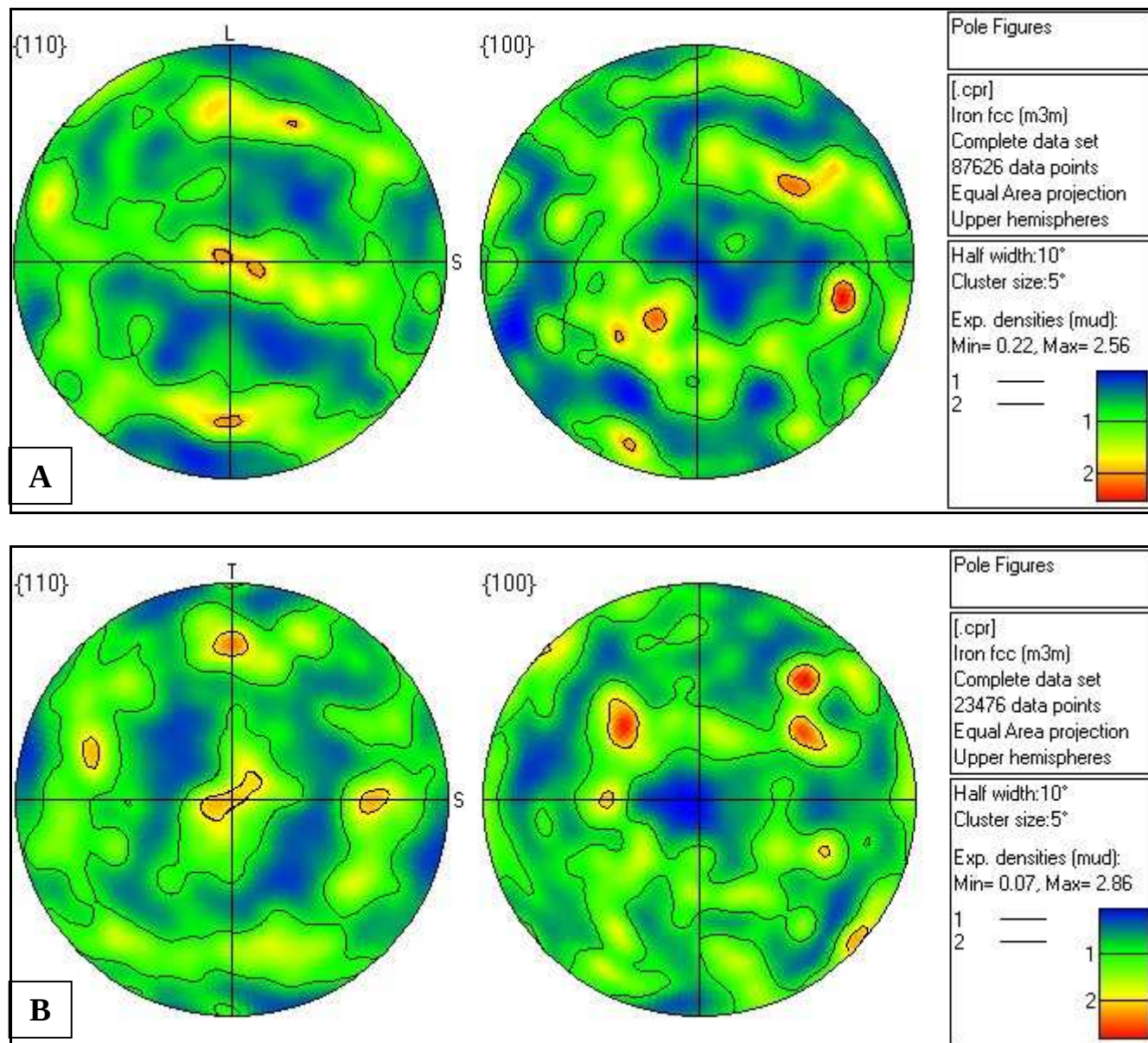


Figure 56: Pole Figures of Specimen Q016 (20% Bi-Rolled) on S-L Plane (A) and S-T Plane (B)

3.4.7.2 Specimen Q021 (40% Uni-Rolled)

The pole figures of specimen Q021 (Figure 57) were quite unique to those of specimen Q016. Specimen Q021 was uni-directionally rolled to 40% reduction. The pole figures were obviously less random and in contrast to bi-directionally rolled specimens, crystallographic planes {110} were more textured than {100} planes. The maximum MUD in S-L plane was 5.54 and in S-T plane, it was 3.78. The S-L plane showed stronger texture than S-T plane.

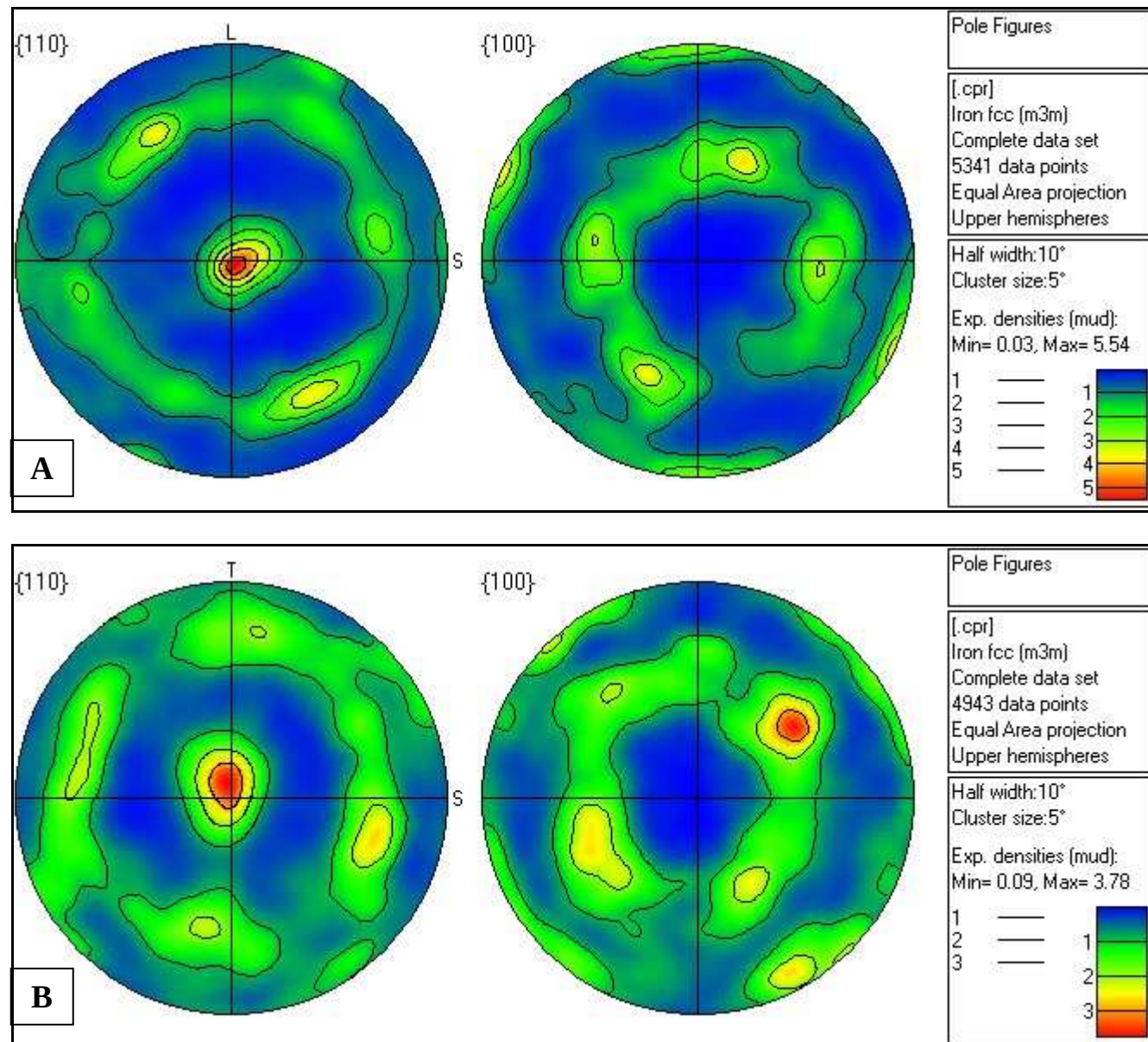


Figure 57: Pole Figures of Specimen Q021 (40% Uni-Rolled) on S-L Plane (A) and S-T Plane (B)

3.5 Results Summary

The summary results of some of the experimental techniques for all the test specimens are illustrated below in tables and figures.

3.5.1 Vickers Hardness

Table 28 and Figures 58 and 59 summarised the Vickers hardness results of all the test specimens. The mean Vickers hardness is shown in Table 28. Figure 58 illustrates the graphical comparison of the hardness profiles of the specimens. And, in Figure 59, the average hardness across the through thickness is plotted against the percent cold reduction.

Table 28: Summary of Vickers Hardness

Through Thickness (mm)	Vickers Hardness, HV30							
	Q032 (0%)	R592 (5%)	R585 (10%)	R591 (30%)	R583 (20%)	Q114 (35%)	Q016 (20%)	Q021 (40%)
1	208	189	224	341	396	377	316	407
2	220	187	225	337	373	382	303	414
3	224	187	224	339	350	390	290	407
4	222	186	222	347	318	400	294	396
5	224	183	221	339	310	400	294	398
6	223			339	323	405	302	389
7	225			346	319	415	309	392
8	231			351	307	429	318	391
9	223			351	320	428	320	404
10	222			349	337	432	318	409
11					345	438	325	397
Mean HV 30	222	186	223	344	336	409	308	400

Standard deviation	5.770	2.191	1.643	5.466	27.931	21.114	12.145	8.274
95% confidence	4.109	2.720	2.040	3.893	18.763	14.184	8.158	5.558
Relative Accuracy,%	1.851	1.462	0.915	1.132	5.584	3.468	2.649	1.390

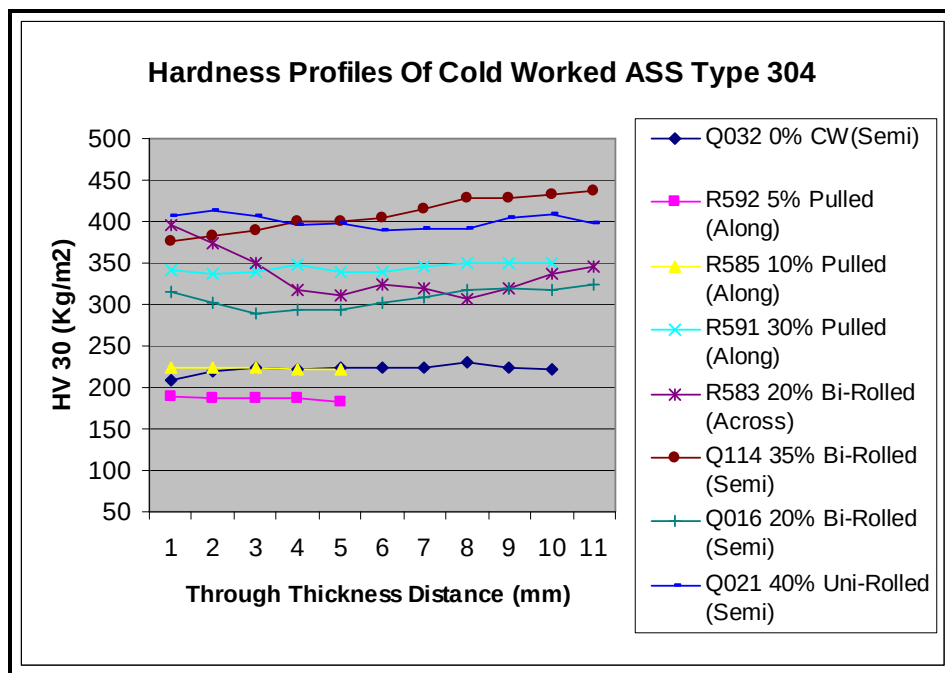


Figure 58: Vickers Hardness Profiles

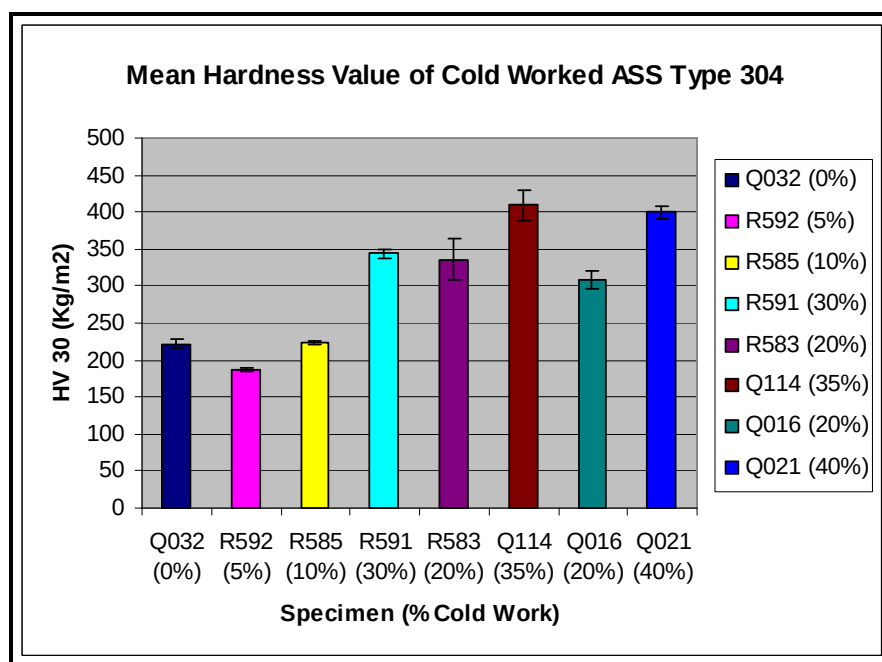


Figure 59: Mean Vickers Hardness

3.5.2 Strain Induced Martensite

The summary result of SIM by XRD is shown Table 29. The plot of percent martensite in all the specimens is shown in Figure 60 and the volume fractions of austenite and martensite in the specimens are illustrated with bar charts in Figure 61.

Table 29: Summary of Strain Induced Martensite

Samples	CW (%)	Integrated Intensity, <i>I</i>							<i>V_γ</i>	Martensite (%)
		γ -200	α -200	γ -220	α -211	γ -311	γ -222	α -220		
Q032	0	23832	143	15156	190	15621	4094	166	0.9855	1
R592	5	11165	0	15816	0	8190	2021	0	1	0
R585	10	23396	0	3714	0	9100	4157	0	1	0
R591	30	24160	300	795	509	4143	7978	258	0.9594	4
R583	20	3139	601	2830	441	3286	907	165	0.8304	17
Q114	35	1985	918	2514	1204	2515	1186	399	0.6938	31
Q016	20	8090	307	2657	256	4760	2661	95	0.9435	6
Q021	40	4414	644	1412	728	2960	1940	451	0.792	21

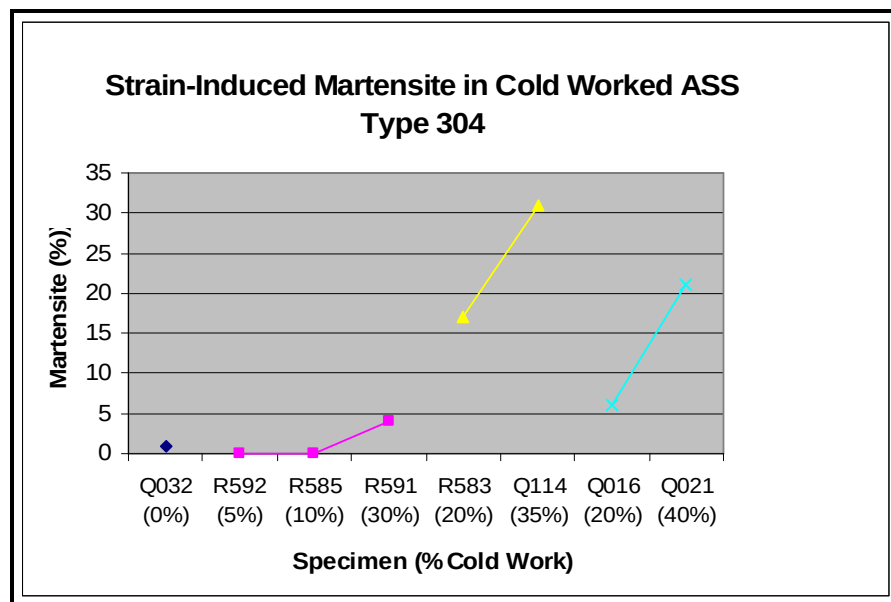


Figure 60: Summary of Strain Induced Martensite

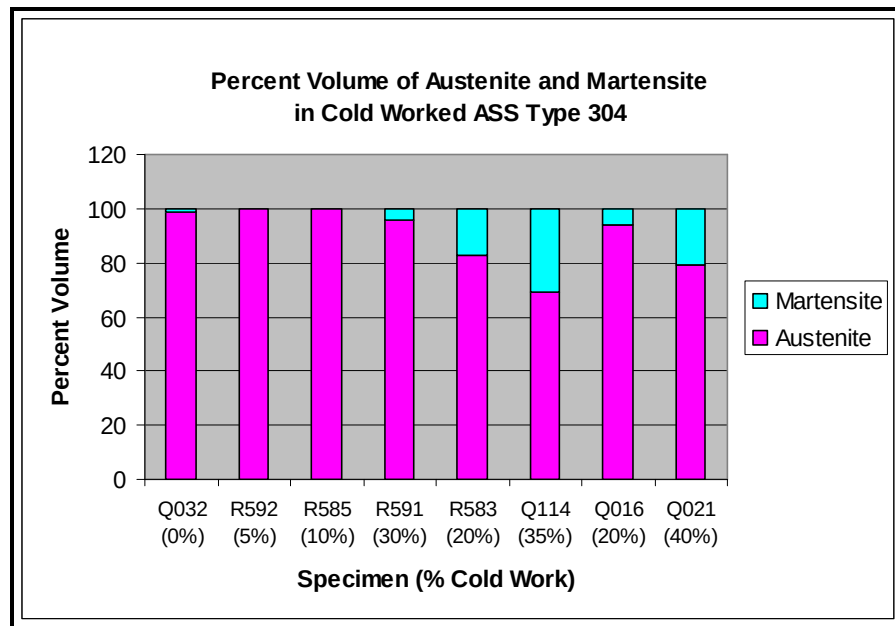


Figure 61: Volume Fraction of Austenite and Martensite

3.5.3 Residual Stress

The summary of longitudinal residual stress profiles in the entire specimens is shown in Figure 62. The profiles were obtained by plotting residual stress against regular interval on the through thickness, marked as points.

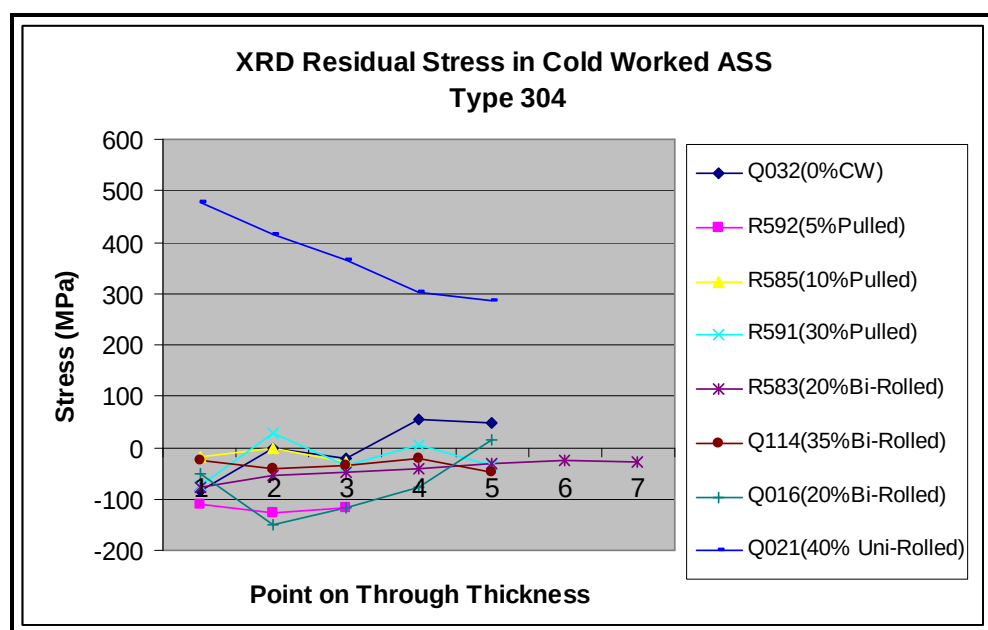


Figure 62: Summary of Residual Stress

3.5.4 Grain Size and Elongation Ratio

The grain size and grain elongation determined from oxalic acid micrographs are summarised in Table 30. Figures 63 and 64 showed the plot of average grain size and mean elongation ratio against the cold worked specimens respectively.

Table 30: Summary of Grain Size and Elongation Ratio

Specimen (%CW)	Grain Size μm				Elongation Ratio			
	T-L plane	S-L plane	S-T plane	Average Grain Size	T-L plane	S-L plane	S-T plane	Mean ER
Q 032 (0%)	31	34	31	32	1.08	1.38	1.26	1.24
R 592 (5%)	32	41	36	36	1.63	1.41	1.22	1.42
R 585 (10%)	27	30	29	29	1.14	1.44	1.04	1.21
R 591 (30%)	30	34	33	32	2.06	1.61	1.63	1.77
R 583 (20%)	53	35	35	40	1.42	1.77	1.53	1.57
Q 114 (35%)	85	26	31	41	1.29	1.53	3.27	2.03
Q 016 (20%)	31	35	29	32	1.38	1.83	1.26	1.49
Q 021 (40%)	33	31	30	31	1.54	2	2.13	1.89

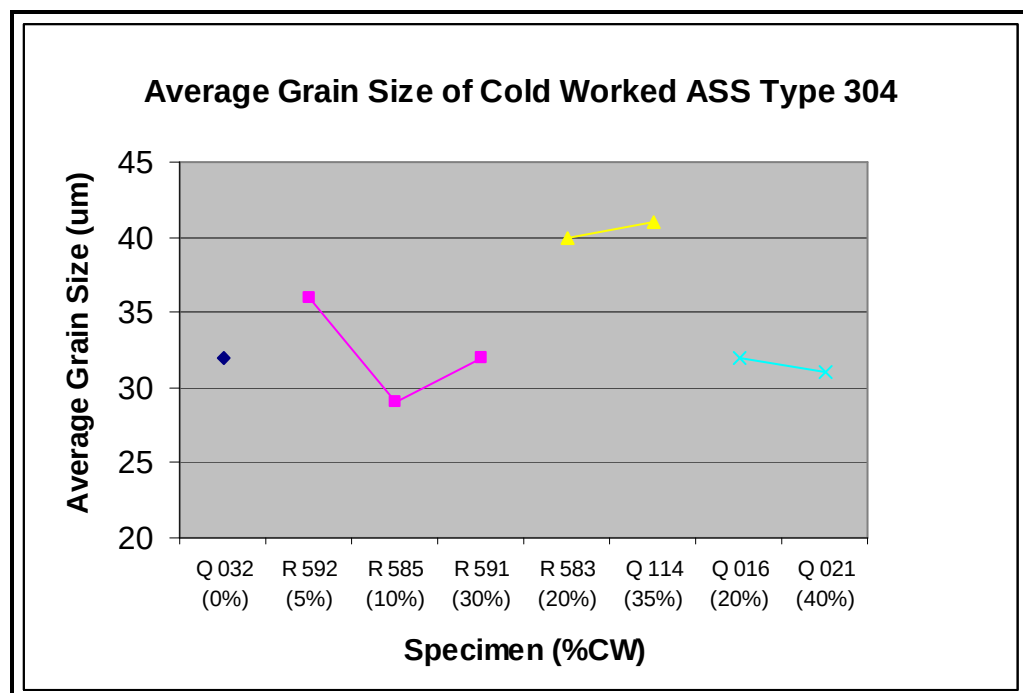


Figure 63: Summary of the Grain Size

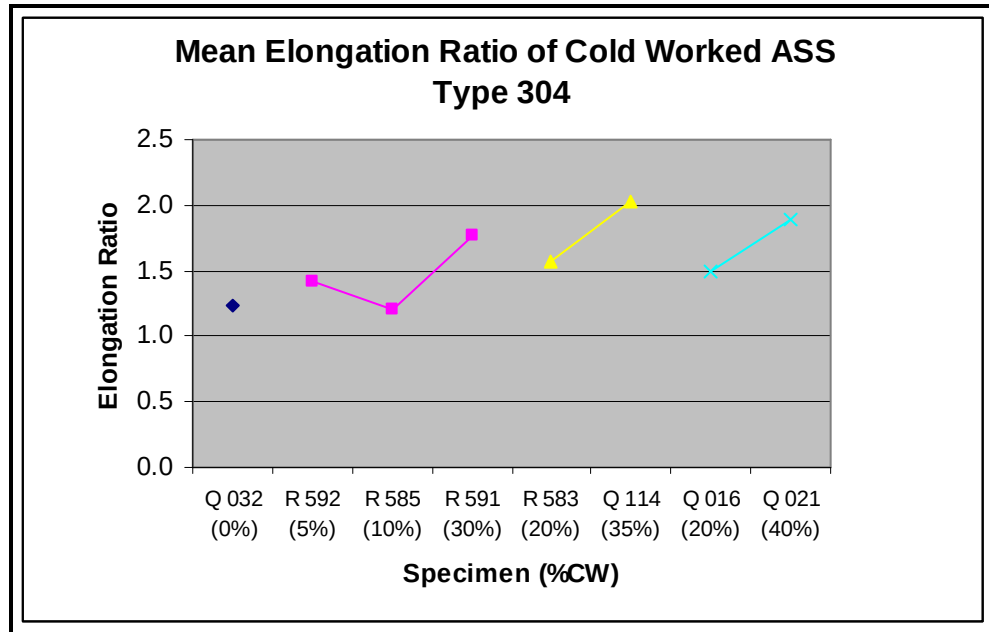


Figure 64: Summary of Elongation Ratio

3.5.5 Pole Figures

Table 31 shows the summary of pole figures results obtained from the test specimens. The maximum value of Multiples of Uniform Density (MUD) obtained from S-L and S-T planes pole figures are tabulated against the cold worked specimen. Table 31 allows comparison of MUD obtained from S-L and S-T planes of each specimen at a glance.

Table 31: Summary Results of Pole Figures MUD

Specimen (%CW)	Max. MUD Number from Pole Figures	
	S-L Plane	S-T Plane
Q032 (0%)	3.35	2
R592 (5%)	2.99	2.23
R585 (10%)	3.62	2.94
R591 (30%)	3.57	2.73
R583 (20%)	3.83	5.49
Q114 (35%)	No Indexing	No Indexing
Q016 (20%)	2.56	2.86
Q021 (40%)	5.54	3.78

4 Discussions

Vickers hardness is significantly influenced by material composition, deformation mode and the level of cold work. For example, the influence of carbon composition on the Vickers hardness is apparent by comparing the mean hardness results for specimens Q032 (Table 28) considered to be 0% cold worked and R595 cold worked to 5% reduction. The higher carbon content in specimen Q032, taken from Heat G (Table 1), yields a higher hardness value than that observed in specimen R595 taken from the lower carbon content material Heat D (Table 1). Indeed, the hardness observed in Q032 is similar to that observed in R585 (also from Heat D) which had received 10% cold work. This influence of carbon on the Vickers hardness is also observed in the other test material. For example, the mean hardness values for specimens R583 (Heat B material containing 0.043 wt% carbon) and Q016 (Heat C material containing 0.026 wt% carbon) are 336HV30 and 308HV30, respectively, even though both materials are cross-rolled to a 20% cold-work.

Another important observation on Vickers hardness, were the influence of deformation mode and degree of cold work. The results of Vickers hardness from specimens R583 and Q016 both of which were bi-directionally rolled to 20% reduction were relatively close to the hardness obtained for R591 which was cold-worked to 30% by pulling. As expected, it was observed that hardness increases with degree of cold reduction in specimens made from the same material. The main reason for these observed effects will be related to the development of dislocation structures during cold deformation. For example, it is likely that rolling in two directions will increase the dislocation density and entanglement more than uniaxial pulling and uni-directional rolling, since cross slip will be promoted under cross-rolling conditions. Areas on the specimen with higher local plastic strain (specimens R583 and Q114 in Figure 58) such as those immediately adjacent to the rolls are found to have high hardness values. This is consistent with the report of Cigada *et al.* [9] which suggested that, high cold reduction promotes rise in local dislocation density, deformation bands formation and appearance of SIM platelets at the intersection of deformation bands.

The amount of SIM showed a consistent increase with carbon content, deformation mode and level of cold reduction in a similar trend to that observed in the Vickers hardness results for all test materials. The results of SIM quantified by XRD were consistent with the level of martensite found in picral micrographs. The percent martensite also appeared to correlate with the degree of elongation of the grains in the rolling and transverse directions. This is evidenced by the observation that bi-directional rolling produces relatively higher amount of SIM than uni-directionally rolled and pulled specimens [10]. This is supported by the XRD results of Heat Band Heat C materials. In the case of Heat D materials, there was no martensite found in specimens R592 (5% pulled) and R585 (10% pulled) and only 4% martensite was found in specimen R591 with 30% cold work by pulling. On the other hand, 17% of martensite was found in R583 (with 20% CW by bi-directional rolling) and this is substantially higher than 4% in R591 despite its high carbon content. SIM nucleates at the intersection of deformation bands, it is reasonable to suggest that deformation process that produce a higher number of intersecting deformation bands will probably have the highest amount of SIM without the influence of other factors like chemical composition.

The optical metallography revealed distinct microstructural features on the 3 principal planes (T-L, S-L and S-T). The grain structures in the pulled specimens (R592, R585 and R591) appeared “sandwich-like” with little elongation physically visible. The only difference in the case of the uni-directionally rolled specimen Q021 was that, its grains showed more elongation because of a higher degree of cold work than that generated in the pulled specimens. But in the case of bi-directionally rolled specimens (specimens R583, Q114 and Q016), the grain structure on T-L planes maintained a distinct feature that appeared “pancake-like” because of elongations in the two orthogonal directions, i.e. the L- and T-directions. The grain structures observed in the S-L and S-T planes were not different from the earlier description for the specimen Q021.

The deformation mode also affects the structural features and distribution of delta ferrite within the austenite matrix. The geometrical morphology of delta ferrite in T-L planes of bi-directionally rolled specimens was found to be flaky and evenly distributed rather than stringer-like elongations which were often segregated on S-L and S-T planes. The even distribution of delta ferrite in austenite matrix is expected to be beneficial to chloride SCC resistance of stainless steels because of widely held view that, delta ferrite has the potential to slow down crack growth rate if present in adequate amount [11, 12].

Another important observation was the volume fraction of delta ferrite found in the test materials. Heat G material showed small stringers of ferrite. No delta ferrite was found in Heat D materials. Conversely, a substantial amount of ferrite was found in Heat B and Heat C materials. Heat C showed the highest volume fraction of delta ferrite amongst the test materials. The proportion of delta ferrite is not unexpected because there appears to be a strong link between the volume fraction of ferrite and the chemical composition of the materials. The amount of delta ferrite is strongly determined by the competing power of ferrite stabiliser, predominantly silicon and austenite stabilising elements such as carbon and manganese [12]. A closer look at Table 1 shows that Heat C material has significantly high Si content (ferrite stabiliser) and the lowest amount of C and Mn (austenite stabiliser). On the other hand, Heat D material without any ferrite contained high amount of C and Mn and lowest amount Si in its chemical composition.

Residual stresses in all the test specimens were mainly compressive except in specimen Q021 which exhibited a tensile residual stress. Whilst there was considerable scatter in residual stress results, the common trend was that, the magnitude of compressive residual stress decreases with increasing cold work, i.e. the residual stress magnitude shifts towards the tensile direction as the level of cold reduction increases (Figure 62). The reason for this behaviour is not fully understood, however, it is reasonable to suggest that the decrease in compressive residual stress with increasing cold work may be linked to the development of a more uniform plastic strain field through the thickness of the specimen, since residual stresses are generated by local mismatches in elastic and plastic strain level. A local (microstructural) effect, not measured in these X-ray diffraction results, will also arise from the austenite-martensite transformation and the presence of delta ferrite in some of the materials. The geometrical morphology of SIM laths is characterised with the potential to absorb large strain as a result of shape effect. Similarly, Reports show that coefficient of thermal expansivity of ferrite ($\approx 1.3 \times 10^{-6} \text{ K}^{-1}$) is almost half of that of austenite ($\approx 2.1 \times 10^{-6} \text{ K}^{-1}$) in lean steels [13]. This suggests that a local tensile residual (mismatch) stress may be present in the austenite material lying adjacent to the delta ferrite phase, which would be under compression. This local (Type 3) residual stress would be generated as a result of the manufacturing process [14].

One issue to note is that the XRD measurements do not account for the Type 2 intergranular residual stresses which may be expected to be significant in heavily cold-worked materials [14]. It may be worthwhile to consider another qualitative residual stress measurement technique that can quantify Type 2 residual stresses.

Finally, deformation texture has been shown to influence SCC susceptibility of stainless steels as described by Raquet *et al.* [15]. In their report, the reduced SCC susceptibility of heavily cold worked materials subjected to Constant Extension Rate Test (CERT) in primary PWR water was linked to deformation texture induced by a tensile stress prior to the SCC test.

5 Conclusions

The following are the conclusions drawn from the characterisation of cold worked austenitic stainless steels Type 304:

1. The Vickers hardness and the amount of strain induced martensite (SIM) increased with carbon content and degree of cold work
2. Higher hardness values and greater amount of SIM were found in specimens subjected to bi-directional rolling than in specimens cold worked by uni-directional rolling and pulling.
3. Bi-directional rolling causes the grains to be stretched in both directions leading to a pancake-like structure on the T-L planes. In the case of pulling and unidirectional rolling, the grains are elongated and appeared rather sandwich-like.
4. The volume fraction of delta ferrite was significantly influenced by the balanced between the ferrite stabiliser (mainly Si) and austenite stabilisers (combined effects of C and Mn) in the chemical composition.
5. The macro residual stresses measured were predominantly compressive in most of test materials but magnitude of the compressive stress reduces with increasing cold work.
6. Deformation texture increases with increasing levels of cold work.

6 Recommendations

The following recommendations are made at the end of the report.

1. Transmission Electron Microscopy (TEM) should be explored to investigate the dislocation density in the cold worked specimens and
2. Alternative qualitative method of residual stress measurement that will incorporate the significant contribution of intergranular residual stress should be considered.

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