Chapter 10 Experimental procedure for determining the AlN solubility in low carbon Al-killed steel using TEP measurements

_____

10.1   Introduction

Low carbon strip steels with different amounts of sulphur were received from Mittal Steel (South Africa), with high and low sulphur contents. The steel alloys were received in the as-cast condition and the chemical compositions are given in table 10.1 below.

The alloys were hot rolled on a laboratory rolling mill at Mintek into 10 mm thick plates and the schematic diagram for the rolling process is given in figure 10.1 below. The finishing temperature for the last pass was checked by pyrometer and for all the alloys was above 900 °C.

10.2   Sample preparation for TEP measurements

Although TEP measurements are not sensitive to specimen size, these were prepared in two sets of strips; the first set was 90 mm long, 4 mm wide and 1 mm thick and the second set was 20 mm long, 4 mm wide and 1 mm thick. The latter set was from material on which the hot deformation and coiling treatment was simulated on a Gleeble 1500^{TM} hot compression facility. In general, thin specimens were preferred for TEP measurements because of the reduced heat transfer path between the cold and hot junctions which aided thermal equilibrium conditions on these junctions.

Chapter 10   Experimental   procedure   for
            determining the AlN solubility in low
   carbon Al-killed steel using TEP
            measurements

_____

Table 10.1: Chemical compositions of the low carbon strip steels that were studied: HS = high sulphur and LS = medium to low sulphur, the first numeral is for the sulphur content and the second for the nitrogen content, both in ppm.

Element (ppm)	HS130 -50	HS90- 34	HS90- 12	HS140 -104	LS70- 38	LS10- 83	LS2-65	LS16- 101
C	320	280	270	510	580	510	380	590
Mn	2300	1870	1720	2240	2560	3140	2500	3600
P	130	60	90	80	210	70	50	76
S	130	90	90	140	70	10	2	16
Si	160	60	50	140	210	330	200	140
Cu	257	140	130	260	30	30	90	-
Ni	175	100	20	110	60	50	10	-
Cr	308	130	200	270	110	110	80	98
Mo	20	20	30	10	0	10	10	33
V	20	10	10	20	60	70	70	80
Nb	30	10	10	10	10	10	20	55
Ti	7	20	20	20	20	20	10	-
Al_{total}	480	430	390	500	200	340	450	240
$Al_{acid\ sol}$	458	379		470		333	370	-
Ca	2	0	0	1	14	26	20	9
N	50	34	12	104	38	83	65	101

_____

Experimental procedure for determining the AlN solubility in low carbon Al-killed steel using TEP measurements

_____
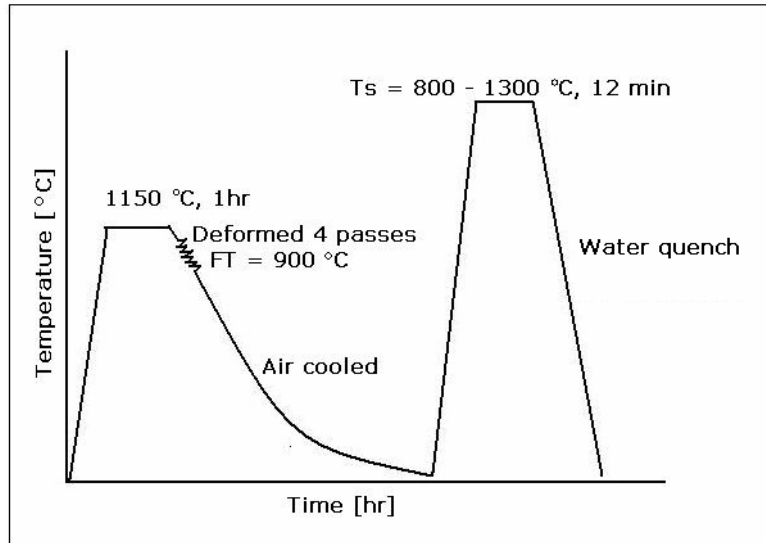
Figure 10.1: Schematic diagram for the hot rolling schedule and the solution heat treatment in the dilatometer and furnace.

10.3   TEP solution treatment

The solution heat treatment (mainly for the dissolution of the AlN) was done under argon gas in a vertical tube furnace and the temperature range was varied from 800 to 1300 °C, figure 10.1. The specimens were suspended in the furnace as shown in figure 10.2 and were soaked for 12 minutes at the test temperature to ensure that equilibrium dissolution conditions of the AlN was attained before quenching into water.
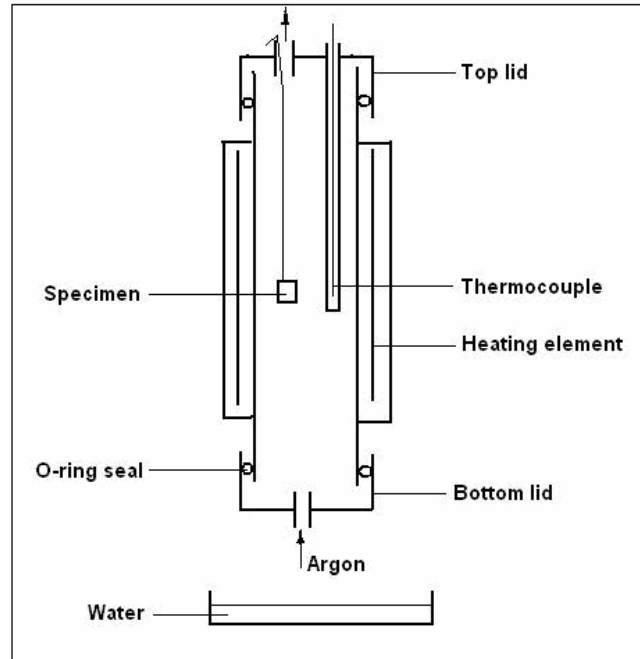
_____

_____

Figure 10.2: The furnace heat treatment arrangement.

10.4 TEP   measurements   in   as-quenched condition

The specimens were clamped to the copper blocks and the TEP measurements were recorded after 1 minute. TEP measurements were immediately taken after the samples had been quenched from the solution treatment furnace in order to avoid strain aging by nitrogen. The furnace and the TEP equipment were, therefore, arranged close to each other.

The photograph and schematic diagram for the TEP measurement arrangement are given in figures 10.3 and 10.4 respectively. The

_____

_____

two copper blocks were kept at a temperature difference of 15 ºC in order to maintain a temperature gradient ΔT between the cold and hot junctions of the specimen. For this, one block was filled with ice and the temperature at the cold junction was maintained at 5° ± 1°C while the other one at the hot junction was filled with water at room temperature at 20° ± 1°C. K-type thermocouples were spot welded onto the hot and cold junctions of the specimen.

A data logger was used to log both the temperature data and the voltage drop $\triangle V$ across the hot and cold junctions (as shown schematically in figure 10.4) into different analog channels of the Data-Taker dt800 which amplified and converted this data into digital data that was recorded using Delogger software. The voltage drop $\triangle V$ was measured from the Chromel wires from the cold and hot junctions. The advantage with the arrangement in figure 10.4 was that the instantaneous voltage difference $\triangle V$ was measured together with the corresponding temperature difference $\triangle T$ between the cold and the hot junctions of the sample out of which the instantaneous   absolute TEP measurement was obtained.
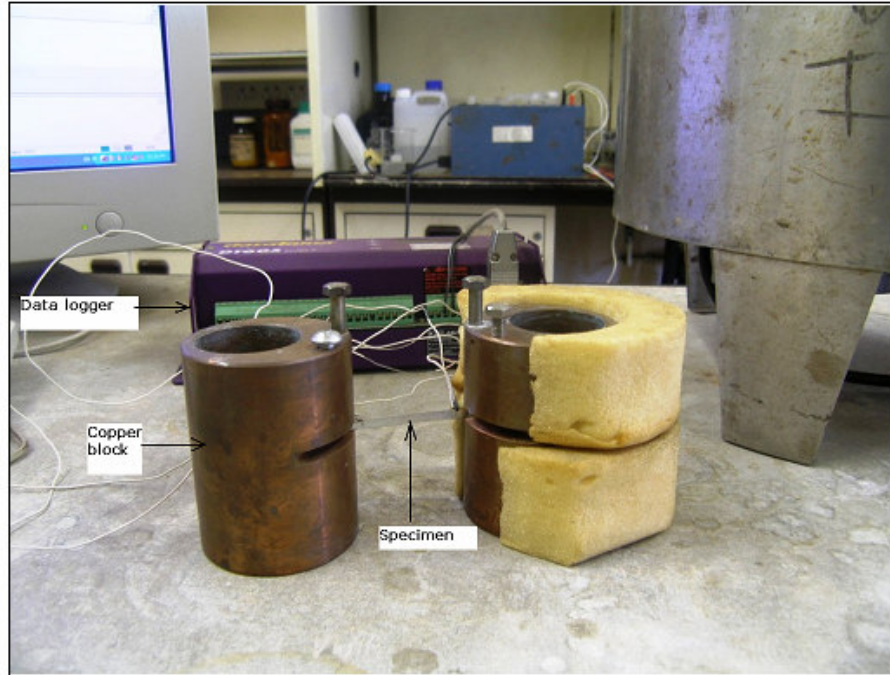
_____

Figure 10.3: Photograph of the TEP measurement arrangement.
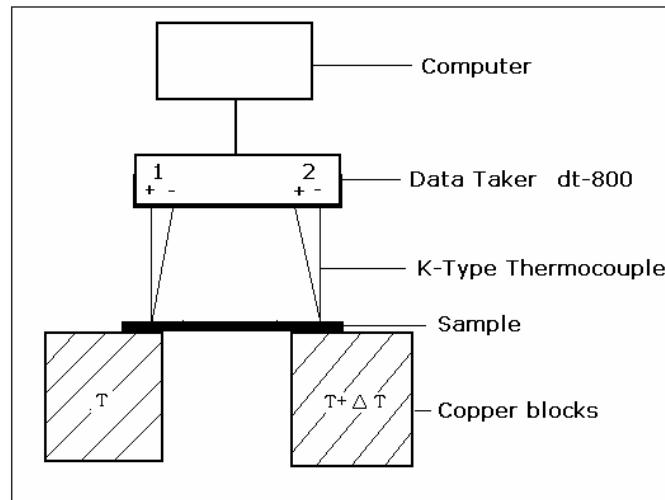
Figure 10.4: Schematic diagram for the TEP measurements.

_____

10.5 TEP measurements after hot rolling and coiling simulations on the Gleeble 1500^{TM}

TEP measurements were conducted on the specimens after hot rolling and coiling simulated in the Gleeble 1500^{TM} according to the rolling schedule and heat treatment cycle specified in table 11.1 and figure 11.1 below. The objective was to assess whether the AlN had partially or fully precipitated during coiling. These measurements were carried out on the specimens that were simulated in their hot rolling and coiling at 600 °C as this lower coiling temperature was more sensitive to the precipitation of AlN.

The heat treatment cycle prior to the TEP measurements is given in figure 10.5 below. The first set of the absolute TEP measurements, i.e. TEP1, measured the trend of nitrogen in solid solution immediately after coiling while the second one (TEP2) checked whether further precipitation of AlN took place after coiling or not. If there is no significant variation in the TEP values between the first and second set that would imply complete precipitation of AlN after coiling. Further precipitation of AlN after coiling, that is, during the second heat treatment of isothermal annealing at 800 °C for 2 hours, would lead to further depletion of nitrogen from solid solution which would lead to higher measured TEP2 values and from this observation it would be possible to establish whether the AlN had partially or fully precipitated during the coiling process.

_____

Chapter 10 Experimental procedure for determining the AlN solubility in low carbon Al-killed steel using TEP measurements
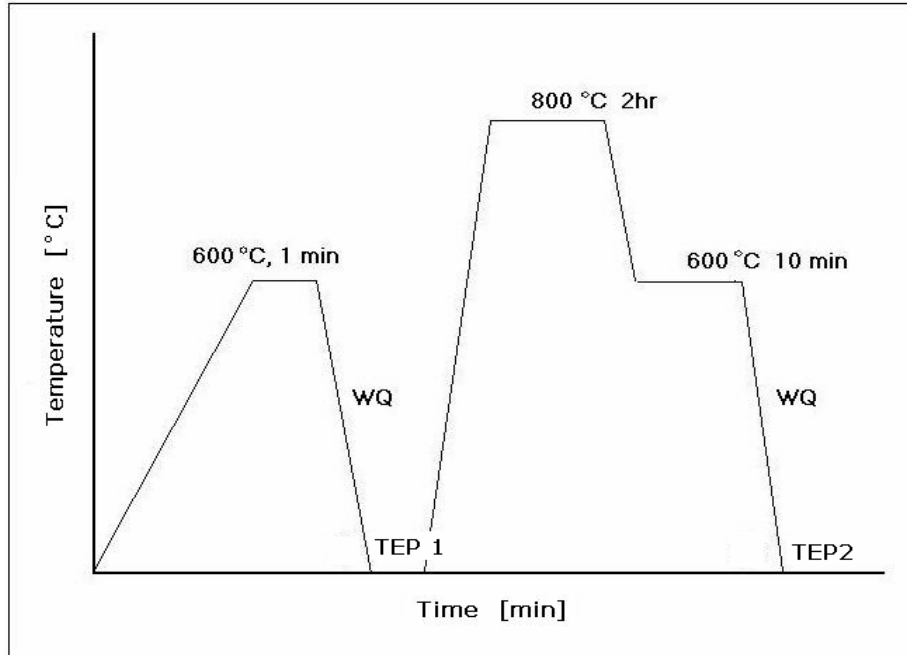
Figure 10.5: Heat treatment cycles for TEP measurement after coiling simulation on the Gleeble 1500^{TM}, TEP 1 is TEP measurement after the first heat treatment cycle while TEP 2 is after the second one.

In the first heat treatment cycle in which TEP measurements TEP1 were carried out, the specimens were annealed at 600 °C for 1 minute and quenched into water. The purpose for this heat treatment was to ensure that all specimens had the same amount of carbon in solid solution during the TEP measurements and these measurements were carried out immediately after quenching into water in order to avoid strain aging by nitrogen. In the second set of TEP2 measurements, the same specimens were soaked at 800 °C for 2 hours to precipitate any remaining nitrogen into AlN and, thereafter, the specimens were cooled to

Chapter 10   Experimental   procedure   for
              determining the AlN solubility in low
              carbon Al-killed steel using TEP
              measurements

_____

600 °C, annealed for 10 minutes and then quenched into water. The purpose for holding the sample at 600 °C for 10 minutes was to ensure that the same amount of carbon was in solid solution before the TEP measurements were taken.

10.6   TEP methodology for the determination of the dissolution of AlN during reheating

Figure 10.6 shows a schematic illustration of the heat treatment cycles and TEP measurement sequences for the study of the dissolution of AlN during reheating of the hot rolled Al-killed low carbon steel. In the as-hot-rolled condition, the AlN is either fully or partially precipitated and the expected absolute TEP value would be S_o. The volume fraction of the precipitated AlN in austenite and ferrite X_v, which is dependent on the cooling rate, is estimated by[48]:

$$X_v = K\phi^m \qquad\qquad (10.1)$$

where   K is a constant dependent on the initial nitrogen and aluminium levels, the grain size and the transformation behaviour of the steel; ϕ is the cooling rate (K h^{-1}) and m is the cooling rate exponent (for C-Mn steel, Honer et al[48] found that K = 10.8 and m = -0.49).

All the specimens were first (before solution treatment) annealed at 800 °C for six hours in order to ensure that the AlN precipitated

_____

Chapter 10   Experimental   procedure   for
determining the AlN solubility in low
carbon Al-killed steel using TEP
measurements

into incoherent particles because the strain field around any undissolved coherent particles would contribute to the TEP effect, and would thereby, have affected its measurement.

The annealing temperature of 800 °C was chosen because it is close to the TTT nose for the precipitation of AlN in austenite, see figure 4.2. The absolute TEP measurement at this temperature S_{800}, became the reference for "no dissolution". The TEP measurements were conducted near or at room temperature after the steel had fully transformed to ferrite.

The samples were then soaked at various test temperatures and quenched into water, after which the absolute TEP measurements S_i were recorded. Assuming the diffusion component of the TEP was predominant at room temperature and as the concentrations of both the aluminium and nitrogen were low (< 0.1 percent), the Gorter-Nordheim rule could be used with confidence. Therefore, for a stoichiometric composition of the AlN, the relative TEP value $\triangle S_N$ could be obtained through the linear relationship:

$$\Delta S_N = S_{800} - S_i = K_{AlN}[N] \qquad (10.2)$$

where K_{AlN} is the aluminium nitride TEP coefficient and N is the nitrogen content (wt percent) in solid solution at the test temperature.

Chapter 10   Experimental   procedure   for determining the AlN solubility in low carbon Al-killed steel using TEP measurements

It is important to point out that the TEP coefficient K_{AlN} includes the specific influence on the TEP measurement of both aluminium (K_{Al}) and nitrogen (K_N) leaving the solid solution during the precipitation of AlN [144].
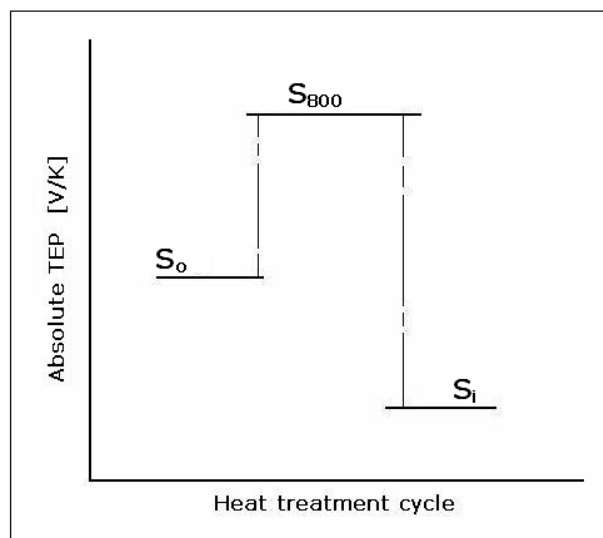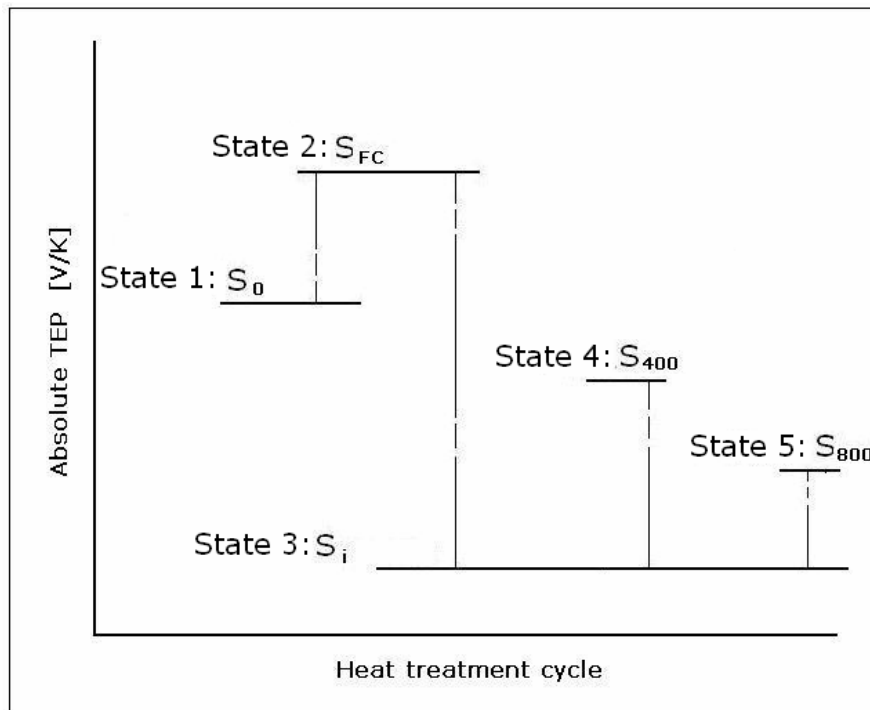

Figure 10.6: Schematic diagram of the sequence of heat treatment cycles and TEP measurements for determining the dissolution of AlN during reheating of Al-killed low carbon steel.

10.7   Checking   the   validity   of   the   AlN equilibrium solubility trend in section 10.6

The trend of the nitrogen in solid solution determined by the schedule in figure 10.6 above was checked by the one in figure 10.7 below and the results were in good agreement as the relative TEP values tallied well with the nitrogen and carbon contents in

![University of Pretoria logo] UNIVERSITEIT VAN PRETORIA
UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

Chapter 10   Experimental   procedure   for
              determining the AlN solubility in low
              carbon Al-killed steel using TEP
              measurements

solid solution, see figure 10.8 below. The TEP coefficient for the dissolved nitrogen K_{AlN} was found to be 10 nV/K.ppm-N which is within the range observed by other authors[148].

Figure 10.7: Schematic diagram of the sequence of heat treatment cycles and TEP measurements for determining the carbon and the nitrogen content in solid solution.

10.7.1   State 1, 2 and 3 of the TEP schedule

In figure 10.7, the absolute TEP measurement S_o represented the initial condition of the steel thus as-received condition which, generally, is as-hot rolled condition. The specimens were then soaked at 1150 °C for 12 minutes and furnace cooled to room

Chapter 10   Experimental   procedure   for
                determining the AlN solubility in low
                carbon Al-killed steel using TEP
                measurements

temperature to fully precipitate both the cementite (Fe_3C) and AlN and the corresponding absolute TEP measurement was designated S_{FC}. The specimens were then heated to the test temperature, (1300 °C in order to dissolve all the AlN), soaked for 12 minutes and thereafter quenched into water as schematically shown in figure 10.1. The absolute TEP measurement S_i represented the carbon and nitrogen that were in solid solution assuming negligible or constant TEP contributions from quenched-in defects and other alloying elements in solid solution.

10.7.2   State 4 and 5 of the TEP schedule

In order to separate the contribution of the carbon from that of nitrogen to the TEP measurements, one set of specimens was annealed at 400 ºC for 3 hours to precipitate the Fe_3C without precipitating the iron nitride (Fe_4N) as this temperature was above the solubility limit of the (Fe_4N) [144,145].

The equilibrium solubility of carbon in α-iron is given by[146]:

$$C_{C\text{-eq}}=6.63\exp\left[-\frac{49400}{RT}\right] \qquad (10.3)$$

where R is the universal gas constant which is 8.314 J mol^{-1} K^{-1} and T is the absolute temperature in K.

Therefore, at 400 ºC, the equilibrium solubility of carbon $C_{c\text{-}eq}$ would be 9.72 x 10^{-4} wt% (10 ppm). This implied that the contribution of the residual carbon to the TEP value would be negligible compared with the amount originally in solid solution that was between 250 and 500 ppm.

According to Fast et al[145], the solubility of nitrogen in equilibrium with Fe_4N in α-iron is given by:

$$C_{N\text{-}eq}=12.3\exp\left[-\frac{34700}{RT}\right] \tag{10.4}$$

Therefore, at 400 ºC, the solubility of nitrogen in equilibrium with Fe_4N would be 250 ppm. Since the maximum nitrogen content for all the steels that were studied was 104 ppm, no iron nitride was expected to form at 400 ºC. The absolute TEP measurement after annealing at 400 ºC is designated as S_{400}. The nitrogen solubility trend could then be correlated to:

$$\Delta S_N = \left(S_{FC} - S_i\right) - \left(S_{FC} - S_{400}\right) = K_N \bullet N_{ss} \tag{10.5}$$

where K_N is the TEP coefficient for the nitrogen in solid solution

In state 5, the specimens are annealed at 800 °C to precipitate the AlN while the carbon remains in solid solution and thereafter quenched in water. At this annealing temperature, AlN equilibrium

Chapter 10   Experimental   procedure   for
determining the AlN solubility in low
carbon Al-killed steel using TEP
measurements

_____

solubility models predict that less than 5 ppm nitrogen will be in solid solution[41,42]. The carbon content can then be correlated to:

$$\Delta S_C = \left(S_{FC} - S_i\right) - \left(S_{FC} - S_{800}\right) = K_C \bullet C_{ss} \qquad (10.6)$$

where K_C is the TEP coefficient for carbon in solid solution.

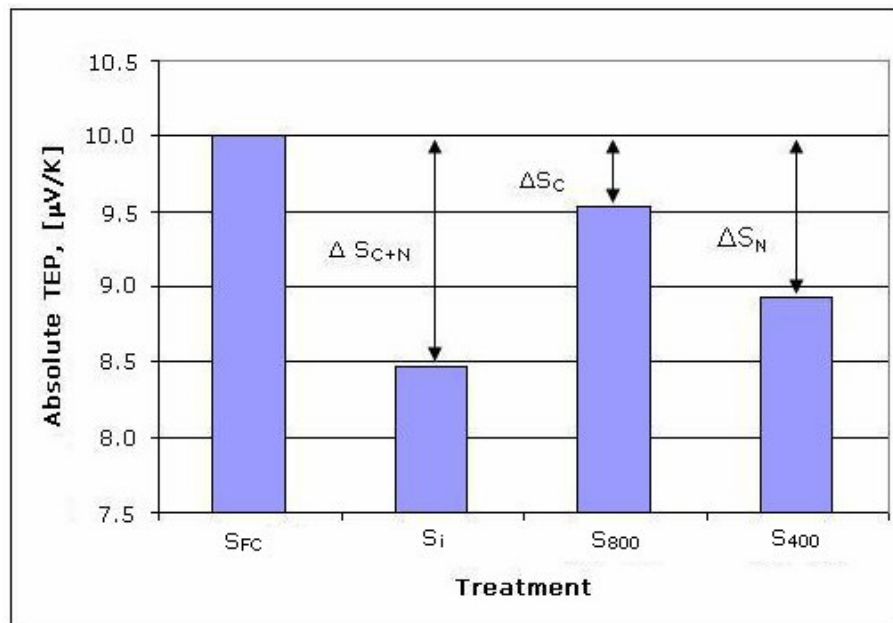Figure 10.8 shows typical results obtained from the TEP measurement sequence shown in figure 10.6.

Figure 10.8: Typical absolute TEP measurements for steel LS16-101 where ΔS_{C+N} represents carbon plus nitrogen, ΔS_C the carbon and ΔS_N the nitrogen in solid solution.

_____

Chapter 11 Experimental procedures for the study of the recrystallisation after cold work

11.1 Introduction

The static recrystallisation after cold work was studied in two stages. Firstly, the study was done on the as-cast steels, which were solution treated at 1300 °C and quenched into water. The purpose for this treatment was to dissolve the AlN, CuS/Cu$_2$S and MnS before cold working and annealing. Secondly, specimens from the as-cast steels were hot rolled and coiled by simulation on the Gleeble 1500^{TM} to follow the industrial conditions before cold working and annealing. In both cases, the primary objective was to investigate the influence of the sulphur and nitrogen content on the recrystallisation process.

11.2 Hot rolling simulation and cold working before isothermal annealing

Steels HS140-104, LS70-38 and LS2-65 whose chemical compositions are given in table 10.1, were hot rolled and coiled according to the hot rolling schedule given in table 11.1 and the heat treatment cycle given in figure 11.1 below. Both the hot rolling and the coiling were done in an argon atmosphere in order to avoid excessive oxidation.

As may be seen in table 11.1, the only variable was the coiling temperature. It was not possible to simulate the very high industrial strain rates on the Gleeble due to machine and friction limitations. The highest strain rate that could be achieved with

good repeatability was 2 s^{-1} which was significantly below the industrial strain rates in hot rolling that are as high as 150 s^{-1}.

Table 11.1: The hot rolling and coiling schedule simulating the industrial hot rolling and coiling processes on the Gleeble 1500^{TM}, the interpass time is before the rolling pass, RT = reheat temperature, FMH = finishing mill head and F = rolling pass.

Simulated CCR hot rolling on the Gleeble 1500^{TM}						
Sample size: length 15 mm; diameter 10 mm						
RT : 1150 ºC						
Parameter	RT	F1	F2	F3	F4	FMH
Initial height H in (mm)		15.00	11.11	7.83	5.80	4.75
Final height h out (mm)		11.11	7.83	5.80	4.75	4.75
Entry temperature (ºC)	1150	1100	1050	980	900	900
True strain per pass		-0.30	-0.35	-0.30	-0.20	
Cumulative true strain		-0.30	-0.65	-0.95	-1.15	
Cumulative Δh in (mm)		-3.89	-7.17	-9.20	-10.25	
Strain rate per pass (s^{-1})		-2.0	-2.0	-2.0	-2.0	
Interpass time (s)		30.00	2.00	2.00	2.00	
Coiling for 1hr (ºC)						650
Coiling for 1hr (ºC)						600
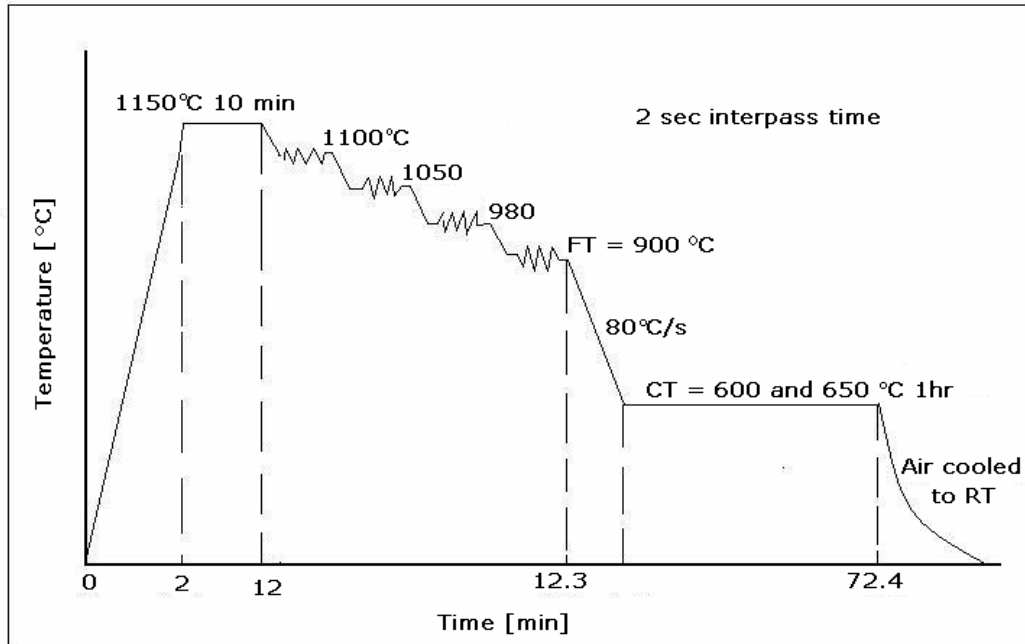
Figure 11.1: The schematic diagram for the simulated hot rolling and coiling processes on the Gleeble 1500™ hot rolling simulator.

After coiling, the specimens were cold worked using a laboratory cold rolling machine and the total reduction in thickness was 70 percent. The rolling was done in small reductions of less than 5 percent per pass in order to achieve a final uniformly deformed structure as large reductions had produced "cobbles" on the specimens, probably arising from the small diameter rolls on this machine.

Chapter 11 Experimental procedures for the study of the recrystallisation after cold work

11.3 Isothermal Annealing after cold work

The isothermal annealing was carried out in a lead bath that was pre-heated to the annealing temperature. The specimens were immersed for different times and temperatures depending on the experimental requirements. The arrangement for the lead bath is shown in figure 11.2.
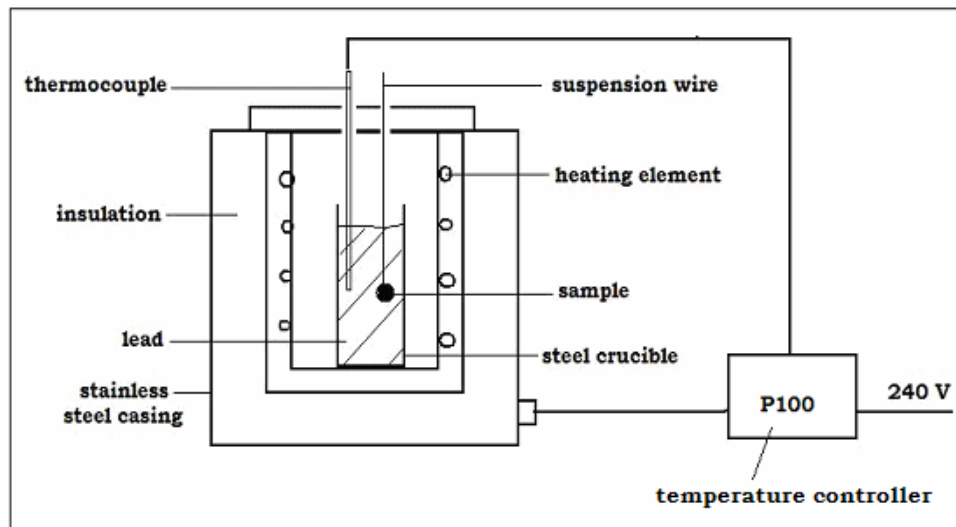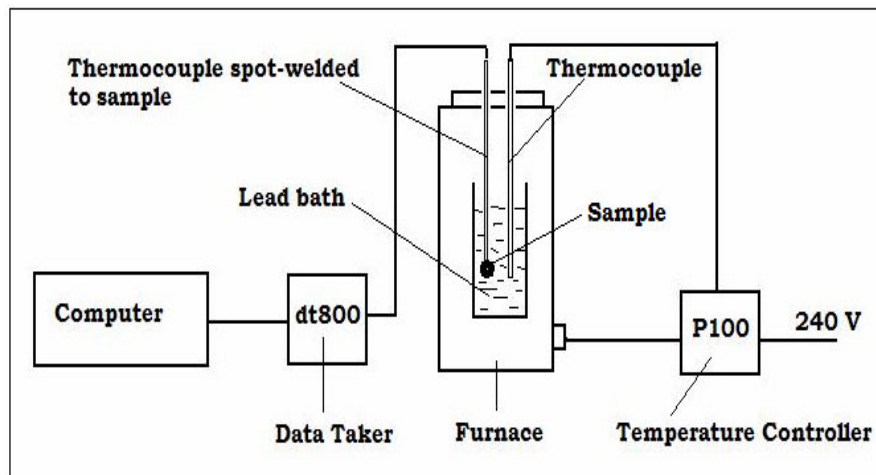

Figure 11.2: The experimental arrangement for isothermal annealing in the lead bath.

The specimen size was minimised to avoid undesirable temperature gradients with time between the surface and the interior of the sample, which was relevant at very short annealing times. The optimum specimen size was found to be 15 mm long, 4 mm wide and 1.5 mm thick. The steel crucible that contained the molten lead was 65 mm in diameter and 140 mm deep. The

volume of the molten lead was approximately 400 cm^{-3} and no temperature change was observed in the lead when immersing the 0.09 cm^{-3} specimen as the thermal mass of the specimen was negligible compared to that of the lead. In optimising the specimen size, it was observed that when larger specimens were immersed in the lead bath there was an immediate drop in temperature of up to 20 °C, depending on specimen size, and it took up to 200 seconds for the lead bath to be reheated to the programmed annealing temperature, in the process affecting the accuracy of the annealing temperature as well as the entire results. The experimental arrangement that was used to determine the heating rate of the 15 x 4 x 1.5 mm specimen in the lead bath is shown in figures 11.3 and 11.4.
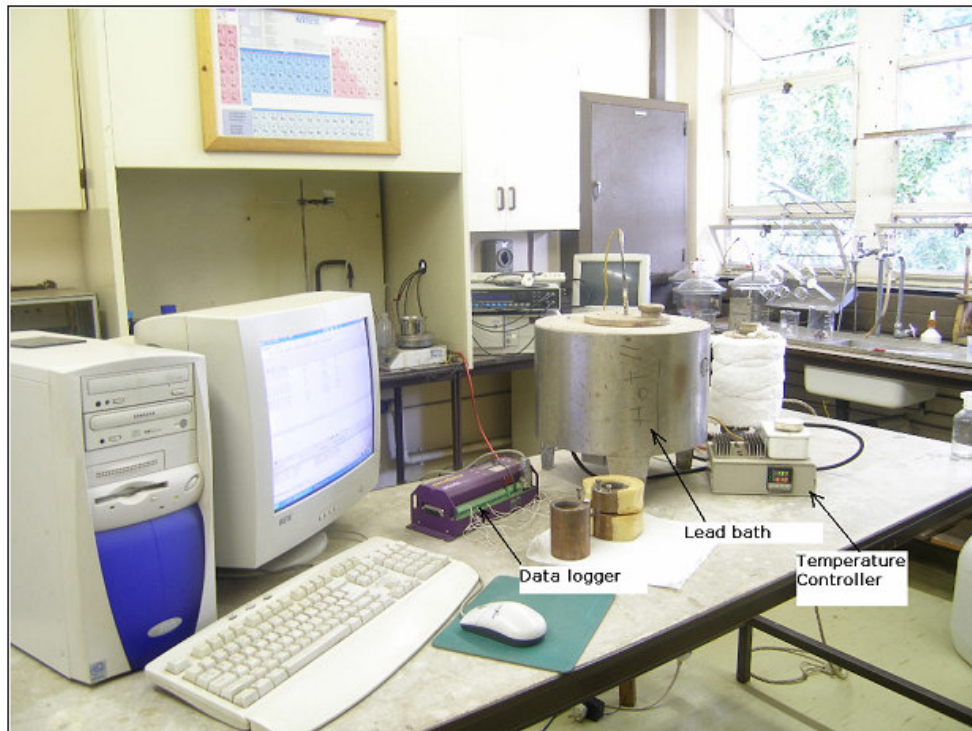

Figure 11.3: The schematic experimental arrangement for measuring the heating rate of the specimen at the start of isothermal annealing in a lead bath.

Chapter 11 Experimental procedures for the study of the recrystallisation after cold work

A 0.384 mm diameter R-Type (Pt/Pt-13%Rh) thermocouple was spot welded onto the sample to measure the heating rate upon immersion into the lead bath. The thermocouple was coated with a paste of alumina and insulated with a high temperature sheath. The data logger sampled the analogue voltage signal from the thermocouple at a frequency of 1 Hz. The data was downloaded and was analysed using Microsoft Excel, see figure 11.5.

Figure 11.4: Photograph of the experimental arrangement for measuring the heating rate of the specimen at the start of isothermal annealing.

As may be seen from figure 11.5, both the heating to the annealing temperature and the quenching to room temperature

were almost instantaneous when compared to the longer isothermal annealing times of more than 15 seconds i.e. the respective heating and cooling time of the specimens was negligible compared to the isothermal annealing time.
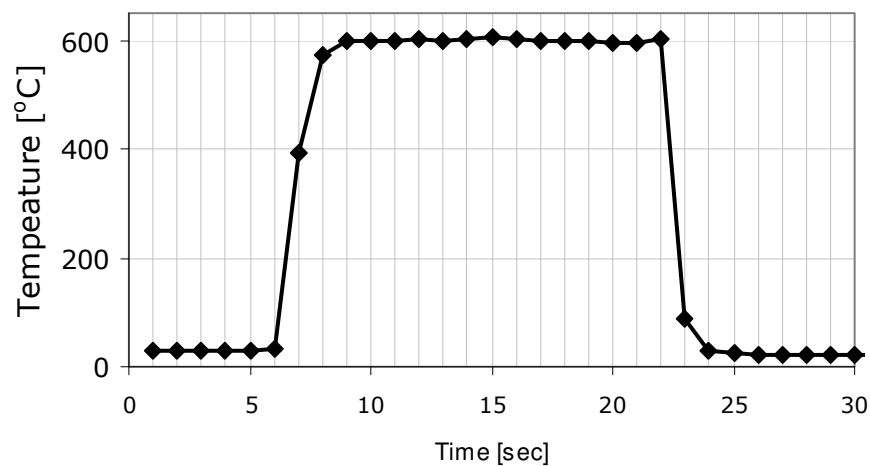

Figure 11.5: A typical heating rate curve for a 15 x 4 x 1.5 mm specimen at the start of isothermal annealing for steel HS140-104 in a lead bath followed by water quench.

11.4 Experimental   procedures   for   the metallographic analysis

Metallographic analysis of the microstructures was done for three reasons; namely to confirm the AlN equilibrium solubility trend for the TEP results, to study the percentage recrystallisation after cold work and to study the precipitation of AlN and MnS in these
 steels. Both optical and electron microscopy methods were applied in this study.

11.4.1   Preparation of the carbon extraction replicas for the observation of MnS and AlN particles

The samples were mounted in epoxy, ground to 3 μm and etched in 4% Nital. The carbon was then deposited on these samples using a carbon coating unit (Speedivac Coating Unit 12E6/1419) under a vacuum pressure of 10^{-4} Torr. Squares of approximately 3 x 3 mm^2 were scribed on the carbon coated surface using a surgical knife and removed by electrolytic etching of the substrate using an electrolytic solution of 10 percent nitric acid and 90 percent ethanol at a potential of 10 volts and a current density of about 0.2 A cm^{-2}. Higher voltages were avoided because faster separation of the extraction replica from the substrate resulted in broken carbon replicas. The replicas generally floated off after about 4 to 10 minutes etching times.

Alternatively, the samples were floated off by chemical etching in the same 10 percent Nital. In these instances, the floating off took a bit longer, 10 to 60 minutes depending on the microstructure and depth of etching. After floating off, the replicas were stripped in distilled water and dried on filter paper.

The carbon extraction replicas were observed by transmission electron microscopy (TEM) on a Phillips CM200, which uses a tungsten filament and was operated at 160 kV. The particles were
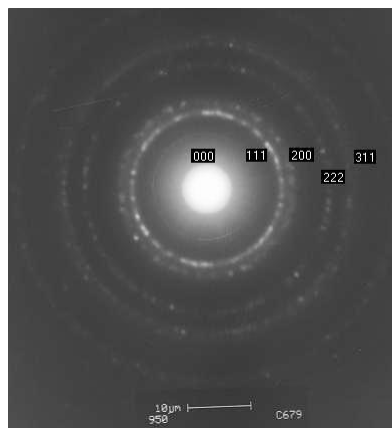
Chapter 11   Experimental   procedures   for   the
study   of   the   recrystallisation   after
cold work

identified by electron dispersive x-ray spectroscopy (EDS) and X-ray diffraction patterns.

All diffraction work was carried out at the same TEM settings which were as follows: the accelerating voltage was 160 kV, selected area diffraction (SAD) aperture number 1, the condenser lens current C2 (intensity) was set at 1600 mA, the camera length was 950 mm, the exposure time was 40 seconds and the diffraction lens current was 1.170 mA.

The TEM's camera constant was calibrated with a gold film that was sputtered onto a carbon film. Figure 11.6 shows the diffraction pattern for the gold layer that was used to determine the camera constant.

Figure 11.6: The diffraction pattern for the polycrystalline gold film that was used to calibrate the camera constant for the Phillips CM200 TEM.

Using the lattice parameter for gold of 0.40788 nm, the camera constant was determined from the following equation which is derived from the Bragg equation:

$$\lambda L = r_{(hkl)} d_{(hkl)}$$
(11.1)

where λ is the wavelength of the incident beam, L [mm] is the camera length and the product λL is the camera constant, d [nm] is the lattice spacing of the reflecting plane and r is the measured spacing of the $d_{(hkl)}$ planes on the photographic plate.

From this calibration, the camera constant was found to be 2.776 (nm.mm). This value was compared to one determined from the calculated camera constant where:

$$\lambda = \frac{h}{\sqrt{2m_0 Ee \left(1 + \dfrac{Ee}{2m_0 c^2}\right)}}$$
(11.2)

where h is Planck's constant (6.626×10^{-34} J Hz^{-1}), m_0 is the rest mass of an electron (9.11×10^{-31} kg), e is the elementary charge (1.602×10^{-19} C), c is the speed of light in vacuum (2.998×10^8 m s^{-1}) and E is the accelerating voltage in volts (160 kV).

The camera length that was used to photograph diffraction patterns was 950 mm and the calculated camera constant was found to be 2.66 (nm.mm), giving an error of 4 percent from the

experimentally determined one. In the identification of the particles from the diffraction patterns the experimentally determined camera constant was used.

11.4.2 Optical metallography for the study of the recrystallised volume fraction

The samples were mounted in epoxy, ground and polished to 6 μm and chemically polished by immersing them in a 3 ml hydrofluoric acid (HF) and 97 ml hydrogen peroxide (H_2O_2, 30 percent) solution for 5 seconds and rinsed in H_2O_2. Immersing the samples for longer times resulted in undesirable pitting. An alternative chemical polishing solution that was equally suitable was 14 ml HF, 100 ml H_2O_2, 100 ml H_2O solution.

Generally, grinding disturbs the structure of the surface metal and, therefore, the chemical polishing was intended to remove this disturbed metal in order to minimise any artefacts that would interfere with the quantification of the recrystallised volume fraction. After polishing, the samples were etched by swabbing up to 5 seconds in 4 percent Nital. Under the optical microscope, the recrystallised regions appeared lighter while the unrecrystallised ones darker, creating the needed phase contrast for the quantitative analysis of the recrystallisation process.

Two methods, both of them based on metallographic analysis were used, namely, the point count method and the image

analysis technique that employs a computer software package called anlySIS^{TM}. Each point that was plotted in the graphs of recrystallised volume fraction versus the isothermal annealing time was an average of the recrystallised volume fraction taken from twenty-five different areas of the specimen and the error bar was the standard deviation.

11.4.3   Preparation of thin foils for TEM observations

Specimens for thin foil TEM observations were electro-erosion cut into discs 3 mm in diameter and 0.6 mm thick. They were then mechanically ground to a thickness between 40 and 100 μm before thinning them in a twinjet thinning apparatus. The electrolyte that was used was 1.25 litres of acetic acid, 0.08 litres of perchloric acid and 0.7 g of chromium oxide (CrO_3) while the voltage was set at 50 V. Figure 11.7 shows the characteristic curve of current versus voltage that was obtained from the twinjet that was used in the thinning of the foils. The flat region was the applied voltage range where electrolytic polishing was achieved and below that, etching took place instead of polishing.

After thinning i.e. after a small hole appeared in the foil, the foil was rinsed thoroughly in ethanol to remove all the acids that could corrode and contaminate the specimen.
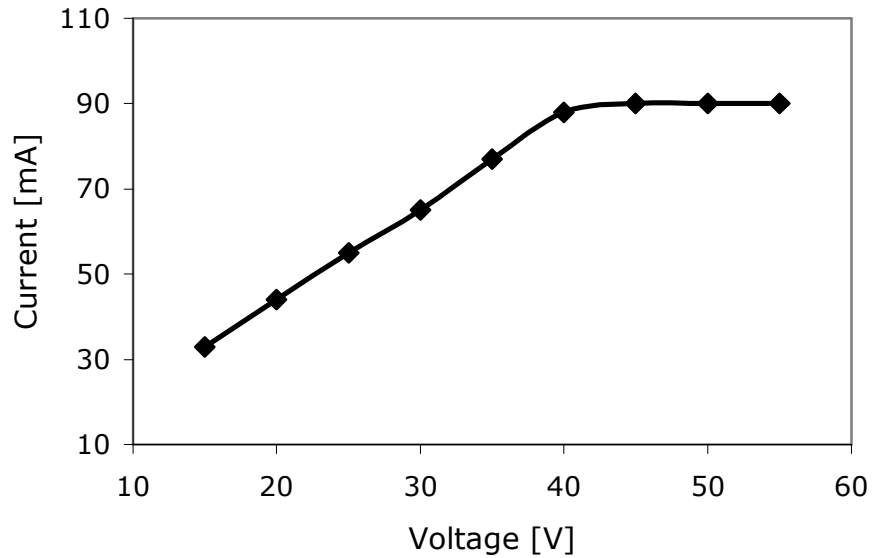
Figure 11.7: Voltage-current characteristic curve for electrolytic polishing in the twin jet apparatus that was used in this work.