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APPLICATION OF TAGUCHI METHOD FOR INVESTIGATING THE MECHANICAL PROPERTIES OF A MICROALLOYED STEEL

ZASTOSOWANIE METODY TAGUCHI DO BADANIA WŁAŚCIWOŚCI MECHANICZNYCH STALI NISKOSTOPOWYCH

In the present investigation, the effects of processing parameters, such as roughing, finishing and coiling temperatures, on the strain hardening exponent and yield strength of a Nb-microalloyed steel has been studied by Taguchi method. In order to achieve maximum n-value and yield strength, tests were done in a laboratorial pilot considering a L16 orthogonal array of Taguchi method under following condition: roughing temperature of 1000, 1050, 1100, 1150°C, finishing temperature of 800, 850, 900, 950°C, coiling temperature of 550, 600, 650, 700°C. Then, analysis of variance and signal to noise ratios are performed on the measured data. The results indicated that the finishing and coiling temperatures were the major factor affecting the mechanical properties. The confirmation tests at optimal conditions approve the effectiveness of this robust design methodology in investigating the hot rolling process of the microalloyed steels.

Keywords: Taguchi method, Strain hardening exponent, Yield strength, Microalloyed steel, Hot Rolling

W niniejszej pracy, metodą Taguchi badano wpływ parametrów obróbki takich jak temperatura obróbki zgrubnej, wykańczającej i zwijania na wykładnik utwardzenia i granicę plastyczności stali niskostopowej. W celu uzyskania maksymalnej wartości wykładnika *n* i granicy plastyczności, badania przeprowadzono w skali laboratoryjnego pilotauwzględniając ortogonalną macierz L16 metody Taguchi pod następującymi warunkami: temperatura obróbki zgrubnej: 1000, 1050, 1100, 1150°C, temperatura obróbki wykańczającej: 800, 850, 900, 950°C, temperatura zwijania: 550, 600, 659, 700°C. Następnie dokonano analizy wariancji stosunku sygnału do szumu na zmierzonych danych. Wyniki wskazują, że temperatury obróbki wykończeniowej i zwijania były głównym czynnikiem wpływającym na własności mechaniczne. Testy przeprowadzone w optymalnych warunkach potwierdziły skuteczności zaprojektowanej metodologii w badaniach procesu walcowania na gorąco stali niskostopowej.

1. Introduction

Microalloyed steel sheets with high strength properties and improved formability can increasingly be used in the automotive industry. Controlled hot rolling is considered as a thermo-mechanical process for rolling of microalloyed steel sheets. The hot rolling of microalloyed steel sheets consists of reheating of slabs, reduction of thickness in rolling mill, roughing and finishing the rolling at specific temperatures and additionally accelerated water cooling on run-out table and coiling the final products [1,2]. Efforts to introduce Nb into steel for hot-rolled sheets gave rise to development of important group of microalloyed steel, important for technical and economical reasons. Benefits of micro-additions in steel were fully revealed in High Strength Low Alloy steels (HSLA). Their final properties are provided directly after deformation process as a result of controlled rolling [3]. Niznik and Pietrzyk [4] simulated phase transformation of Nb-microalloyed steels and stated that strain induced precipitation is a phenomenon, which controls the evolution of the microstructure in these steels during thermo-mechanical treatment. Derda and Wiedermann [5] stated that with the advances in continuous steel casting and after developing the thermo-mechanical rolling technology, an increasing number of grades of steels with micro-additions of niobium, titanium and vanadium have been manufactured on a mass scale.

The improvement in the mechanical properties of steels is an important priority for industry. Among different approaches to achieve this, ferrite grain refinement improves the yield strength (YS) of steel without sacrificing the toughness [6]. Although these fine-grained steels show superior strength and fracture behavior, uniform elongation deteriorates with a decrease in ferrite grain size. This inferior ductility is attributed to the low strain hardening ability due to grain refinement [7]. Strain hardening is represented by the exponent n in the flow stress equation $\sigma = K\varepsilon^n$, which approximates the relation between true stress, σ, and true strain, ε, during plastic deformation of a metal. The larger the n-value, the more the material can elongate before necking. Thus, as the n-value in-

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creases, the material's resistance to necking increases, and the material can be stretched farther before necking starts [8-10].

The method presented in this study is an experimental design process called the Taguchi design method. Taguchi design, developed by Dr. Genichi Taguchi, is a set of methodologies by which the inherent variability of materials and manufacturing processes has been taken into account at the design stage. By using the Taguchi techniques, industries are able to greatly reduce product development cycle time for both design and production, therefore reducing costs and increasing profit [11, 12].

In this study, orthogonal arrays of Taguchi method and signal to noise (S/N) ratio are used to measure the performance of the process responses. A statistical analysis of variance (ANOVA) is also performed to identify the process parameters that are statistically significant. In the last section of the paper, experimental confirmation of the results is carried out.

2. Experimental procedure

In this investigation, a microalloyed steel containing niobium with chemical composition shown in the TABLE 1 was used.

TABLE 1

Chemical composition of steel (wt%)

Elements	Fe	C	Mn	Si	Cu	Ni	S	P	Nb	N (ppm)
Balance	Base	0.05	0.5	0.2	0.01	0.02	0.002	0.006	0.04	80

To carry out hot rolling process at a laboratory scale, specimens with 10 mm thickness, 100 mm width and 120 mm length were machined from steel billets. The various critical transformation temperatures, i.e. the start and finish of the austenite-to-ferrite transformation (A_{r3}, A_{r1}) and the non-recrystallization temperature (T_{nr}), were determined by continues cooling compression (CCC) testing [13]. The thicknesses of the specimens were reduced from 10 mm to 3 mm by five rolling passes. The processing schedule of hot rolling cycles is illustrated in Fig. 1.
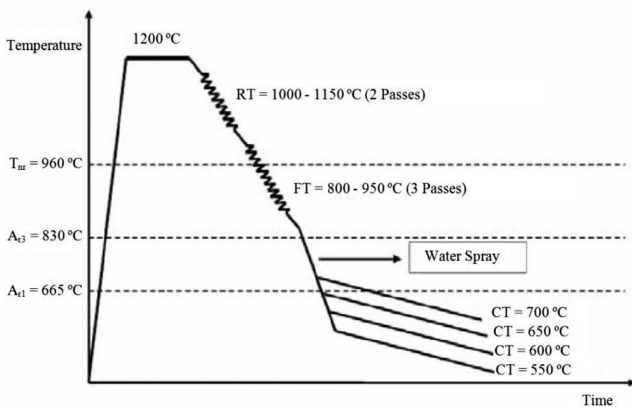

Fig. 1. Schematic representation of laboratory hot rolling cycles

To homogenize the structure before rolling and to dissolve the precipitates like NbC and NbCN, the specimens were reheated for 30 minutes at 1200°C. The rolling was performed on a reversible rolling mill (20 tons) with rolls of 300 mm diameter. The cooling rate (water spray cooling) of the specimens between the finishing temperature and the coiling temperature was about $15°Cs^{-1}$.

The yield strength and n-value were obtained by conducting a simple tensile test at room temperature, in which the specimen is stretched until it fractures. Per ASTM-E646-93 standard, the average gradient of uniform strain area was measured to calculate the n-value. Then, the samples were prepared by metallographic techniques and microstructures were observed by optical microscopy and scanning electron microscopy (SEM) on a longitudinal surface of the specimens.

TABLE 2

Levels of the factors used in studying the effect of RT, FT and CT

Factors	Level			
	1	2	3	4
RT	1000°C	1050°C	1100°C	1150°C
FT	800°C	850°C	900°C	950°C
CT	550°C	600°C	650°C	700°C

Parameters investigated in this study include roughing temperature (RT), finishing temperature (FT) and coiling temperature (CT). Each parameter was studied at four levels as given in TABLE 2. Therefore, a L16 orthogonal array that has 3 factors and 4 levels was chosen.

TABLE 3

The experimental results for a Taguchi L16 orthogonal array

Run	RT (°C)	FT (°C)	CT (°C)	n-value	YS (MPa)
1	1000	800	550	0.184	511.2
2	1000	850	600	0.225	419.3
3	1000	900	650	0.245	345.2
4	1000	950	700	0.261	279.2
5	1050	800	600	0.192	441.3
6	1050	850	550	0.203	440.8
7	1050	900	700	0.260	287.1
8	1050	950	650	0.258	325.1
9	1100	800	650	0.212	393.9
10	1100	850	700	0.250	307.2
11	1100	900	550	0.212	407.1
12	1100	950	600	0.243	378.1
13	1150	800	700	0.236	335.8
14	1150	850	650	0.242	369.8
15	1150	900	600	0.240	350.1
16	1150	950	550	0.225	385.2

The selection of the orthogonal array is based on the condition that the degrees of freedom for the orthogonal array should be greater than or equal to sum of those parameters

[14]. The experimental results for the n-value and YS using the L16 orthogonal array are shown in TABLE 3.

3. Results and discussion

3.1. Analysis of S/N ratio

Taguchi recommends analyzing means of S/N ratio using conceptual approach that involves graphical method for studying the effects and visually identifying the factors that appear to be significant. The term 'signal' represents the desirable value (mean) for the output characteristic and the term 'noise' represents the undesirable value (standard deviation) for the output characteristic [15]. Again based on the objective of the experiment, S/N ratio characteristics can be categorized into three criteria: lower-the-better (LB), higher-the-better (HB) and nominal-the-best (NB). The parameter level combination that maximizes the appropriate S/N ratio is the optimal setting. In the study of the mechanical properties, higher the n-value and YS are expected. Thus, S/N ratio characteristic the higher-the-better is applied in the analysis, which can be calculated using Eq. (1).

$$\frac{S}{N} = -10\,log10\left(\frac{1}{n}\sum_{i=0}^{r}\left(\frac{1}{y_i^2}\right)\right) \qquad (1)$$

Where r is the number of repeated experiment and y_i is the response measured to the ith time [16]. The S/N ratio values are obtained from MINITAB®16 and calculated for each experiment by taking into consideration Eq. (1) (TABLE 4).

TABLE 4

S/N ratio for the n-value and YS

Run	S/N ratio of n-value	S/N ratio of YS
1	-14.7036	54.1718
2	-12.9563	52.4505
3	-12.2167	50.7614
4	-11.6672	48.9183
5	-14.3340	52.8947
6	-13.8501	52.8848
7	-11.7005	49.1607
8	-11.7676	50.2403
9	-13.4733	51.9077
10	-12.0412	49.7484
11	-13.4733	52.1940
12	-12.2879	51.5521
13	-12.5418	50.5216
14	-12.3237	51.3593
15	-12.3958	50.8838
16	-12.9563	51.7137

The means of each level should be calculated for estimating the influential rank of the three factors. By applying Eq. (1), the mean S/N ratios for each level of the parameters are calculated and given for n-value and YS in TABLES 5 and 6, respectively. The levels of the factors that contribute to the highest average values are desired. Also, Figs. 2 and 3 show graphically the S/N ratio of the three hot rolling parameters on n-value and YS, respectively.

TABLE 5

S/N ratio for n-value by factor levels (HB)

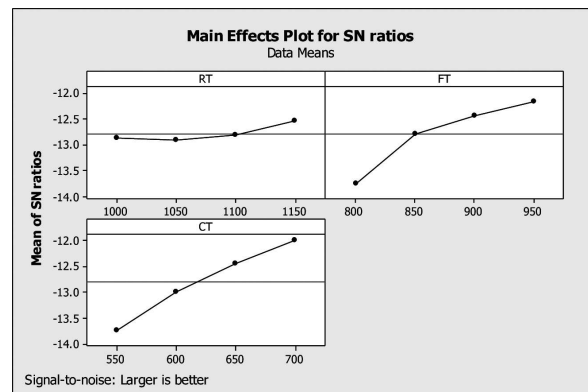| level	RT	FT	CT
1	-12.89	-13.76	-13.75
2	-12.91	-12.79	-12.99
3	-12.82	-12.45	-12.45
4	-12.55	-12.17	-11.99
Δ	0.36	1.59	1.76
Rank	3	2	1

Fig. 2. Main effect plot for S/N ratio of n-value
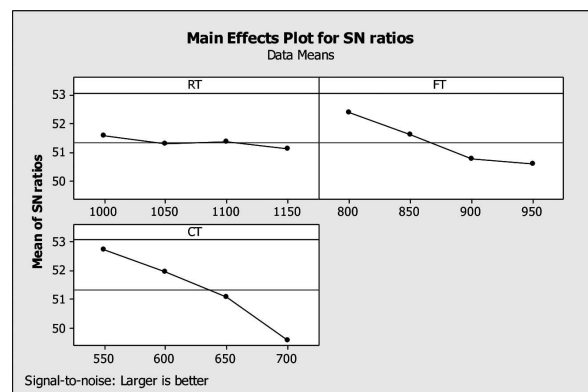
Fig. 3. Main effect plot for S/N ratio of YS

TABLE 6

S/N ratio for YS by factor levels (HB)

level	RT	FT	CT
1	51.58	52.37	52.74
2	51.30	51.61	51.95
3	51.35	50.75	51.07
4	51.12	50.61	49.59
Δ	0.46	1.77	3.15
Rank	3	2	1

Based on the analysis of the S/N ratio in TABLE 5 and Fig. 2, the optimal factor levels for the n-value were obtained at 1150°C RT (level 4), 950°C FT (level 4) and 700°C CT (level 4). Moreover, according to TABLE 6 and Fig. 3, the optimal factor levels for the YS were obtained at 1000°C RT (level 1), 800°C FT (level 1) and 550°C CT (level 1). Kang et al. [17] showed that the strengthening effects caused by solid solution, grain refinement, precipitation and dislocation are significant on the YS. Also, they can interact to some extent, especially as regards the grain refinement and precipitation strengthening effects. Liang-yun et al. [18] reported that by decreasing the rolling temperature, the substructure and dislocation with the high density are formed and retained in austenite, which increase the nucleation sites of the acicular ferrite and promote the acicular ferrite transformation. Acicular ferrite is beneficial to improve the YS.

Delta values for every given factor in TABLES 5 and 6 are defining difference between highest S/N ratio and lowest S/N ratio of factors. From the highest delta values, it is understood that CT has the highest effect and RT has the lowest effect on n-value and YS.

3.2. Analysis of variance (ANOVA)

Analysis of Variance (ANOVA) is a statistical technique for modeling the relationship between a response variable and independent variables (factors). After identifying factors of interest and a response variable along with their levels the order

TABLE 7

ANOVA for S/N ratios of n-value

Source	DF	Seq SS	Adj SS	Adj MS	F	P
RT	3	0.3226	0.3226	0.10754	2.73	0.136
FT	3	5.7987	5.7987	1.93289	49.13	0.000
CT	3	6.8702	6.8702	2.29005	58.21	0.000
Residual Error	6	0.2361	0.2361	0.03934		
Total	15	13.2275				

TABLE 8

ANOVA for S/N ratios of YS

Source	DF	Seq SS	Adj SS	Adj MS	F	P
RT	3	0.4242	0.4242	0.14141	2.030	0.211
FT	3	8.1159	8.1159	2.70531	38.87	0.000
CT	3	21.9036	21.903	7.30120	104.9	0.000
Residual Error	6	0.4175	0.4175	0.070		
Total	15	30.8613				

is randomized in which each set of conditions is run to obtain data. The ANOVA results are used to determine if the predictors or parameters are significantly related to the response. For a 95% confidence level the P-value should be less than or equal to 0.05 for the effect to be statistically significant [19]. The main results from ANOVA for S/N ratios of n-value and YS are given in TABLES 7 and 8, respectively.

The analysis of ANOVA tables based on the P-values indicates that FT and CT are significantly related to n-value and YS, while RT is insignificant.

3.3. Confirmation Experiments

The experimental confirmation test is the final step in verifying the results drawn based on Taguchi's design approach. The optimal conditions are set for the significant factors and a selected number of experiments are run under specified factor conditions. The average of the results from the confirmation experiment is compared with the predicted average based on the parameters and levels tested. The confirmation experiment is a crucial step and is highly recommended by Taguchi to verify the experimental results [20].

The predicated S/N ratio and mean of n-value using the optimum level of the parameters is calculated using Eq. (2) in MINITAB®16.

$$Y = Y_m + \sum_{i=0}^{q} (Y_i - Y_m) \qquad (2)$$

Where Y_m is the total mean S/N ratio, Y_i is the mean S/N at the optimum level and 'q' is the number of significant parameters from ANOVA tables.

The purpose of the confirmation experiment in this study was to validate the optimum factor conditions that were suggested by the experiment which corresponded with the predicted value. The results of the confirmation runs with the optimum factor conditions for n-value and YS are given in TABLE 9.

TABLE 9

Result of the confirmation experiment for the n-value

	Optimal parameters for n-value		Optimal parameters for YS	
	Prediction	Confirmation experiment	Prediction	Confirmation experiment
Level	RT_4 FT_4 CT_4	RT_4 FT_4 CT_4	RT_1 FT_1 CT_1	RT_1 FT_1 CT_1
Mean of response	0.273	0.268	498.3 MPa	504.7 MPa
S/N ratio	-11.1257	-11.2767	54.0201	53.9794

TABLE 9 shows the comparison of the estimated S/N ratio and mean of responses with the calculated S/N ratio and mean of responses obtained in an experiment under the optimum test parameters. These values represent an improvement over the original results and hence confirm the effectiveness of the Taguchi design methodology in optimizing the hot rolling process.

3.4. Development of reduced model

Different regression models were fitted with regression analysis and the coefficients values are calculated using least squares method on MINITAB®16 software. By neglecting insignificant coefficients on responses equation using regression

analysis and ANOVA, final models for n-value and YS were developed as follow:

$$n - value = -0.205707 - 0.00227167RT + 0.00320887FT +$$
$$7e - 007RT * RT + 1.29708e - 006RT * CT$$
$$-1.65e - 006FT * FT - 8.728e - 007CT * CT$$
(3)

$$YS = 4237.33 - 1.3714RT - 5.84956FT +$$
$$0.00191425RT * CT + 0.00308FT * FT -$$
$$0.00234938CT * CT$$
(4)

TABLE 10 and 11 show the ANOVA for the reduced models. As seen from ANOVA tables, regression models are statistically significant. Also, for the statistical analysis of the experimental data, it is essential to assume that the data come from a normal distribution. To determine whether or not the data set is normally distributed, the normal probability plots of residual values for n-value and YS are shown in Fig. 4. The data set has normal distribution if the points fall close enough to the straight line. It is evident from the figures that the experimental points follow a straight line suggesting normal distribution of the data.

TABLE 10

ANOVA for regression model of n-value

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	6	0.0086465	0.0086465	0.0014411	233.554	0.0000000
RT	1	0.0000968	0.0001047	0.0001047	16.973	0.0025987
FT	1	0.0034584	0.0003358	0.0003358	54.428	0.0000420
CT*CT	1	0.0045532	0.0001328	0.0001328	21.518	0.0012213
FT*FT	1	0.0002723	0.0002722	0.0002722	44.123	0.0000945
RT*CT	1	0.0002168	0.0002168	0.0002168	35.137	0.0002214
RT*RT	1	0.0000490	0.0000490	0.0000490	7.941	0.0201111
Residual Error	9	0.0000555	0.0000555	0.0000062		
Total	15	0.0087020				

TABLE 11

ANOVA for regression model of YS

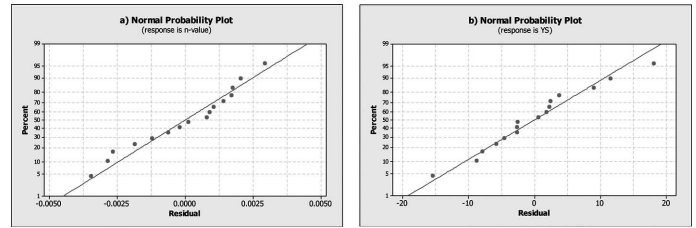| Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	56746.9	56746.9	11349.4	111.215	0.0000000
RT	1	1531.2	616.4	616.4	6.040	0.0338118
FT	1	14889.4	1116.0	1116.0	10.936	0.0079200
FT*FT	1	948.6	948.6	948.6	9.296	0.0122742
CT*CT	1	38905.3	962.0	962.0	9.427	0.0118335
RT*CT	1	472.2	472.2	472.2	4.627	0.0509581
Residual Error	10	1020.5	1020.5	102.0		
Total	15	57767.3				

Fig. 4. Normal probability plots of residuals for a) n-value and b) YS

3.5. Microstructure analysis

The final microstructure of hot rolled sheets is evaluated at four levels of each parameter. Fig. 5 illustrates microstructure of Nb-microalloyed sheets that quenched in four levels of parameters.
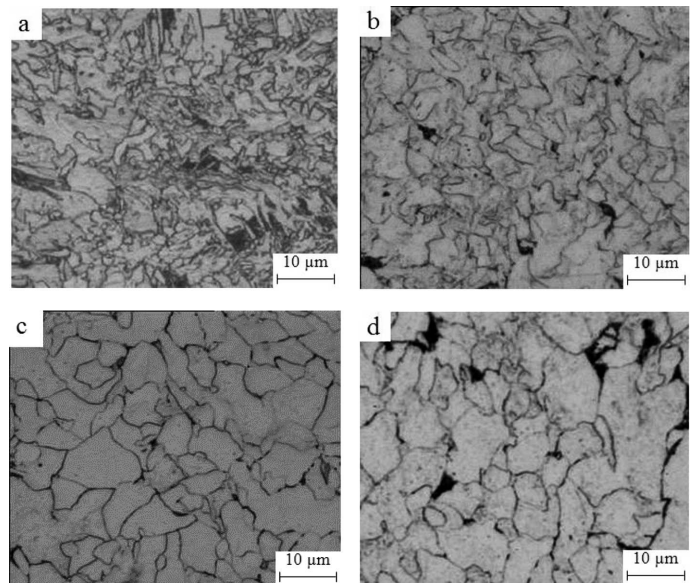

Fig. 5. Optical microstructures of hot rolled sheets at four different level of parameters; a) RT= 1000; FT=800 and CT= 550°C, b) RT= 1050; FT=850 and CT= 600°C, c) RT= 1100; FT=900 and CT= 650°C, d) RT= 1150; FT=950 and CT= 700°C

As shown in Fig. 5, the microstructures have two different features under different conditions, which can be considered and evaluated as ferrite grain size and ferrite morphology. As seen from Fig. 5, the microstructure is varied by increasing the RT, FT and CT of rolling. The effect of the rolling temperature applied in the last pass on grain elongation can be observed in the samples corresponding to low values of RT, FT and CT (Fig. 5a and b). Here the stress accumulates in the austenite, whose recrystallization between passes is partially inhibited by the strong pinning effect of strain-induced precipitates of microalloying elements. Besides, an increase in the YS has been observed, which denotes the accumulated stress due to the incomplete recrystallization of austenite between finishing passes [21]. Also, the increasing of the coiling temperature implies an increasing of the n-value and a decreasing of the YS. This statement is probably due to the presence of the strengthening particles produced by the interstitials and micro-alloying elements [22].

In addition to ferrite grain size changes, the morphology of microstructures is also changed under different conditions.

Roughing and finishing at low temperatures and accelerated cooling of the specimens (the cooling rate about $15°Cs^{-1}$) after the last pass of rolling down to the low coiling temperatures results in acicular ferrite or bainitic ferrite [9]. Acicular ferrite is known as a non-polygonal phase with high substructures which appears through continuous and high cooling rates [10].

The specimens representing hot-rolled steels were also inspected in the scanning electron microscopy (Fig. 6). Small amounts of pearlite and a number of dispersed niobium carbide particles were present in the ferritic matrix.
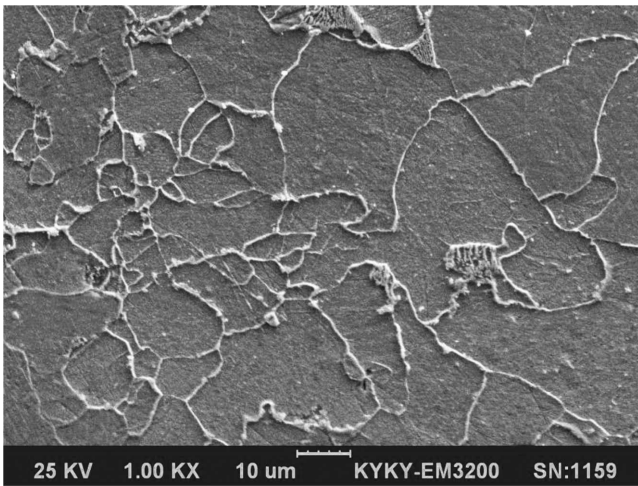

Fig. 6. SEM micrographs obtained at RT= 1100, FT= 900, CT= 600°C

4. Conclusions

The effect of the temperatures of hot rolling on the n-value and YS of Nb-microalloyd steels were studied by using the developed mathematical model and the Taguchi method. The results are summarized as follows:

1. Based on the analysis of the S/N ratio, it can be observed that the optimal factor levels for the n-value and YS were obtained at level 4 of RT, FT and CT, and level 1 of RT, FT and CT, respectively.
2. From the ANOVA, it was understood that the selected factors are related to the n-value and YS of the hot rolled Nb-microalloyed steels but FT and CT have a significant role.
3. The confirmation experiment has been shown that, hot rolling parameters set at their optimum levels can ensure significant improvement in the mechanical properties.
4. A polynomial regression model is developed to control whether the n-value and YS represent a fitness characteristic. Adequate of models are inspected using normal probability plot.

5. The strain-induced precipitates formed by microalloying elements and interstitials play a very significant role on the microstructure evolution and final mechanical properties of conventional microalloyed steels with a microstructure of ferrite–pearlite.

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