## III. EXPERIMENTAL PROCEDURE

The material studied was a commercial Nb-Ti microalloyed steel having the composition given in Table 3.1 . The starting condition was 25 mm thick transfer bar which has been hot rolled and air cooled

Table 3.1 Chemical composition of studied steel

C	Mn	Si	P	S	Al	Cu	Nb	Ti	v	Zr	N
0.10	0.91	0.01	0.011	0.008	0.041	0.029	0.031	0.041	0.003	0.005	0.0076

## 3.1 Simulation of reheating stage

In order to simulate reheating stage, the samples of 25x25x50 mm in size were cut and then reheated at temperature between 900°C to 1200 °C in steps of 50 °C for a period of 25 minutes. Details are shown in Figure 3.1 Immediately following reheating, samples were water-quenched in a salted water bath.

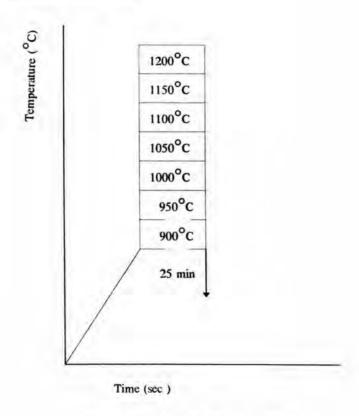


Fig. 3.1 Simulation of reheating stage

Quenched samples were sectioned and prepared for metallographic examination. The samples were etched in saturated aqueous picric acid solution containing 1-2 drops of FeCl<sub>2</sub> and 3-5 drops of saponate. Eching was carried out at 70 -80 °C for time ranging from 10 to 30 minutes to adequatly reveal prior austenite grain size. The mean grain size was measured using quantitative metallographic technique (circle intercept method) according to ASTM E112, details are described latter, result on austenite average grain size. Reheating temperature range and 25 min holding time were selected in order to study more about reheating parameters. Three different temperature of 1100°C,1150°C,and 1200 °C, temperature were selected with holding time of 15, 35, 45 and 60 minutes for each temperature as showen in Fig. 3.2. Subsequently microstructure and prior austenite grain size were examined by the same method as mentioned before.

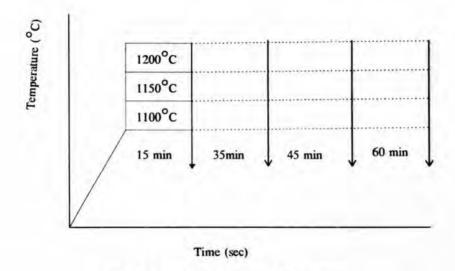


Fig. 3.2 Study the effect of holding times

# 3.2 Simulation of deformation in austenite recrystallization region.

The samples were reheated at 1150 °C for 30 minutes. This reheating temperature and soaking time came from reheating stage analysis. At strain rate of 8 s<sup>-1</sup>, three reductions 30%, 40% and 50 % were carried out using single pass rolling in experimental pilot rolling mill. The rolled sample were water quenched immediately after rolling (in less than 5 sec). Section and polished samples were etched in saturated aqueous picric acid solution containing 1-2 drops of FeCl<sub>2</sub> and 3-5 drops of saponate at 60°C-70°C for 5-10 minute. Microstructures

were examined to obtain prior austenite average grain size. Detail of this experamental are described in Fig. 3.3.

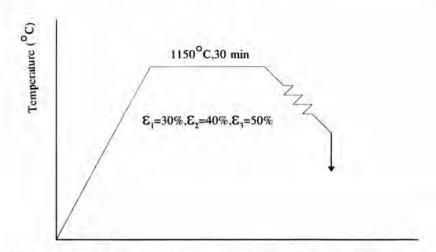


Fig. 3.3 Simulation of deformation in austenite recrystallization region

## 3.3 Simulation of deformation in nonrecrystalization region.

Two sets of sample were subjected to different reduction of 40% and 50% respectively in recrystalization region, after reheated at 1150 °C for 30 minutes. After that five reduction 30%, 40%, 50%, 60% and 70% were carried out at 860 °C which were considered as nonrecrystallization region. All rolling passes were maintained at strain rate of 8 s<sup>-1</sup>. Samples were held either 6 or 360 seconds before quenching to room temperature. Detail of whole experiments are decribed in Fig. 3.4. All sample had a thermocouple embedded 10 mm into middle portion of sample to accurately record thermal history through the rolling and isothermal holding procedure. Section and polished samples were etched in the same way as mentioned in section 3.2. Microstructures were examined to obtain average grain size of deformed austenite. Both parallel and perpendicular to the largest dimension of the deformed austenite were calculated according to method described in section 2.3.

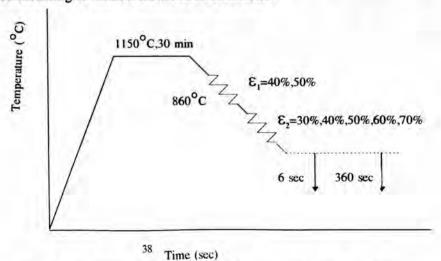


Fig. 3.4 Simulation of deformation of noncrystallization region

## 3.4 Simulation of coiling stage.

To balance both precipitation strengthening and grain refinement strengthening, it is neccessary to optimize the coiling temperature. After evaluating the result from previeous stage of controlled rolling process parameters selected, experiments were conducted as follows:

- -Samples were reheated at 1150°C for 30 minutes.
- -Samples were subjected to first pass reduction of 40% in recrystallization region followed by in furnace to achieve 860°C for 180 secconds.
- -Second pass reduction was to sample at 50% and 60% respectively in nonrecrystallization region.
- -Samples were cooled down by air for various time to achieve six temperatures.

(520, 550, 580, 610, 640 and 670°C)

-Samples were placed immediately in the furnace and kept for 24 hour.

Detail of whole experiment were described in Fig. 3.5. Tensile tests were conducted on samples with tensile direction parallel to rolling direction and ferrite grain size measurments were carried out also.

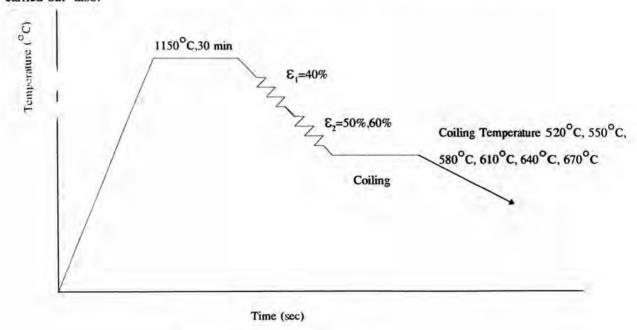


Fig. 3.5 Simulation of coiling stage

## 3.5 Grain size measurement method

Grain sizes, or diameters, have been determined by several methods. Because the grains normally found in alloys have irregular shape, the definition of a diameter is usually arbitrary.

A general, quantitative length parameter provides a unique assumption free value for any granular, space-filling structure, regardless of grain shape, size, or position. This length parameter is the mean intercept length  $L_3$  (true, three-dimensional parameter) obtained from simple  $L_2$  (true, two-dimensional parameter) intercept measurements on the plane of polish. For many random planes, of course, the averaged  $L_2$  values become the true, three-dimensional ( $L_3$ ) parameter.

For space-filling grains, the mean intercept length is defined as:

$$L_3 = L_2 = L_T = D$$

PM

Where D is grain diameter.

L<sub>T</sub> is total test-line length

M is magnification

P is the number of grain-boundary intersections

At least six different fields of measurement need to be conducted.