CHAPTER III

EXPERIMENTAL PROCEDURE

3.1 Method and procedure

The simplified flow sheet for the test procedure of this study is shown in Fig.3-1. The procedures begin at the characteristic examination of tested steels. The hot tensile equipment were carried out to investigate the reduction of area and the critical temperature range (ZST, ZDT, ΔT) of the tested steel. The quenching equipment was used to freeze the actual microstructure of the tested steel at the same temperature of hot tensile test. The microstructure of specimen was analyzed with optical microscope and SEM-EDX.

3.2 The tested steels

Billets of tested steels were taken from Krupp Stahl GmbH. The chemical compositions of the steels, as shown in Table 3-1, were analyzed with an Atomic Emission

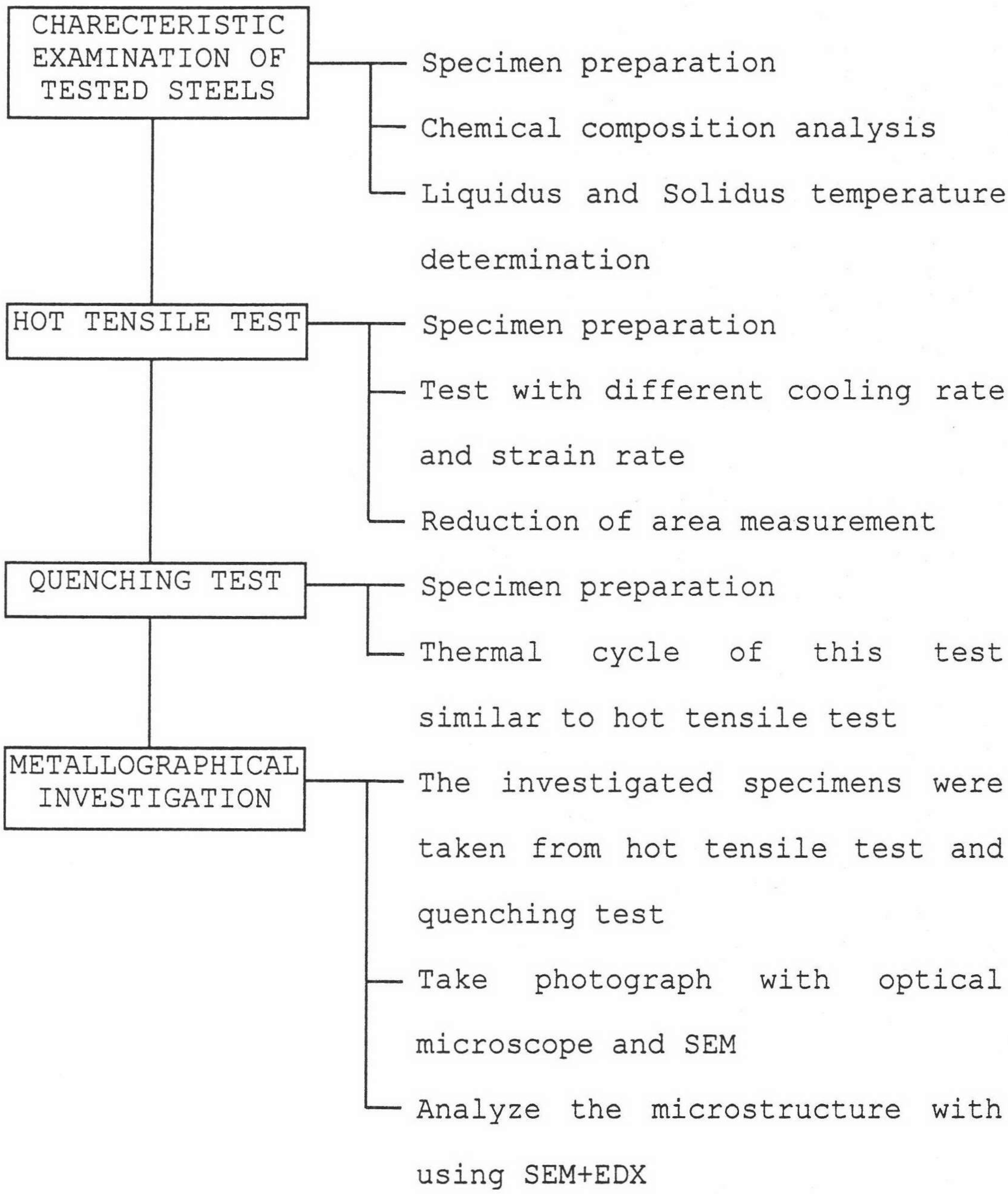


Fig.3-1 Flow sheet for the test procedure.

Spectrometer. The liquidus and solidus of the tested steels were investigated with the aid of Differential Thermal Analysis (DTA). The DTA-curves of the tested steel are shown in Fig.3-2 for steel grade AISI L3 and Fig.3-3 for steel grade AISI O1. The liquidus and solidus temperature of the steels are shown in Table 3-2. The liquidus of the tested steels were taken for adjustment of the temperature about 20-40°C over melting point of each steel grade in hot tensile test.

3.3 Hot tensile test

The hot tensile tests were performed on tensile testing equipment (brand name: Trebel). The testing equipment, as shown in Fig.3-4, consists mainly of a vertical servohydraulic tensile testing machine. The machine which is designed for maximum tensile load of 120 kN, a high frequency generator for the inductive heating (24 kW, 250 kHz) of the specimen, and a connecting electronic unit for controlling the operation and data collector. The hot tensile test specimens were cut and machined in dimension as shown in Fig.3-5. The dimension of the specimen is 20 mm in diameter, 130 mm long. The specimen is bored 2.5 mm below the surface along axial

Table 3-1 Chemical compositions of the tested steels

Steel Grade	Mass content in percent (%)							
AISI L3	C	Si	Mn	P	S	Cr	Ni	Mo
	1.01	0.307	0.290	0.0157	0.0225	1.451	0.0267	0.002
	Co	W	V	Al	Cu	Nb	Ti	
	0.0054	0	0.0022	0.0288	0.0376	0.0002	0.0025	
AISI O1	C	Si	Mn	P	S	Cr	Ni	Mo
	1.02	0.238	1.072	0.0169	0.0042	0.541	0.1239	0.0374
	Co	W	V	Al	Cu	Nb	Ti	
	0.0132	0.573	0.1447	0.0239	0.1057	0.0012	0.0033	

Table 3-2 Liquidus and Solidus temperature of the tested steels

Steel grade	Solidus temperature (°C)	Liquidus temperature (°C)
AISI L3	1381	1443
AISI O1	1394	1450

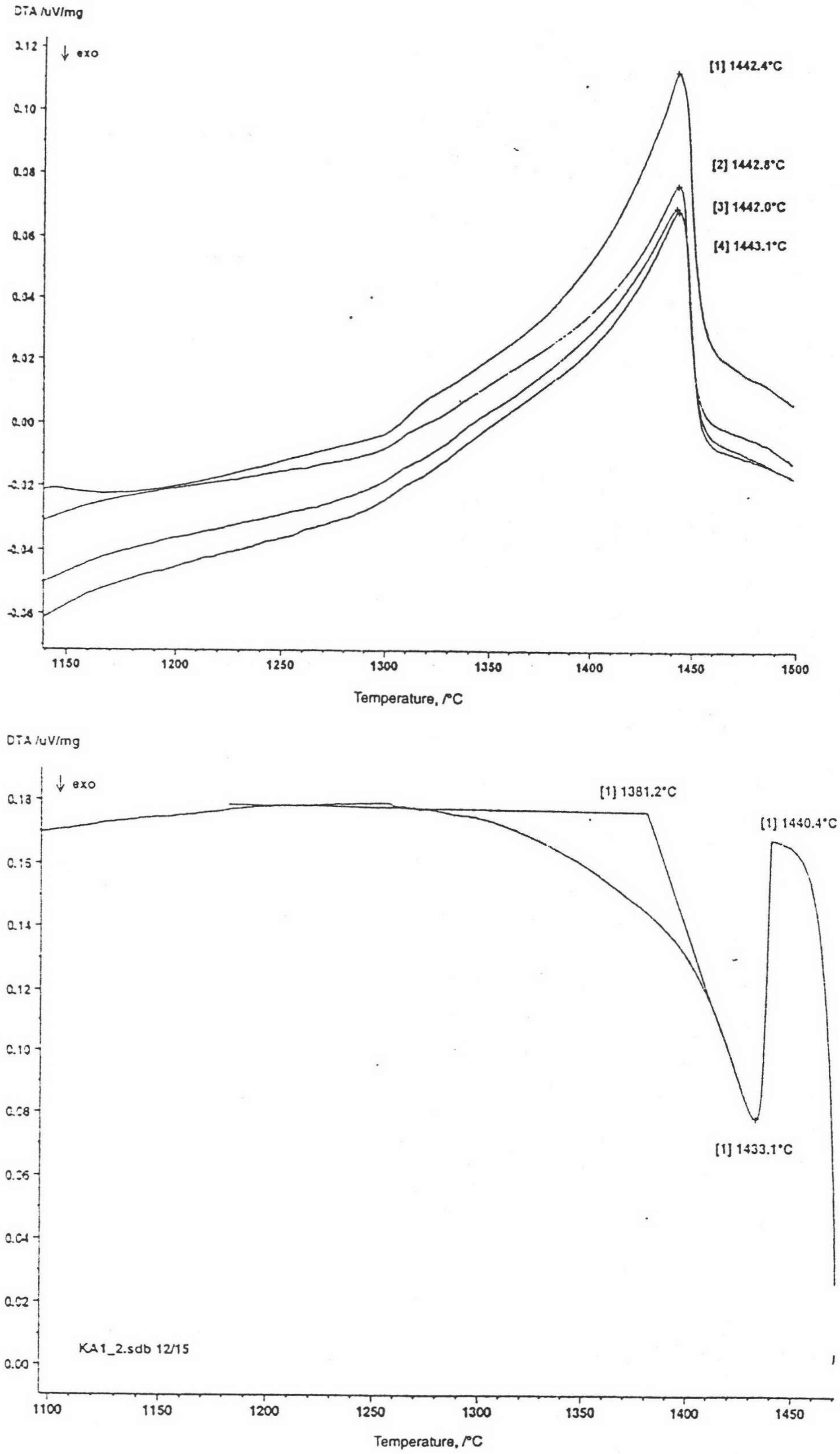
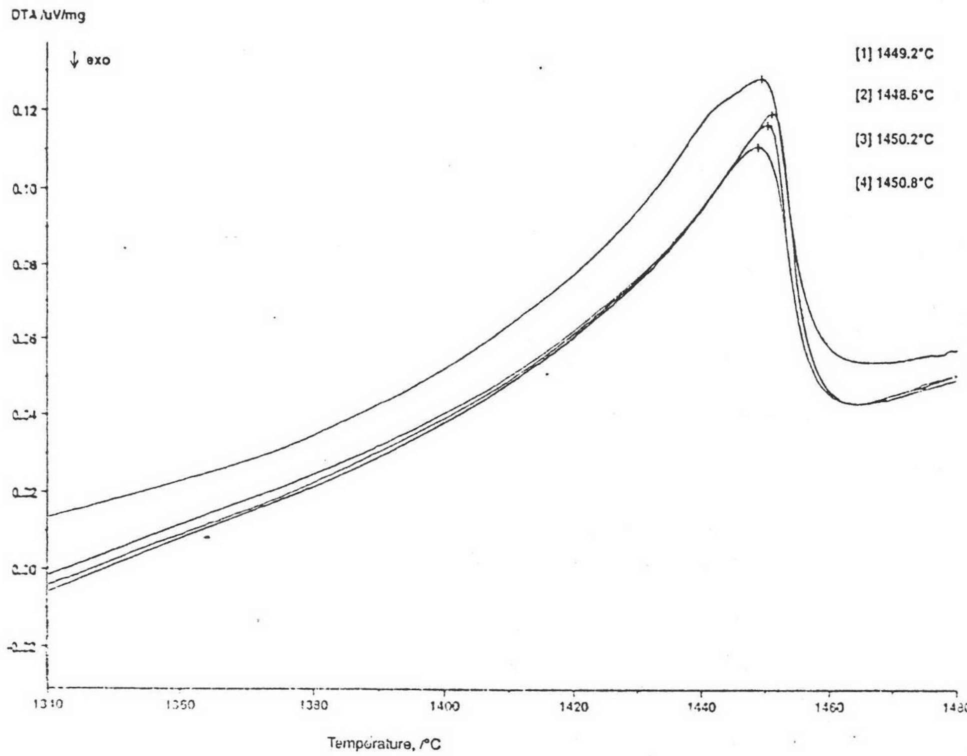
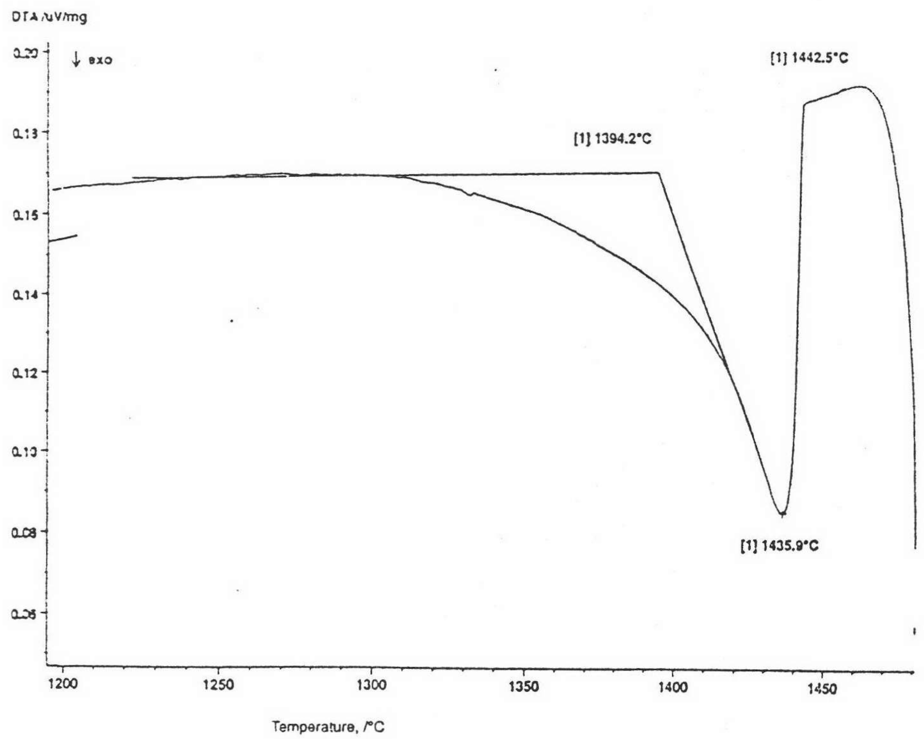


Fig.3-2 DTA-curve of steel grade AISI L3.

(A) Heating curve (B) Cooling curve



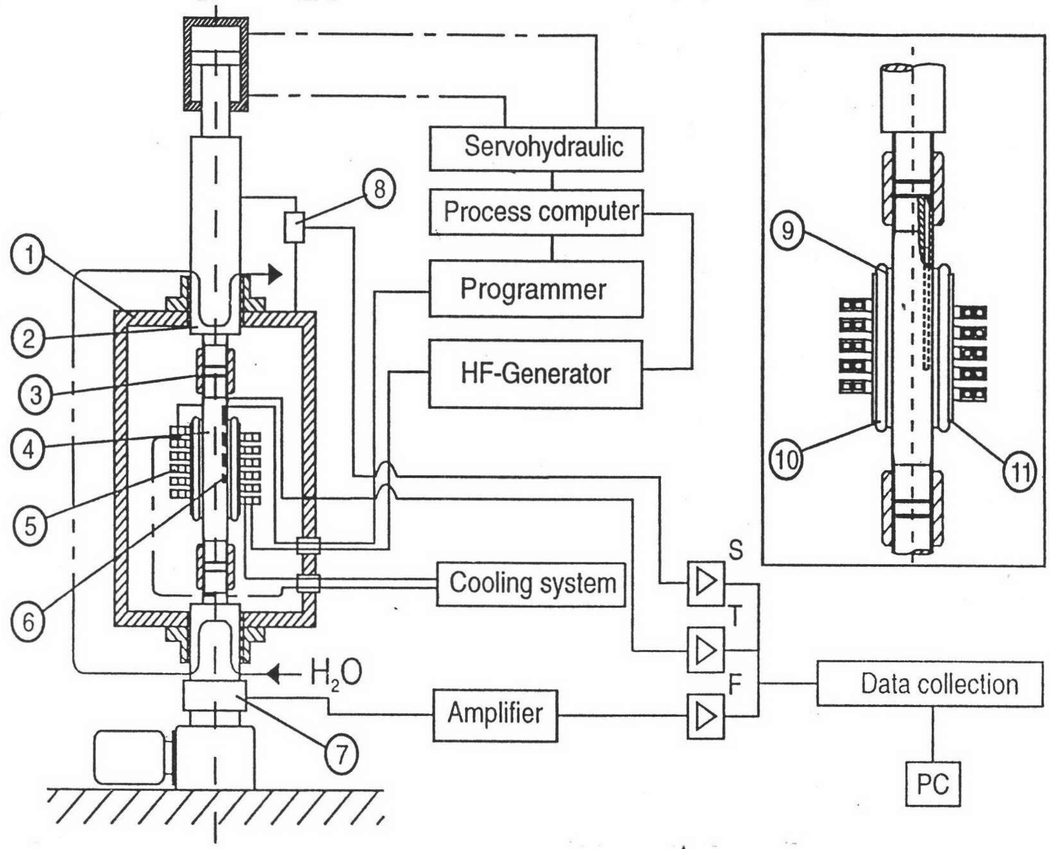
(A)



(B)

Fig.3-3 DTA-curve of steel grade AISI O1.

(A) Heating curve (B) Cooling curve



Heating	induction, 250kHz
Power	24 kW
Heating rate	max. 100°C/s
Cooling	Air and/or Inert gas
Cooling rate	max. 6 °C/s
Cross head speed	0.004 - 10 mm/s
Maximum force	120 kN
Specimen geometry	Ø 7-25 mm x 130 mm
Measurement possibilties	F, S, T
Solidification structure	axial, radial, mixture

1. Vacuum chamber
2. Tension bar (water cooled)
3. Mounting
4. Specimen
5. Induction coil
6. Tube for thermocouple
7. Force measurement unit
8. Inductive length unit
9. Al_2O_3 - Crucible
10. Refractory isolation material
11. Protection quartz tube

Fig.3-4 Experimental set-up of the hot tensile test equipment and the main data.

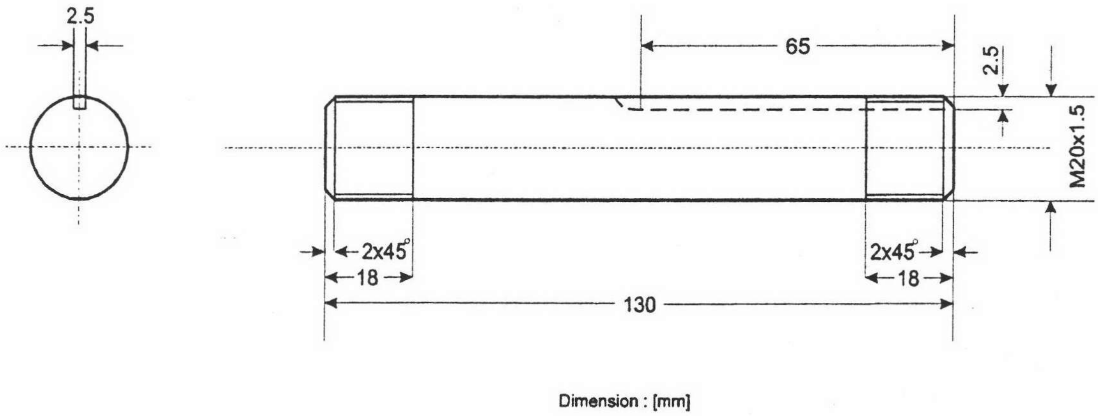


Fig.3-5 Dimension of the hot tensile specimen.

direction. In this gap a thermocouple is placed so that its tip is later positioned in the middle of the 30 to 40 mm long melted-down zone. In order to stabilize this melted area during the melting of the specimen the middle part of the specimen is surrounded by an inert Al_2O_3 tube, which is additionally covered with a ceramic texture and a further covered with quartz glass tube. This arrangement is positioned in the central of an induction coil. The heating facility is then attached in the center of a chamber, which is water-cooled. The hot tensile test of the specimen is conducted in argon atmosphere at a pressure of 0.02 bar to prevent oxidation.

The temperature cycle of this test is shown in Fig.3-6. The specimens were heated up with a heating rate

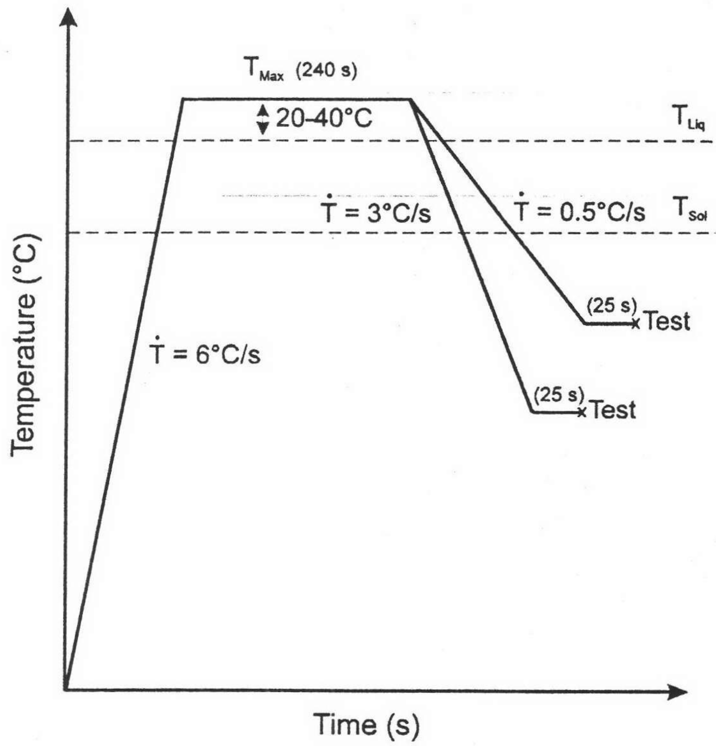


Fig.3-6 Temperature cycle of the hot tensile test.

Of 6°C/s to a maximum temperature (T_{max}) of 30°C above liquidus temperature of the tested steel and held for 4 minutes. The maximum temperature of steel grade AISI L3 was 1473°C and of steel grade AISI O1 was 1480°C. After the steel melted the diameter of the remaining liquid in the ceramic tube changed to be 20.6 mm due to the inner diameter of the tube is 20.6 mm. The melted zone was subsequently solidified with a cooling rate of 0.5°C/s and 3°C/s to various tensile test temperatures. The slow cooling rate of 0.5°C/s represented the solidification rate, which seems more suited to the condition in the center of strand. The fast cooling rate of 3°C/s represented the cooling of solidified shell or at the region near-by the surface of the strand. The range of hot tensile test temperature varies between liquidus temperature and 900°C. After reaching the hot tensile test temperature, the specimen were further held for 25 second, to homogenize the specimen at the test temperature. Then the specimens were strained at a strain rate of $2x10^{-3}/s$ and $2x10^{-2}/s$ until the specimen reached fracture. The various strain rates represented in the condition of different casting velocity. The hot tensile condition of each steel grade were designed in four group as follows:

<u>Steel grade AISI L3</u>

Group L3-1; cooling rate: 0.5°C, strain rate: $2x10^{-3}/s$

Group L3-2; cooling rate: 0.5°C, strain rate: $2x10^{-2}/s$

Group L3-3; cooling rate: 3°C, strain rate: $2x10^{-3}/s$

Group L3-4; cooling rate: 3°C, strain rate: $2x10^{-2}/s$

<u>Steel grade AISI O1</u>

Group O1-1; cooling rate: 0.5°C, strain rate: $2x10^{-3}/s$

Group O1-2; cooling rate: 0.5°C, strain rate: $2x10^{-2}/s$

Group 01-3; cooling rate: 3°C, strain rate: $2x10^{-3}/s$

Group 01-4; cooling rate: 3°C, strain rate: $2x10^{-2}/s$

Reduction of area (RA) at fractured specimens was measured at room temperature and the maximum load (F_{max}) as well as the test temperatures is recorded with the help of a recording device. The hot ductility is measured by percentage reduction of area, which is calculated by the equations as follow.

$$\%\text{Reduction of Area } (\%RA) = \frac{D_0^2 - D_F^2}{D_0^2} \times 100 \quad \ldots\ldots\ldots\ldots\ldots (1)$$

Where D_0 is the diameter of the cross section before straining, D_0 = 20.6 mm

D_F is the minimum diameter of the cross section at fracture in mm

So,

$$\%\text{Reduction of Area } (\%RA) = \frac{424.36 - D_F^2}{424.36} \times 100 \quad \ldots\ldots\ldots\ldots (2)$$

3.4 Quenching test

The quenching test is applied to freeze the microstructure of the tested steel at the same testing temperature of hot tensile test. This procedure is also applied to investigate the microstructure of solidification sequence of the tested steel. It is necessary to carry out quenching tests due to the fact that the hot tensile test specimen after fracture remains in the chamber until being cooled down to room temperature. The holding of fractured specimen until being cooled down to room temperature resulted in modification of microstructure occurred during hot tensile test.

The quenching equipment and the main function, which is used in this study, is shown in Fig.3-7. The equipment consists mainly of quenching furnace, cooling system, heating source, driving mechanism (to adjust position of the specimen in the furnace), temperature measurement, atmosphere system, water bath and controlling system. The closing system is installed to put the sample into the furnace. The quenching test specimen is 4.9 mm in diameter and 6 mm long. The specimen is placed in an inert Al_2O_3 crucible and then is dropped into an inert Al_2O_3 tube in the position of heating zone of the furnace. A power unit heats the specimen and the test temperature is measured with a Pt-Pt/Rh13-thermocouple. The furnace is under Ar-atmosphere to protect the specimen against oxidation all the time during test. The temperature-time cycle of the test is the same to hot tensile test. A computer program controls the operation of the test. To freeze the microstructure at a certain test temperature the specimen is dropped after the temperature-time cycles

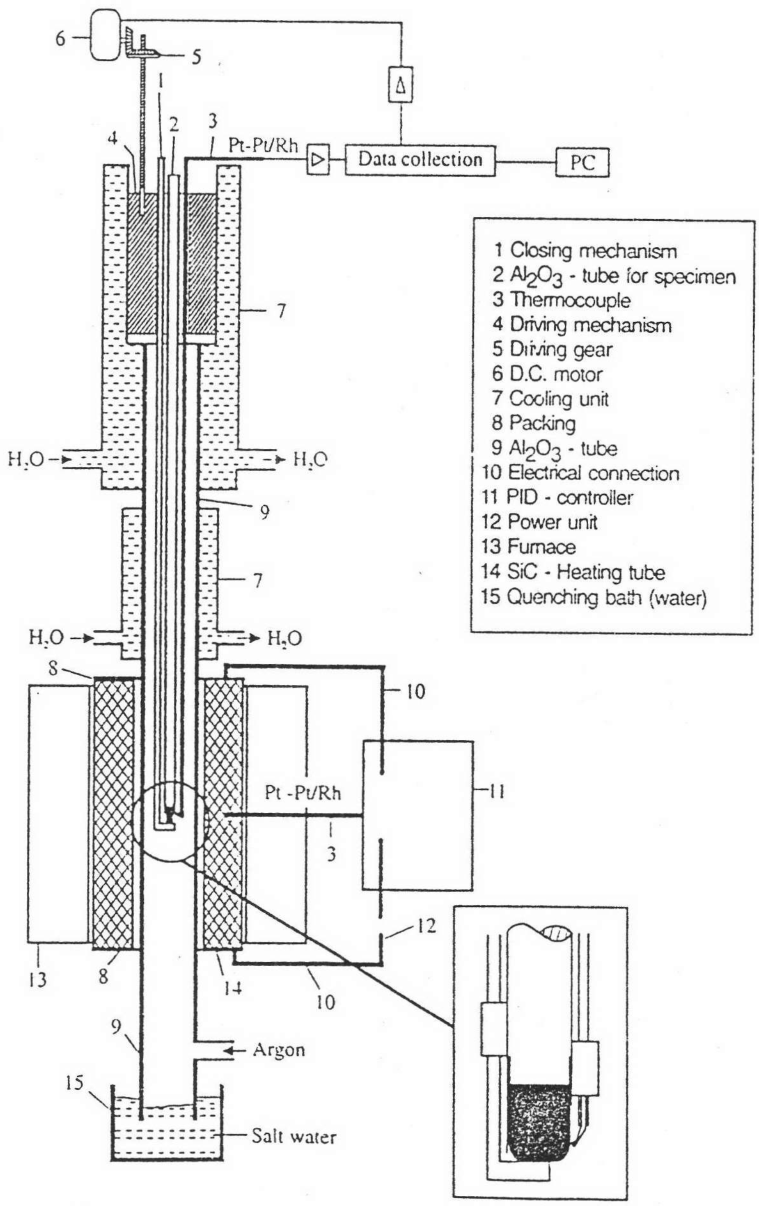


Fig.3-7 Schematic diagram of the quenching equipment.

in a cooled water bath, with approximate temperature of 0° C, located below the furnace. Then the specimen was prepared to investigate the microstructure with metallographical technique.

3.5 Metallographical investigation

The specimens of hot tensile test and quenching test were analyzed with an optical microscope and SEM. The elements that occurred in quenching specimens were analyzed with SEM-EDX. The specimens ~~were~~ prepared with the following steps as described below.

(1) Mounting, the hot tensile test specimens were cut about 2 cm from the fracture edge. Then the specimens were made a groove along the direction of tension, cleaned with alcohol, dried and then cold-mounted. The quenching test specimens were prepared by hot mounting.

(2) Grinding and polishing, the specimens were ground with coarse grinding stone and were then ground with various SiC-papers, with fine number of 80, 120, 240, 320, 400 and 600. Finally the specimens were polished with diamond paste in a fines number of 6 μm and 1 μm, respectively.

(3) Etching, the specimens were etched in a solution developed by Lichteneggen and Beoeck in the so-called LBI agent. It consists of 100 ml distilled water, 20 g of ammoniumbifluoride and 0.5 g of potassium-disulfide.

(4) Analysis, the etched specimen photographs were taken with optical-microscope and SEM. The elements of quenching specimens were analyzed with using SEM-EDX.