Chapter III

Experiment

Investigated Steel

In this work the investigated steel is an aluminium killed commercial quality, St.15. However, Herman and Messien [34] found that r value of this steel can be improved by use of appropriate lubricant during hot rolling and adapted cold rolling reduction. Bleck, Bode and Feld [41] stated that this steel grade can be increased up to DDQ steel grade. The composition of this steel are shown in table 3-1.

Table 3-1 : Chemical composition in wt% of St.15

C	Mn	Al	P	S	N	Si	Cu	Cr	Ni
0.022	0.18	0.034	0.008	0.008	0.0026	0.01	0.19	0.029	0.02

Figure 3-1 illustrates the development of the yield strength of cold rolled strip within the last 20 years. For unalloyed steels the trend is to produce steel grade with lower yield strength for drawing applications.

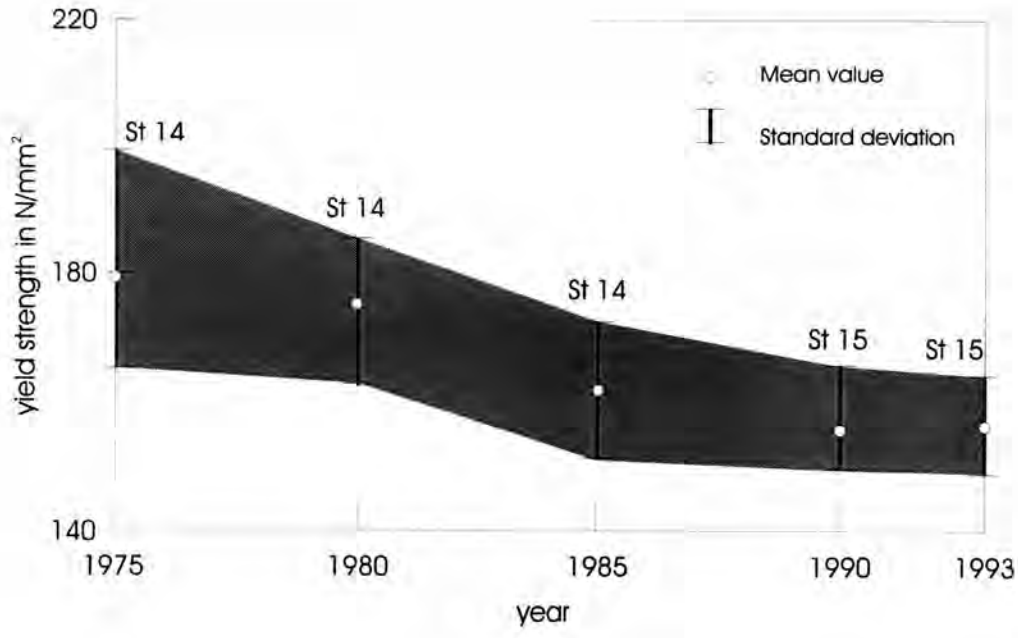
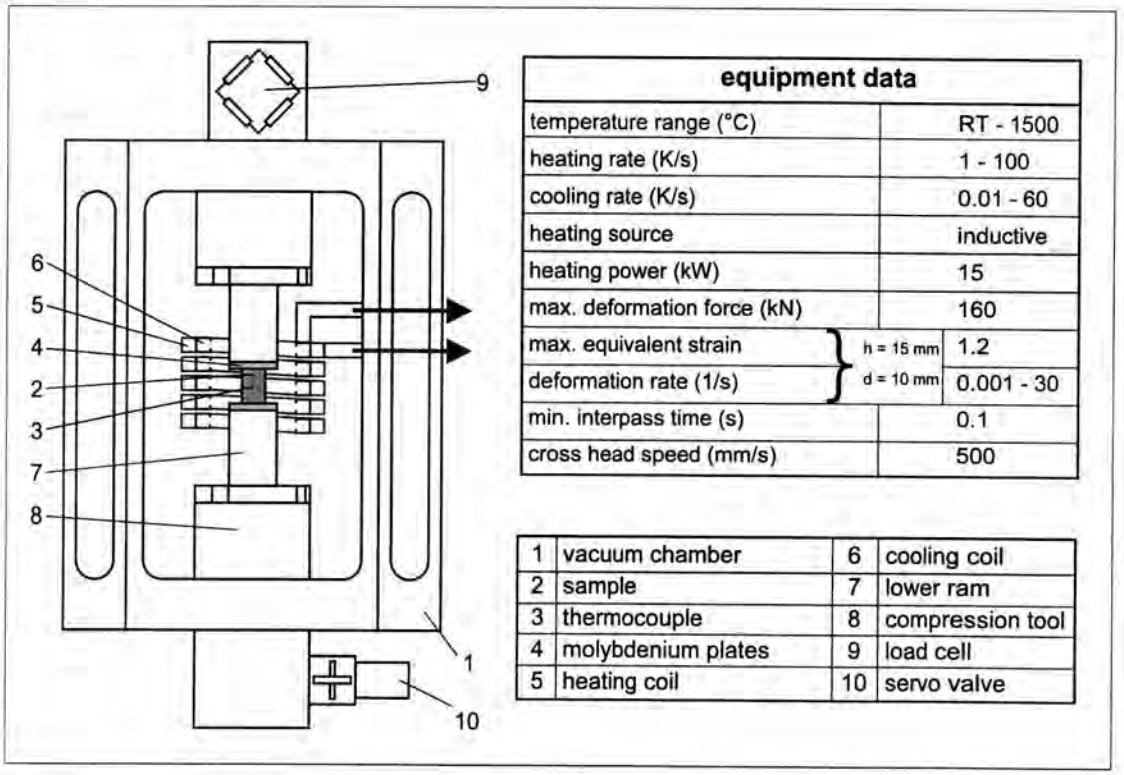


Fig. 3-1 : Development of the yield strength of cold rolled commercial qualities (Bleck et al. [41])

Hot Deformation Equipment

In this work, the " Schenck" hot deformation simulator at the IEHK, RWTH was used to perform hot compression tests. The experiments were curried out in this servohydraulic 160 kN-universal-test-machine. The machine can be used for compression and tensile test. In this work, compression test is selected due to the benefits as described above. In this apparatus various temperatures and deformation schedules can be applied on the sample. All parameters such as time, temperature, strain and strain rate can be varied in a certain range as shown in figure 3-2 (a). The use of a vacuum chamber ensures a non-reactive atmosphere during processing. The samples are heated by an

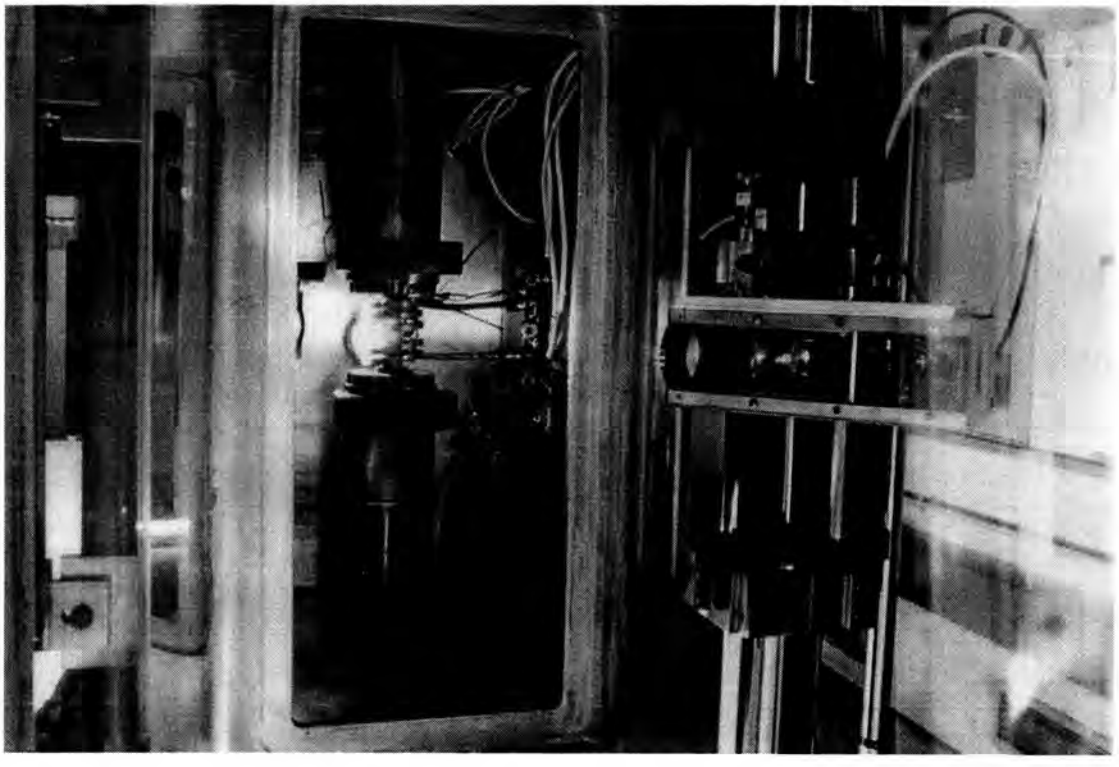
induction facility and can be rapidly cooled after processing. By this the microstructure directly after deformation is preserved to room temperature and can be evaluated. Figure 3-2 (b) illustrates the "Schenck" deformation equipment.



equipment data		
temperature range (°C)		RT - 1500
heating rate (K/s)		1 - 100
cooling rate (K/s)		0.01 - 60
heating source		inductive
heating power (kW)		15
max. deformation force (kN)		160
max. equivalent strain	h = 15 mm	1.2
deformation rate (1/s)	d = 10 mm	0.001 - 30
min. interpass time (s)		0.1
cross head speed (mm/s)		500

1	vacuum chamber	6	cooling coil
2	sample	7	lower ram
3	thermocouple	8	compression tool
4	molybdenium plates	9	load cell
5	heating coil	10	servo valve

(a)



(b)

Fig. 3-2 : (a), (b) "Schenck" hot deformation simulator at the Institute of Ferrous Metallurgy of Aachen University of Technology

Experimental Procedure

The used specimen geometry is a cylinder according to the RASTEGAEV specimen geometry, which is about 7.5 mm in diameter and 15.0 mm in height. Moore [38] described that RASTEGAEV specimens retain a cylindrical shape up to high strains due to lubricant. Figure 3-3 illustrates the dimension of hot compression samples.

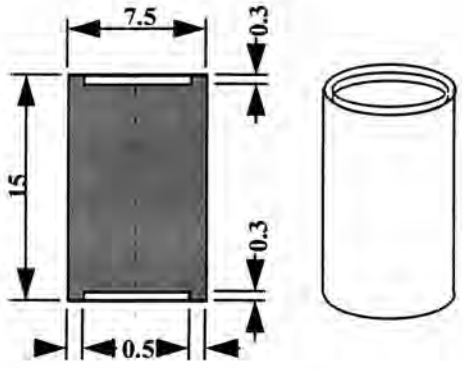


Fig. 3-3 : Specimen geometry

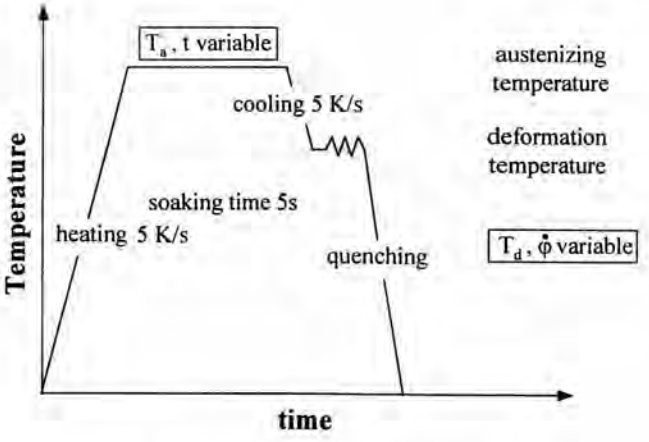


Fig. 3-4 : Experiment procedure

The temperature and deformation schedule for the experiments carried out in the work is schematically shown in figure 3-4.

1. All specimens were heated to austenitizing temperature with a heating rate of 5 K/s under a nitrogen atmosphere.

2. Two different austenitizing conditions were chosen. Austenitization at 1250 °C for 10 minutes was carried out to achieve a coarse initial austenite structure. Austenitization at 1000 °C for 5 minutes was performed to obtain fine grains. The different austenitizing conditions were applied to investigate the effect of reheating temperature and subsequent initial grain size on deformation behaviour.

3. After austenitization the specimens were cooled to different deformation temperatures between 700 °C and 1250 °C(or 700 °C and 1000 °C) with a cooling rate of 5 K/s. Before deformation the samples were hold at this temperature for 5 s to homogenize the temperature distribution.

4. The specimens were deformed with different strain rates ($\dot{\varepsilon}$) of 0.01, 0.1, 1.0 or 10.0/s. Glass powder was used as a lubricant to reduce friction during deformation and ensure a homogeneous strain distribution in the sample.

5. Immediately after deformation with a maximum strain of 1.2 or 1.6, the specimen were quenched to room temperature by nitrogen gas.

Calculation of Flow Curves

During the hot compression test all process parameters i.e. force, upset distance, temperature and time were recorded. After compression tests the data is used to calculate the flow stress-strain curves. For the calculation and the evaluation of stress-strain curves, AWPROG (a special computer program at the IEHK) was used. The resulting flow curves were changed into ASCII files, which can be read by other computer programs. In this work, Microsoft Excel program was used for plotting the flow curve results.

After getting the stress-strain curves, the critical strain was calculated by equation 1. Critical flow stress (S_{crit}) which means the equivalent stress for initiation of dynamic softening, can be evaluated and equals to the stress at critical strain.

After calculating S_{crit} and plotting the curve as a function of deformation temperature, the ferritic, dual-phase and austenitic range can be determined approximately from this curve as demonstrated in figure 4-1.

Metallographic Examination

After deformation, the specimens were prepared for metallographic examination as follows:

1. The specimens were cut, cleaned and cold embedded.

2. The sample were ground with SiC grinding paper (number 80, 100, 200, 320, 400 and 600).

3. The specimens were cleaned by ultrasonic shaking in alcohol and were polished with diamond powder (6 μm), washed and dried.

4. In the last step the specimens were polished with diamond powder (1 μm) after cleaning in an ultrasonic bath.

5. For development of the microstructure the samples were etched with an ethanol based nitric acid 3% for 25-40 s, washed and dried.

6. Micrographs were taken at a magnification of 100 (at the position documented figure 3-5 (b) for measuring the grain size. The grain size numbers were evaluated by comparing the photographs of the microstructure with ASTM standard pictures at the same magnification.

Normally, photographs are taken at a position shown in figure 3-5 (a). At this position the obtained deformation is equal to the nominal strain. This is important because as the strain distribution in the sample volume is not homogeneous, the amount of recrystallization usually increases from the middle to the surface of the specimens similar to the strain gradients in the rolling process (Hansen and Speer [42]). Because, in this work, the minimum height of the samples after deformation was only 3 mm the photographs were taken at the position illustrated in figure 3-5 (b).

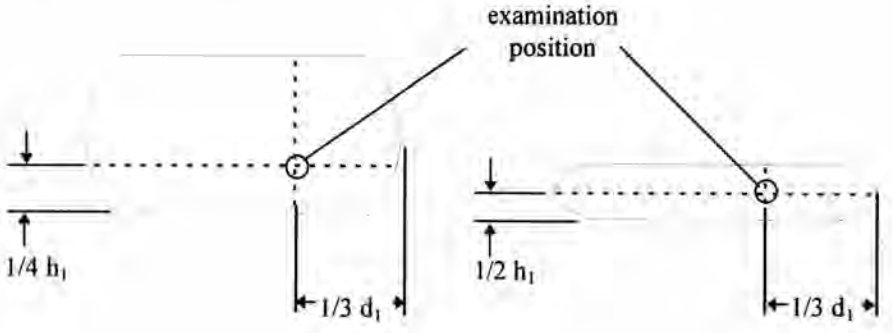


Fig. 3-5 : Illustration of relative position for metallographic examination

(a) normal specimen (b) thin specimen

Techniques for Measuring Austenite Grain Size

The initial grain size before deformation was determined from samples which were quenched with water after austenitization. The etching used for measuring the austenite grain size is etching reagent based on picric acid, which composes with

40 ml aqueous picric acid

60 ml distilled water

5 ml Mirasol

13-15 drops hydrochloric acid

The etching temperature was about 30-60 °C. With this a grain size of about 35 μm was obtained after austenitizing at 1000 °C for 5 min, according to ASTM grain size number 7. After high temperature austenitizing at 1250 °C for 10 min a coarse austenite structure with a mean grain size of about 370 μm, according to ASTM grain size number 0 was achieved. By this, effect of initial grain size on the softening behaviour can be investigated.