Effect of thermomechanical treatment parameters on mechanical properties of duplex ferrite–martensite structure in dual phase steel

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The quantitative effects of the variables used in the thermomechanical treatment (TMT) of a dual phase steel, in the temperature region of intercritical annealing, have been studied by statistical design of experiments. The initial microstructure has tremendous influence on the final microstructure and properties of the steel. The kinetics of transformation is enhanced by the deformation process as has been evidenced by optical and TEM microstructures. The mechanical properties such as tensile strength, yield stress, and relative elongation have been correlated with the TMT parameters and are brought out in the form of regression equations. Percentage phase of ferrite or martensite formed owing to thermomechanical treatment by two different routes has also been quantified in the form of regression equations. The adequacies of the equations were assessed by a Fisher *F* test and the accuracies of the equations have been further verified by performing random experiments in the range of variation of the variables. Isoproperty lines have been constructed using the regression equations developed. The equations can predict the properties within the range of variation of the variables. MST/4261

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Introduction

The structure property correlation of a ferrite–martensite aggregate obtainable from the intercritical annealing of dual phase steels has been reported by various workers.[1–7] The initial microstructure has been shown to influence the morphology and kinetics of the formation of austenite and ferrite in the intercritical annealing zone and, therefore, on the final properties of the product.[8–12] In short, the alloy chemistry, intercritical annealing temperature, intercritical annealing time, and initial microstructure interact in a complex manner. The situation can become more complicated if hot rolling is performed in the intercritical temperature range. Several investigators have studied these aspects,[13–18] however, they have studied the transformation of similar steel taking austenite as starting microstructure.

The present authors[19] have studied the transformation behaviour as a result of hot rolling in the intercritical zone taking first austenite, and second ferrite and pearlite as the initial microstrucutres. At present no report is available quantifying the effect of operational variables on the resultant microstructure and mechanical properties of these steels with different initial microstructures. Therefore, in the present work, quantitative studies on the influence of the above mentioned variables have been attempted using a very powerful technique called 'statistical design of experiments'.[20–22] The authors have demonstrated the usefulness of this technique in earlier work.[23–25]

Wherever necessary, the authors have used standard tools such as TEM, optical microscopy, etc. to explain the quantitative effect of the variables in influencing the microstructure of the steels used in the investigation. A set of regression equations was developed and analysed for understanding the physical metallurgy of the process. These equations can be used for optimisation purposes as has been observed by one of the authors.[26] These equations can also be used for selection of thermomechanical treatment conditions for obtaining desired mechanical properties within the range of variation of variables studied in the experiment.

Experimental

The composition of the steel used in the investigation was Fe–0·08C–1·00Si–1·21Mn–0·02P–0·012S–0·42Cr–0·41Mo (wt-%). The Ac_1 and Ac_3 temperatures, as determined by a Netzch dilatometer, were 700 and 960°C respectively. This enabled the investigators to select the intercritical annealing temperatures at which the thermomechanical treatment experiments could be carried out. In the present studies hot rolling was carried out in the intercritical range by systematically varying the process parameters (i.e. temperature and amount of deformation) following a p^n factorial design,[20–22] where p is the number of factors varied at a time and n is the number of levels each factor is varied. The design matrix employed for the present study is given in Table 1. The variation of temperature and relative deformation were centred around their mean values of 810°C and 20% respectively which are defined as base level.

The design matrix was used for carrying out thermomechanical operations following two routes, which are shown schematically in Fig. 1. For each route the treatment combination was the same, except for the initial microstructure. This enabled comparison of quantitative effect on the variables of the properties resulting from the two different initial microstructures. Steel specimens of size 130 × 40 × 3·6 mm were used. Initial conditioning heat treatment was carried out at 1000°C for 30 min followed by cooling in air to room temperature prior to adopting the different thermomechanical treatment (TMT) routes.

Table 1 Dependence of reduced factors X_1 and X_2 on TMT conditions

	Deformation temperature, °C	X_1	Deformation, %	X_2
Upper level	840	+1	30	+1
Base level	810	0	20	0
Lower level	780	−1	10	−1

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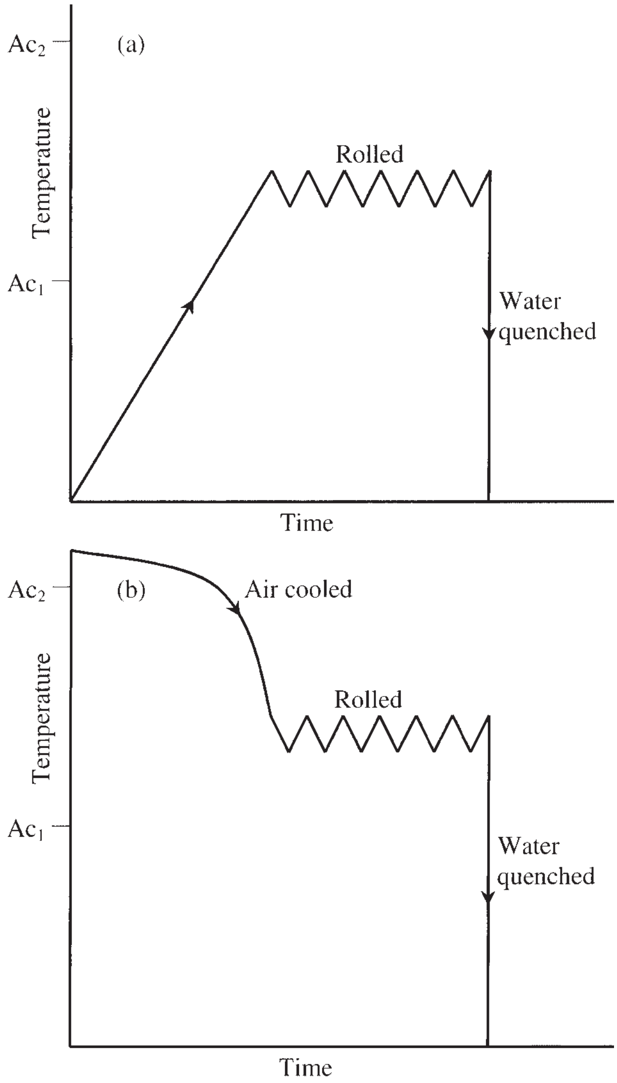


a route 1; *b* route 2

1 Schematic diagrams of TMTs

Chromel/alumel thermocouples (47 SWG) were spot welded on to the edge of the specimens. The cold junction of the thermocouple was connected to a fast response strip chart recorder for monitoring the heating and cooling rates of the specimen. The specimen along with thermocouple was heated in a furnace (situated close to the Hille 50 laboratory rolling mill) at a heating rate of 20 K min^{-1}.

For TMT route 1 the specimens were heated to 1000°C and held for 10 min followed by cooling at a rate of 7 K s^{-1} to a temperature of 780, 810, or 840°C after which the specimens were immediately rolled. At the exit end of the rolling mill the specimens were quenched in water (at a cooling rate of ~833 K s^{-1}). All these operations were carried out with the thermocouple in contact with the specimen and full recording of temperature *v.* time for the entire process was performed.

In TMT route 2 the specimens were heated at the same rate (i.e. 20 K min^{-1}) to one of the intercritical temperatures, i.e. 780, 810, or 840°C and held for 10 min. The specimens were taken out of the furnace and immediately rolled to different degrees at the above prescheduled temperatures, in order to quantify the effect of temperature and amount of deformation. All the specimens were quenched in water at a cooling rate of ~833 K s^{-1} at the exit end of the rolling mill. All these operations were carried out with the thermocouple in contact with the specimen and full recording of the temperature *v.* time for the entire process was performed.

Tensile specimens were tested in an Instron (1195 model) testing machine. Metallographic specimens were studied by optical microscopy. Transmission electron microscope specimens were studied using a Jeol 200CX microscope operating at 160 kV.

Table 2 Treatment combinations and resulting mechanical properties and percentage ferrite (TMT route 1)

Treatment combination	Rolling temperature X_1	Rolling temperature T, °C	Deformation X_2	Deformation d, %	YS, MPa	TS, MPa	e, %	Ferrite, %
1	−1	780	−1	10	740	1080	12	18
2	+1	840	−1	10	390	870	15·8	8
3	−1	780	+1	30	510	980	14·5	48
4	+1	840	+1	30	338	780	17·5	30

Results and discussion

As shown in Fig. 1*a*, TMT route 1 indicates the TMT experiments conducted with austenite as initial microstructure, while TMT route 2 indicates the TMT experiments conducted with ferrite and pearlite as starting microstructure (Fig. 1*b*).

TMT ROUTE 1

Table 2 shows the response obtained as a result of deformation using various treatment combinations. Each response is the average of three separate experimental results carried out at a particular combination of deformation temperature and the amount of deformation.

By treating the data of Table 2, the regression equations (1)–(3) were developed between the yield stress YS, tensile strength TS, relative elongation *e*, and the process variables (i.e. temperature of rolling *T* and relative deformation *d*)[20–21]

$$\mathrm{YS/MPa} = 494{\cdot}5 - 130{\cdot}5X_1 - 70{\cdot}5X_2 + 44{\cdot}5X_1X_2 \qquad (1)$$

$$\mathrm{TS/MPa} = 916{\cdot}25 - 102{\cdot}5X_1 - 47{\cdot}5X_2 + 2{\cdot}5X_1X_2 \qquad (2)$$

$$e/\% = 14{\cdot}95 + X_1 + 1{\cdot}05X_2 + 0{\cdot}2X_1X_2 \qquad (3)$$

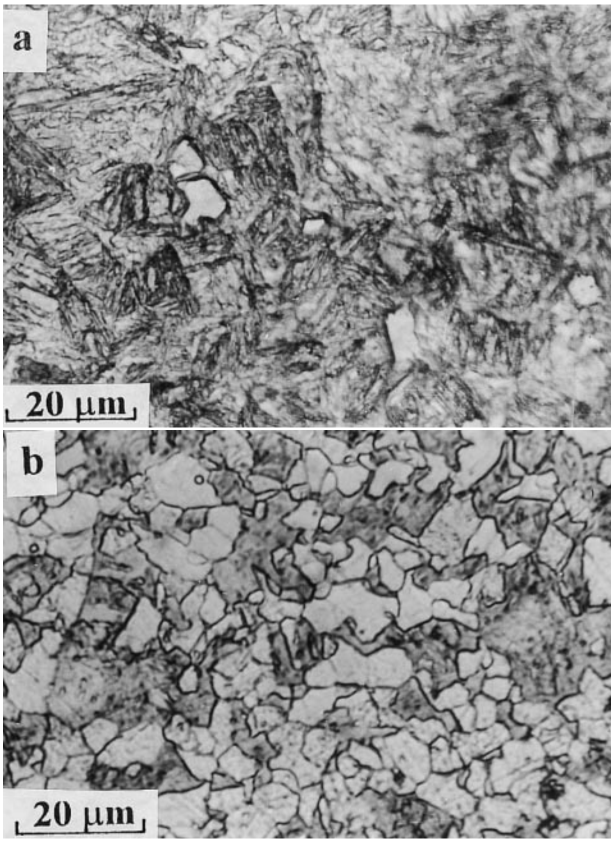
where X_1 and X_2 are in reduced form and can be decoded for their physical values using the following relationships

$$X_1 = (T/^\circ\mathrm{C} - 810)/30$$

$$X_2 = (d/\% - 20)/10$$

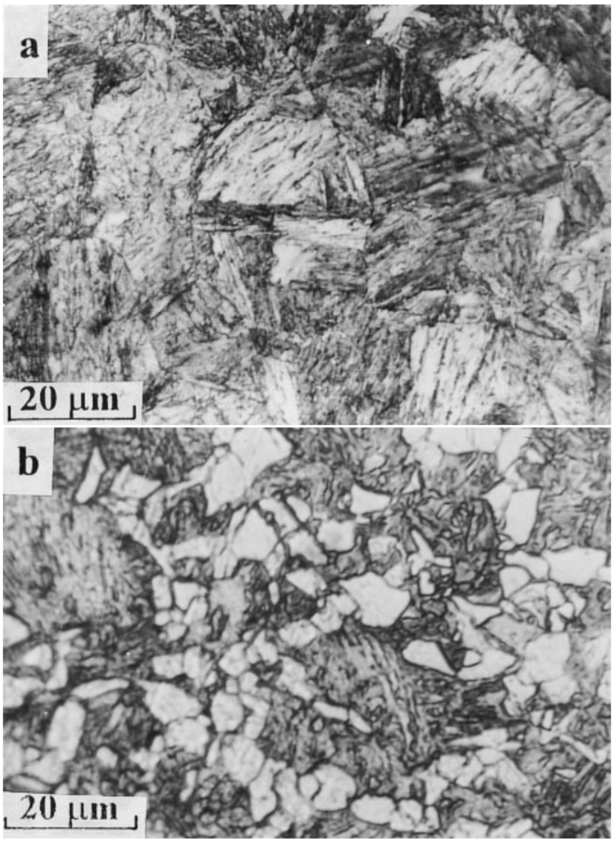
Table 3 Comparison of mechanical properties calculated from equations (1)–(3) and values from random experiments (TMT route 1)

Random experiments: Temperature, °C	Random experiments: Deformation, %	YS, MPa: Equation (1)	YS, MPa: Experimental	TS, MPa: Equation (2)	TS, MPa: Experimental	e, %: Equation (3)	e, %: Experimental
810	20	494·5	505	916·25	910	14·95	14·00
780	20	625	640	1018·75	1030	13·25	13·00
840	20	364	343	813·75	800	16·25	16·50
810	10	565	595	963·75	960	13·90	13·00
810	30	424	435	868·75	865	16·00	16·00



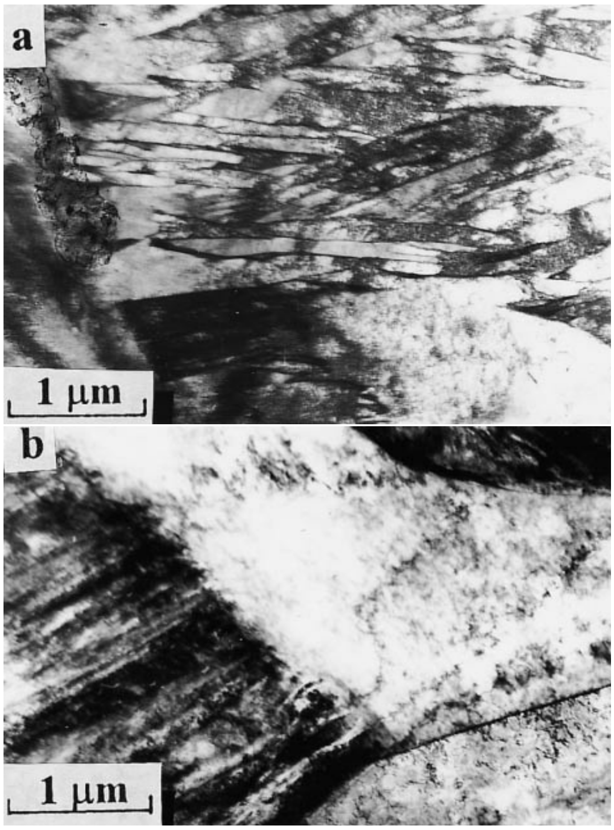
a intercritically annealed at 780°C followed by quenching; *b* intercritically annealed at 780°C, rolled 30%, followed by quenching (TMT route 1)

2 Optical micrographs of steel after given treatment



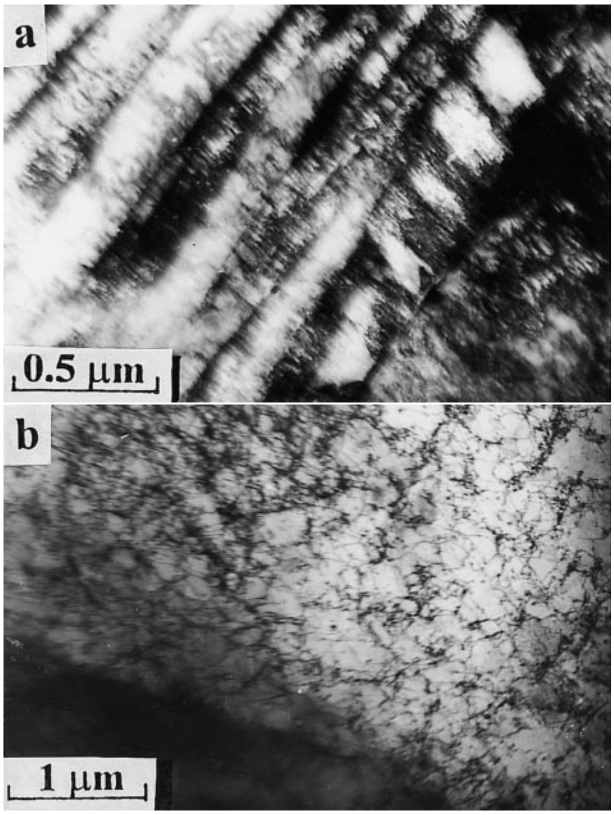
a intercritically annealed at 840°C followed by quenching; b intercritically annealed at 840°C, rolled 30%, followed by quenching (TMT route 1)

3 Optical micrographs of steel after given treatment



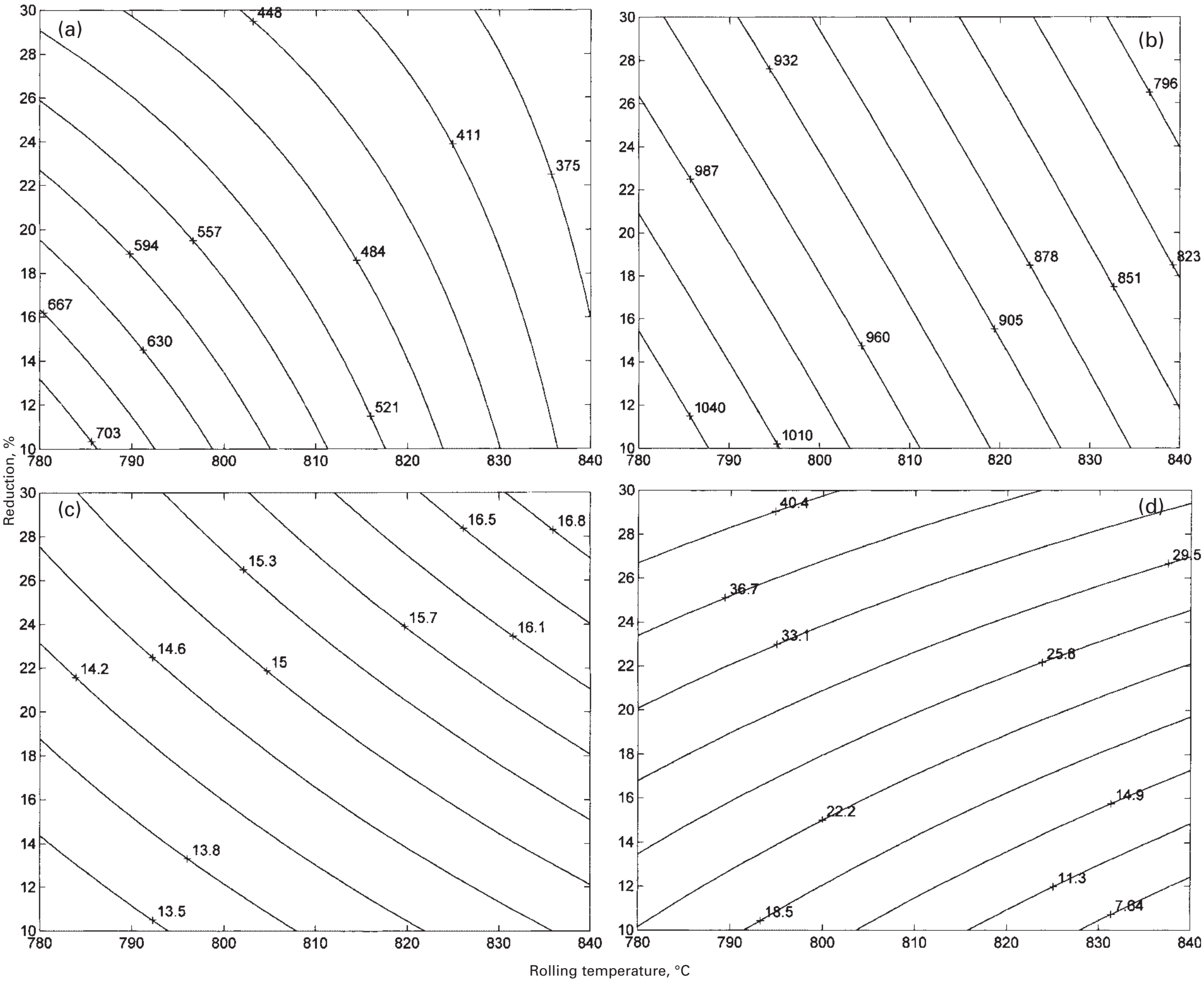
a intercritically annealed at 780°C followed by quenching; b intercritically annealed at 780°C, rolled 30%, followed by quenching (TMT route 1)

4 Transmission electron micrographs of steel after given treatment



a intercritically annealed at 840°C followed by quenching; b intercritically annealed at 840°C, rolled 30%, followed by quenching (TMT route 1)

5 Transmission electron micrographs of steel after given treatment



a yield stress, MPa; *b* tensile strength, MPa; *c* elongation, %; *d* ferrite, %

6 Isoproperty lines for given quantity (TMT route 1)

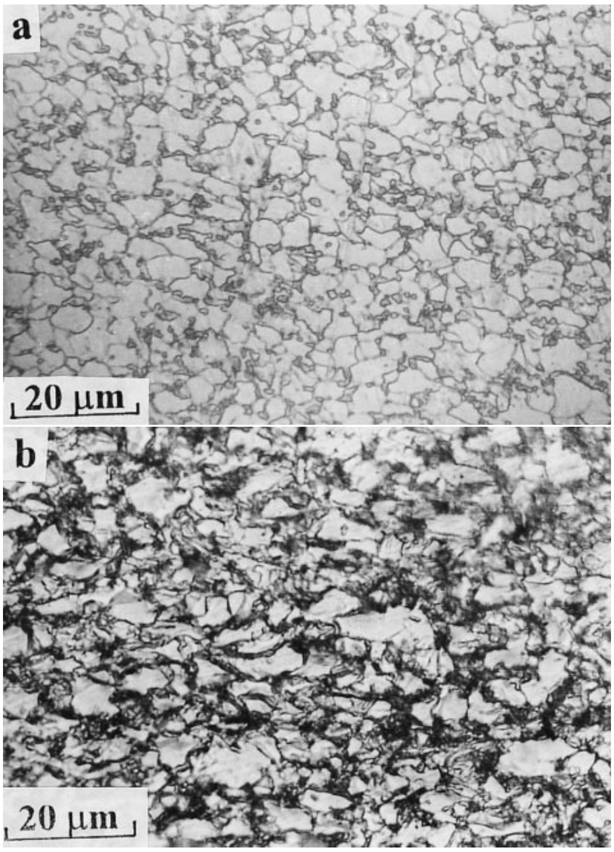
After removing the insignificant coefficients, the accuracy of the equations was checked by a Fisher F test at the 95% confidence level. It was found that within the range of variation of the variables, the equations can predict the properties accurately.[20–21]

The validity of the equations was checked by performing random experiments in the range of variation of temperature of rolling and relative deformation. Table 3 gives comparison between the calculated values of properties obtained from equations (1)–(3) and the experimentally obtained values by performing random experiments. The experimental values given in Table 3 are the average results of three experiments performed with random treatment combinations.

Examination of the results indicate there is a close match between the properties obtained by performing random experiments with those calculated from the respective regression equations by inserting the reduced values of the parameters corresponding to the random experiments in the respective equations. The close matching of the experimental results with those of the calculated ones indicates that the equations are quite accurate within the range of variation of variables.

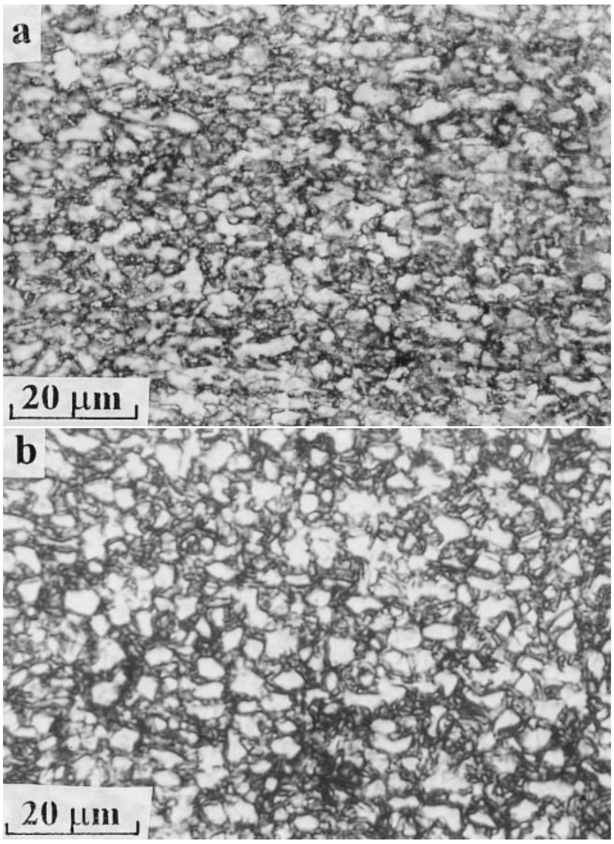
Equation (1) explains the effect of simultaneously varying the temperature of rolling from 780 to 840°C and the relative deformation from 10 to 30%. In equation (1), for yield stress, the coefficient of X_1, i.e. the rolling temperature is $-130{\cdot}5$ and the coefficient X_2, i.e. the percentage deformation is $-70{\cdot}5$. The negative coefficients indicate that increasing the values of these parameters above base level decreases yield stress values. This is attributed to the strain induced nucleation of ferrite, thus decreasing the yield stress. This feature is revealed in Figs. 2–6. Figure 2 shows optical microphotographs of specimens with 0 and 30% deformation at 780°C. It can be seen that with 30% deformation more ferrite is formed than with 0% deformation. Figure 3 shows a similar effect for specimens deformed 0 and 30% at 840°C. Here also the effect of the deformation on nucleating a greater amount of ferrite can be seen. However, the amount of ferrite formed in the case at 840°C is less than that formed in the case at 780°C (compare the micrographs shown in Figs. 2*b* and 3*b*). The same features are revealed in the transmission electron micrographs. Figure 4 shows the amount of ferrite and martensite obtained by deforming the steel with 0 and 30% deformation at 780°C. Both ferrite and martensite are dislocated. It is evident that for the same deformation temperature the area of ferrite observed in the case of deformed steel is larger in comparison to the steel without deformation. Figure 5 shows the transmission electron micrographs of specimens with 0 and 30% deformation at 840°C. Figure 5*b* shows dislocated ferrite in the case of 30% deformed steel at different regions while no such region was observed in the TEM structure of 0% deformed steel (Fig. 5*a*). In general, as observed from Figs. 4*b* and 5*b*, the ferrite region was larger for 30% deformed steel at 780°C in comparison to 30% deformed steel at 840°C.

Mano *et al.*[27] showed that in a dual phase steel containing silicon and chromium the 'ferrite start' curve of the continuous cooling transformation diagram shifted towards the left. The formation of ferrite was accelerated as a result of deformation. Other authors[13–18] found the same effect in



a intercritically annealed at 780°C followed by quenching; b intercritically annealed at 780°C, rolled 30%, followed by quenching (TMT route 2)

7 Optical micrographs of steel after given treatment



a intercritically annealed at 840°C followed by quenching; b intercritically annealed at 840°C, rolled 30%, followed by quenching (TMT route 2)

8 Optical micrographs of steel after given treatment

similar Si–Cr–Mo dual phase steels. This may be attributed to the increased dislocation density, which facilitates 'pipe diffusion', thus enabling partitioning of solute atoms from austenite to ferrite and resulting in more ferrite formation. Since in the present case the deformation has been carried out below the Ac_3 temperature, there is a chance of retention of a greater number of dislocations and therefore formation of ferrite is better facilitated because of the above mechanism.

Equation (2) shows that TS decreases as shown by the negative coefficients for temperature of rolling X_1 ($-102{\cdot}5$) and percentage deformation X_2 ($-47{\cdot}5$). These are also attributed to the strain induced nucleation of ferrite and thus the decreasing influence on TS. However, comparison of the coefficients of X_1 in equations (1) and (2) reveals that the coefficients for YS are more negative than the corresponding values for TS. This is because YS is more sensitive to substructure and structure than the TS.

The interaction coefficients, i.e. the coefficients of X_1X_2 in equations (1) and (2) are positive and the values are lower than the coefficients of either X_1 or X_2. Thus, the combined effect of the variables is to enhance the properties in contrast to the main effect of the variables. The coefficients of X_1 and X_2 in equation (3) for relative elongation are $+1$ and $+1{\cdot}05$ respectively. Thus elongation is increased by increasing the deformation temperature and relative deformation over the base level. This is again attributed to the strain induced ferrite nucleation, which is a ductile phase.

In order to quantify the effect of temperature and amount of deformation on the volume fraction of ferrite formed, data from Table 2 were used to form the following regression equation

$$\text{ferrite}/\% = 26 - 7X_1 + 13X_2 - 2X_1X_2 \quad . \; . \; . \; . \; . \quad (4)$$

While increasing austenitising temperature above 810°C reduces the volume fraction of ferrite (i.e. yields a negative coefficient of X_1), for each 10% deformation increase between 10 and 30%, the volume fraction of ferrite is increased by 13 times (coefficient of X_2). The combined effect of temperature and deformation, i.e. the coefficient of X_1X_2 does not show significant contribution compared to the effect of these factors separately.

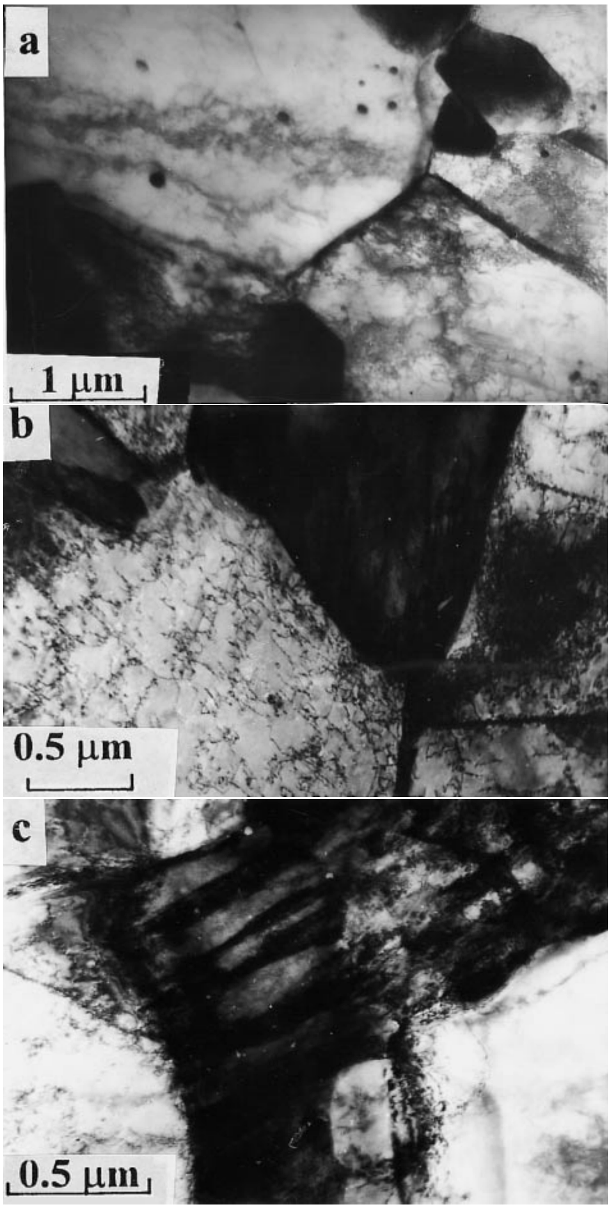
Using equations (1)–(3), isoproperty lines were plotted between temperature of rolling and relative deformation. (*see* Fig. 6*a–c*). The decoding was done by using equations (1)–(3). A similar plot was constructed using equation (4) for isopercentage ferrite against different combinations of rolling temperature and relative deformation (*see* Fig. 6*d*). These plots can be used for selecting operating variables for obtaining different combination of properties.

TMT ROUTE 2

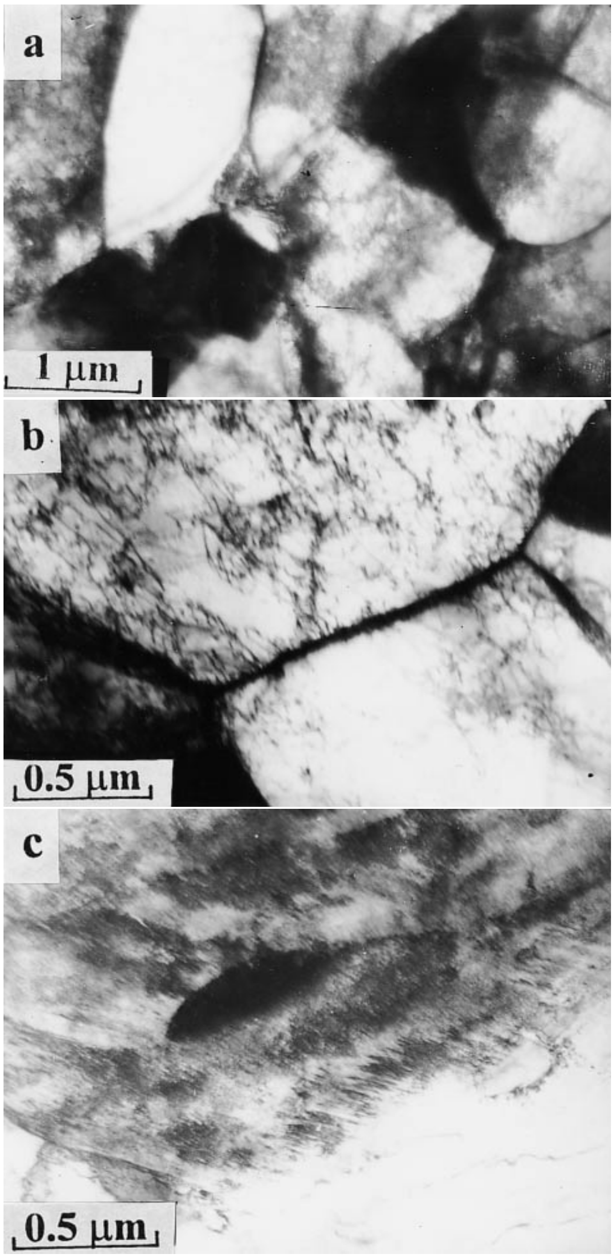
Table 4 shows the response obtained as a result of deformation using various treatment combinations. Each result is an average of three separate experimental results

Table 4 Treatment combinations and resulting mechanical properties and percentage martensite (TMT route 2)

Treatment combination	Rolling temperature X_1	T, °C	Deformation X_2	d, %	YS, MPa	TS, MPa	e, %	Martensite, %
1	−1	780	−1	10	356	820	21	28
2	+1	840	−1	10	416	890	18	42
3	−1	780	+1	30	404	870	17·5	40
4	+1	840	+1	30	430	910	16·5	55



9 Transmission electron micrographs of steel intercritically annealed at 780°C followed by quenching (TMT route 2)



10 Transmission electron micrographs of steel intercritically annealed at 780°C, rolled 30%, followed by quenching (TMT route 2)

carried out at a particular combination of deformation temperature and the amount of deformation.

From the data of Table 4, regression equations were developed between YS, TS, *e*, and the process variables

$$\mathrm{YS/MPa} = 401 + 21X_1 + 16X_2 - 8X_1X_2 \quad (5)$$

$$\mathrm{TS/MPa} = 872{\cdot}5 + 27{\cdot}5X_1 + 17{\cdot}5X_2 - 7{\cdot}5X_1X_2 \quad (6)$$

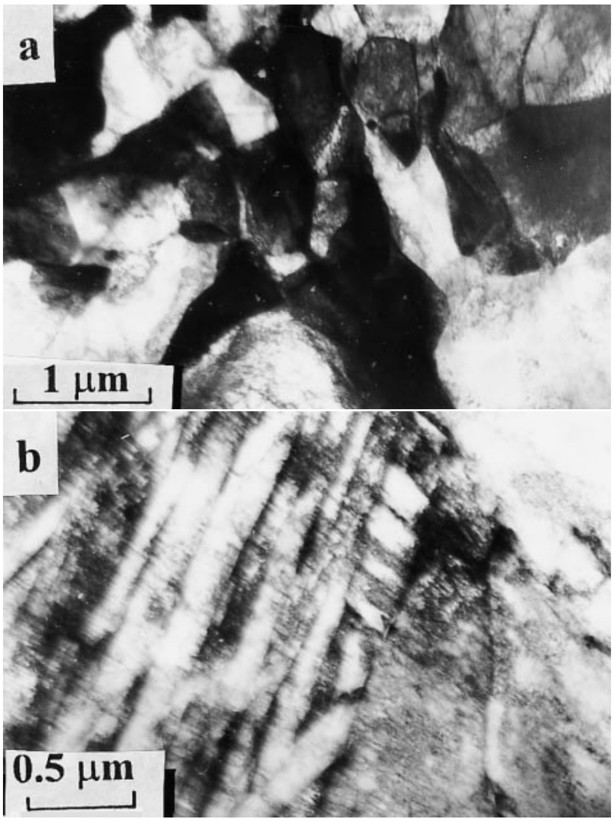
$$e/\% = 18{\cdot}25 - X_1 - 1{\cdot}25X_2 + 0{\cdot}5X_1X_2 \quad (7)$$

where X_1 and X_2 are defined above.

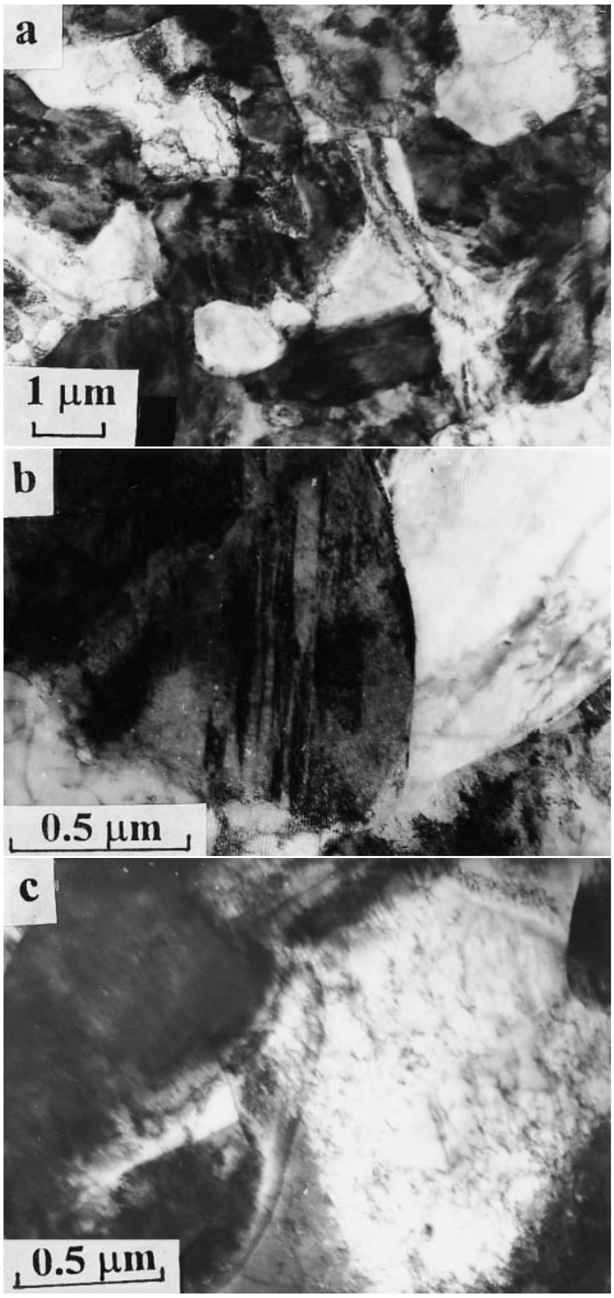
The adequacy of the equations was checked statistically by carrying out a *t* test and *F* test simultaneously at the 95% confidence level.[20–21] The validity of the equations was checked by performing random experiments in the range of the variables, as for the case of TMT route 1. Table 5 compares the calculated values of properties obtained from equations (5)–(7) and the experimentally obtained values by performing random experiments. The

Table 5 Comparison of mechanical properties calculated from equations (4)–(6) and values from random experiments (TMT route 2)

Random experiments		YS, MPa		TS, MPa		*e*, %	
Temperature, °C	Deformation, %	Equation (4)	Experimental	Equation (5)	Experimental	Equation (6)	Experimental
810	20	401	398	872	880	18·25	18·00
780	20	380	374	845	850	19·25	18·60
840	20	422	420	900	900	17·25	17·00
810	10	385	382	855	860	19·45	19·00
810	30	417	423	890	900	17·05	17·00



11 Transmission electron micrographs of steel intercritically annealed at 840°C followed by quenching (TMT route 2)



12 Transmission electron micrographs of steel intercritically annealed at 840°C, rolled 30%, followed by quenching (TMT route 2)

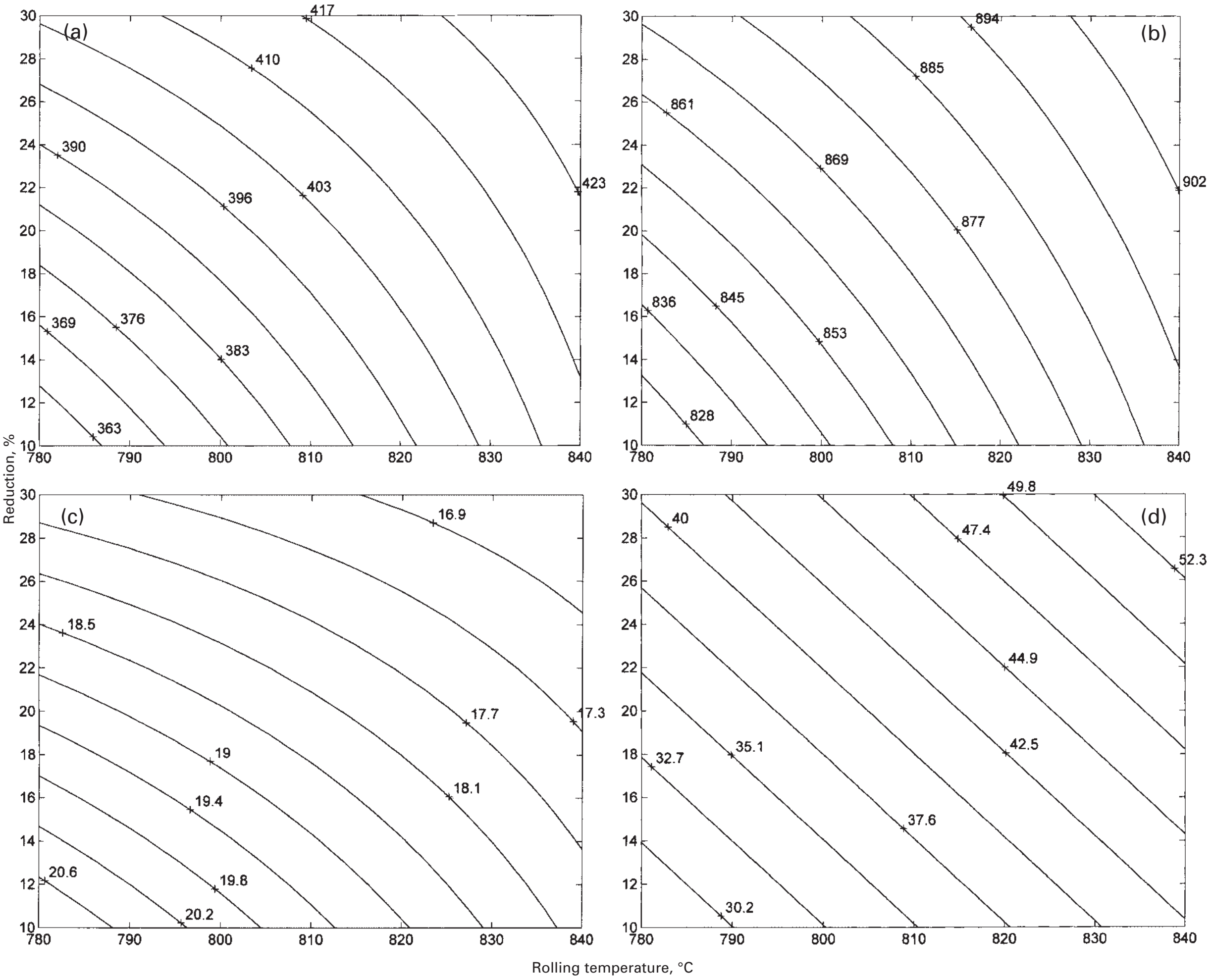
experimental values shown in Table 5 are average results of three experiments performed at the random treatment combinations.

By examining the results of Table 5, it can be observed that the calculated values and the values obtained by performing the corresponding experiment match well. These calculations are made by inserting the reduced TMT values for the particular random experiment. The close matching of the results indicate that the equations are accurate within the range of variation of variables.

It is observed from equation (5) for YS that the coefficient of temperature of deformation X_1 is $+21$ while that for relative deformation X_2 is $+16$. The positive sign of these regression coefficients is indicative of the fact that increases in rolling temperature or relative deformation above the base level result in an increase in the yield stress. Increases in the rolling temperature as well as the deformation in the intercritical range resulted in an increased amount of martensite which made a positive contribution to YS. These features are shown in Figs. 7–13. Figure 7 shows the optical micrographs of specimens with deformation of 0 and 30% at 780°C through TMT route 2. The effect of formation of a greater amount of martensite can be observed in Fig. 7*b* as compared with Fig. 7*a*. Figure 8 shows the effect of 0 and 30% deformation at 840°C. Here also the effect of deformation in forming a greater amount of martensite can be observed. Figure 9 shows the features of ferrite and martensite in the specimens with 0% deformation at 780°C. In these TEM micrographs the darker regions represent martensite areas while brighter regions with dislocation tangles represent ferrite. In contrast, Fig. 10 shows more martensite as a result of 30% deformation at 780°C. Figure 11 shows the effect of 0% deformation at 840°C. Figure 12 shows the effect of 30% deformation at 840°C revealing a martensitic area. However the amount of martensite in the case of 840°C is more than that in the case of 780°C (compare optical micrographs shown in Figs. 7 and 8 from larger field of view compared with TEM micrographs with higher magnification).

Garcia and De Ardo[28] studied the formation of austenite in 1·5 wt-%Mn steel. They observed that the kinetics of austenite formation in such steels is slow but is accelerated by cold working. Deformation enhances the kinetics of austenite formation due to the extra nucleating centres developed by deformation during heating in the intercritical region. As the ferrite in the intercritical region is deformed, it generates more substructure thereby causing formation of strain induced austenite. Thus a greater amount of martensite is observed in the optical micrographs.

In equation (5), the relative contribution of the coefficient of X_1, i.e. the intercritical rolling temperature $(+21)$ is more than that for X_2, i.e. the relative deformation $(+16)$. This is explained by the fact that austenite formation is influenced more by the increasing intercritical annealing



a yield stress, MPa; *b* tensile strength, MPa; *c* elongation, %; *d* martensite, %

13 Isoproperty lines for given quantity (TMT route 2)

temperature above the base level than by the increasing amount of deformation in intercritical region.

In equation (6) for TS, the coefficient of X_1, i.e. the intercritical rolling temperature (+27·5) and that of X_2, i.e. relative deformation (+17·5) have similar effects as those in equation (1) for YS.

The interaction coefficients of X_1X_2 in both equations (5) and (6) are quite small in comparison with those of X_1 and X_2.

In equation (7), for the coefficients of X_1 and X_2 are negative indicating that increasing the intercritical annealing temperature as well as the relative deformation over the base level decreases the ductility. This is attributed to a greater amount of martensite forming in the steel.

By treating the data for percentage of martensite in the design matrix (Table 4), the quantitative effect of the temperature and amount of deformation on the volume percentage of martensite is shown in the following regression equation

$$\text{martensite}/\% = 41{\cdot}25 + 7{\cdot}25X_1 + 6{\cdot}25X_2 \quad . \quad . \quad . \quad . \quad (8)$$

This equation shows that increasing the austenitising temperature above 810°C increases the amount of martensite in the matrix (the coefficient of X_1 being +7·25). Similarly, increasing the extent of deformation above 20%, results in an increase in the amount of martensite (the coefficient of X_2 being +6·25). There is no complex interaction between the temperature of deformation and the amount of deformation as revealed by the absence of any coefficient of X_1X_2.

Using equations (5)–(7) isoproperty lines (Fig. 13*a–c*) were plotted between rolling temperature and relative deformation as natural values. The decoding was carried out using equations (5)–(7). A similar plot was constructed using equation (8) for isopercentage martensite against different combinations of rolling temperature and deformation (*see* Fig. 13*d*). These plots can be used for selecting operating variables for obtaining different combination of properties by adjustment of microstructural constituents.

The regression equations developed in the present work, give some ideas about the quantitative effect of thermomechanical treatment on this steel. These equations can be exploited to maximise the strength properties, while keeping relative elongation as a constraint.[26]

Conclusions

1. Deformation of austenite (TMT route 1) in the intercritical range causes formation of more ferrite in the duplex microstructure produced by quenching.

2. Increasing the deformation temperature and the amount of deformation in the intercritical annealing temperature range resulted in lowering of the yield stress and tensile strength and lowers the yield stress to the tensile strength.

3. Deformation of ferrite and pearlite aggregate in the intercritical annealing temperature causes formation of more martensite due to quenching of TMT material in route 2.

4. The validity of the equations was checked and they were found to be accurate over the range of variables considered.

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