On the Recrystallization Characteristics and Kinetics of Two High-Si DQ&P steels

M C Somani¹, D A Porter¹, L P Karjalainen¹, P Kantanen¹, J Kömi¹, R D K Misra²

¹Materials and Mechanical Engineering, Centre for Advanced Steels Research, University of Oulu, 90014 Oulun yliopisto, Finland
²Dept. of Metallurgical, Materials and Biomedical Engineering, University of Texas at El Paso, El Paso, TX 79968, USA

Email: mahesh.somani@oulu.fi

Abstract. In the direct quenching and partitioning (DQ&P) process to develop tough, ultrahigh-strength steel, the hot rolling stage involving deformation and recrystallization between successive passes affects the state of the austenite prior to the quenching and partitioning step. Therefore, in order to design appropriate thermo-mechanically controlled rolling processes, the recrystallization characteristics of two experimental high-Si DQ&P steels, with compositions (in wt.%) 0.3C-1Si-2Mn-1Cr and 0.25C-1.5Si-3Mn, were determined using the stress relaxation technique on a Gleeble thermomechanical simulator and modelled in terms of the effects of temperature, strain, strain rate and initial grain size. Data analysis resulted in the estimation of the powers of strain (-2.8 and -2.4) and strain rate (-0.23 and -0.14) and the recrystallization activation energies (303 and 289 kJ/mol) for the 0.3C-1Si-2Mn-1Cr and 0.25C-1.5Si-3Mn steels, respectively. This suggests that increasing the contents of Mn and Si made the recrystallization kinetics less sensitive to strain, strain rate and temperature. New regression equations derived to describe the recrystallization kinetics can be used in the design of the rough rolling part of thermomechanical processing.

1. Introduction

Conventionally, quenching and tempering is used to obtain high-strength structural steels with good impact toughness, but with limited strain hardening capacity. The novel concept of quenching and partitioning (Q&P) has been proposed as a potential processing route for achieving desired martensite-austenite microstructures and improving the balance of elongation to fracture and tensile strength for advanced high-strength steels [1, 2]. Based on the principles of the Q&P process, a novel processing route comprising thermomechanical rolling followed by direct quenching and partitioning (TMR-DQP) has been developed with the aim of achieving yield strength ≥1100 MPa combined with good ductility and impact toughness [3, 4, 5].

In order to design appropriate thermo-mechanically controlled rolling processes of the DQ&P steels, it was considered important to characterize the static recrystallization (SRX) behavior in the form of semi-empirical relationships. SRX kinetics have been studied using a fractional softening approach to determine the time for 50% recrystallized fraction (t₅₀) in accord with the modeling performed by Somani et al. [6, 7] using the empirical relation:
where $A$ is a material constant, $\varepsilon$ is strain, $\varepsilon'$ is strain rate, $Q_{\text{app}}$ is the apparent activation energy of recrystallization, $d$ is grain size, $R$ is the gas constant and $T$ is absolute temperature. The material dependent constants $p$, $q$ and $s$ describe the powers of the strain, strain rate and the grain size, respectively. Using $t_{50}$ in combination with an Avrami-type equation, the recrystallized fraction can be predicted as a function of temperature and time. Data for SRX of high-Mn TWIP type austenitic steels have been published [8, 9], but practically no SRX data are available for medium-Mn (3-10% Mn) steels, except a few observations made by Grajcar et al. [10, 11] for 3% and 5% Mn steels and hot deformation and the dynamic recrystallization study of binary Fe-Mn alloys by Cabanas et al. [12]. Somani et al. [6, 7, 13] developed a unique linear regression model that is able to predict satisfactorily the SRX kinetics of common carbon steel grades including microalloyed steels and also several special steel grades. In the course of the development of this model, the influence of alloying with Mn in the range 0.02–2% and Si up to 1.5% alloying was considered by including the instances of a couple of dual-phase and TRIP steels.

In this study, the SRX characteristics of two Si-alloyed Q&P steels with the compositions (in wt.%): 0.3C-2Mn-1Cr-1Si and 0.25C-3Mn-1.5Si were evaluated and kinetics modelled using a fractional softening approach together with metallography to determine the time for 50% recrystallization as a function of strain, strain rate, grain size and temperature by applying SRX tests using a Gleeble 3800 thermomechanical simulator, similar to the regression modelling performed previously by Somani et al. [6, 7] for various steels.

2. Materials and experimental
The chemical compositions of the Si-alloyed C-Mn steels intended for DQ&P processing are given in table 1. Both were procured from OCAS, Belgium. Bars of 11x10 mm cross section cut from the hot rolled plates of the two steels were homogenized at 1250 °C for 2 hours prior to preparing the specimens for stress relaxation testing.

| Steel code     | C    | Si   | Mn  | Al   | Cr |
|---------------|------|------|-----|------|----|
| 2Mn-1Si-1Cr   | 0.3  | 0.99 | 1.9 | 0.007 | 1.0 |
| 3Mn-1.5Si     | 0.25 | 1.45 | 2.9 | 0.024 | 0.01 |

Table 1. Chemical Compositions of the High-Si DQ&P Steels

Cylindrical specimens of φ8 x 10 mm were machined for axisymmetric hot compression testing on a Gleeble 3800 thermomechanical simulator. The specimens were heated at a rate of 10 °C/s to the reheating temperatures of 1200 °C and 1250 °C for the 2Mn-1Si-1Cr and 3Mn-1.5Si steels, respectively, held for 2 minutes, followed by cooling at 2 °C/s to the deformation temperature and then compressed up to a prescribed strain after stabilizing the temperature for 15 s at the deformation temperature. Stress relaxation tests were carried out in the temperature range 850–1100 °C and 900–1200 °C for 2Mn-1Si-1Cr and 3Mn-1.5Si steels, respectively. Both the strain and strain rate were suitably varied at specific temperatures in order to achieve a broad range of deformation parameters for evaluating the SRX characteristics and kinetics of the two steels. For the 2Mn-1Si-1Cr steel, these were strain range 0.125-0.4 and strain rates 0.01-5 s⁻¹ at 1000 °C, and for the 3Mn-1.5Si: 0.125–0.3, 0.001–2 s⁻¹ at 1050 °C.

Austenite grain size was measured using the linear intercept method on specimens directly quenched from the reheating temperature with a water spray and etched in saturated picral/teepol solution. A few segregation bands, possibly Mn-rich regions, were noticed in the 3Mn-1.5Si steel, despite the homogenization treatment given prior to hot rolling and also the reheating at the higher temperature of 1250 °C in the Gleeble simulator. Some specimens were reheated at different temperatures prior to stress relaxation in order to produce relatively coarser or finer grain structure to understand the influence of grain size on SRX kinetics and to check the validity of the empirical equations for fractional softening.
3. Results and discussion

3.1. Flow stress behaviour

Typical examples of true stress – true strain curves obtained on the 2Mn-1Si-1Cr steel compressed to a true strain of ~0.2 at a constant true strain rate of 0.1 s\(^{-1}\) in the temperature range 850–1100 °C prior to stress relaxation is shown in figure 1. The flow stress behaviour displayed the presence of work hardening and dynamic recovery at all deformation temperatures. A similar flow stress behaviour is also observed in the case of 3Mn-1.5Si steel. Subsequent stress relaxation following hot compression should exhibit static recovery and recrystallization processes, except presumably at 1100 °C in the case of 2Mn-1Si-1Cr steel and at 1200 °C in the case of 3Mn-1.5Si steel, where the critical strain \(\varepsilon_c\) (≈ 0.8 times peak strain \(\varepsilon_p\) [14]) for the initiation of dynamic recrystallization (DRX) might have been exceeded, resulting in metadynamic (MDRX) recrystallization.

The flow stress behavior of samples tested at higher strains and lower strain rates were also carefully examined in order to exclude possible MDRX cases from the modelling of the fractional softening equations for SRX. There are no significant differences between the two steels in respect of flow stress levels. Similar observations were made for 3%Mn and 5%Mn steels tested in the range 850–1150 °C by Grajcar et al. [10, 11].

![Figure 1](image1.png)

**Figure 1.** True stress – true strain curves obtained on 2Mn-1Si-1Cr steel following hot compression to 0.2 strain at 0.1 s\(^{-1}\) in the temperature range 850–1100 °C.

![Figure 2](image2.png)

**Figure 2.** Typical stress relaxation curves obtained on 2Mn-1Si-1Cr steel specimens deformed to a true strain of 0.2 at 0.1 s\(^{-1}\).

3.2. Stress relaxation behaviour

Typical stress relaxation curves for the 2Mn-1Si-1Cr steel following the compression tests shown in figure 1, are presented in figure 2. The initial and final linear stages on the curves correspond to the occurrence of static recovery (SRV) and the intermediate fast drop in the stress level is typical of SRX or MDRX process [15]. Stress relaxation curves were carefully analyzed to determine the kinetics of the SRX as a function of deformation parameters. Examples of recrystallized fraction vs. time curves fitted with the sigmoidal Avrami-type curves for the 3Mn-1.5Si steel are shown in figure 3, illustrating the effect of temperature on the kinetics of SRX.

3.3. SRX behaviour and apparent activation energy of recrystallization (\(Q_{app}\))

Referring to figure 2, the stress relaxation curves of the 2Mn-1Si-1Cr steel reveal the influence of temperature on the SRX kinetics following deformation to 0.2 strain at different temperatures in the range 900 – 1100 °C in steps of 50 °C. Complete softening was obtained in most cases, except at the lower temperatures (≤ 900 °C). The stress relaxation curves for the 3Mn-1.5 Si steel also showed that the softening was complete at most temperatures, except at 900 °C, where only partial recrystallization occurred. Similarly, the grain size effect on SRX rate was clearly revealed by the stress relaxation...
technique. For instance, in the case of 3Mn-1.5Si steel samples deformed at 1050 °C to 0.2 strain at 0.1 s\(^{-1}\), the recrystallization rate was greatly accelerated in the case of fine grained structure (80 µm; \(t_{50} = 2.6\) s) in comparison with that of the coarse-grained structure (480 µm; \(t_{50} = 12\) s).

The temperature dependence of the SRX kinetics for the 2Mn-1Si-1Cr and 3Mn-1.5Si steels are shown in figure 4, where \(t_{50}\) times estimated at different temperatures for specimens deformed to 0.2 strain at 0.1 s\(^{-1}\) are plotted against the inverse absolute temperature. The slope of the plots was used to estimate the apparent activation energy for recrystallization (\(Q_{app}\)). When doing this, the cases of incomplete recrystallization (at low temperatures) and MDRX (at high temperatures) were carefully excluded. The estimated \(Q_{app}\) values for the 2Mn-1Si and 3Mn-1.5Si steels were about 225 and 241 kJ/mol, suggesting that an increase in Si and Mn contents increases \(Q_{app}\). The observed \(Q_{app}\) for the two steels fall in the 177–283 kJ/mol range of values reported for other steels, for example, C-Mn 184 kJ/mol [6], 0.2C-2Mn-0.6Cr-0(1.5)Si 193–227 kJ/mol [16], C-Mn-Nb 230 kJ/mol [6] and Type 304 stainless steel 283 kJ/mol [17].

Using a constant \(Q_{def}\) of 340 kJ/mol [6, 13] for both the 2Mn-1Si and 3Mn-1.5Si steels gives the activation energy of SRX (\(Q_{exc}\)) values of 303 and 289 kJ/mol, respectively. The low value of \(Q_{exc}\) for the 3Mn-1.5Si steel is due to its less negative \(q\) (-0.14 as opposed to -0.23), even though its \(Q_{app}\) was higher than that of the 2Mn-1Si steel, as expected from its higher alloy content.

3.4. Powers of strain and strain rate
The powers of strain (p) and strain rate (q) have been estimated based on the plots of (i) \(t_{50}\) vs. strain at 0.1 s\(^{-1}\) at either 1000 °C for the 2Mn-1Si-1Cr steel or 1050 °C for the 3Mn-1.5Si steel (figure 5), and (ii) \(t_{50}\) vs. strain rate following compression to 0.2 strain at the deformation temperature (figure 6). Referring to figure 5, the powers of strain (p) estimated from the slopes of the line fits were estimated to be about -2.8 and -2.35 for the 2Mn-1Si-1Cr and 3Mn-1.5 Si steels, respectively. The power of strain (p) for the 3Mn-1.5Si steel (-2.35) is slightly lower than that of 2Mn-1.5Si (-2.8). The strain exponent for C-Mn steels has been reported to be in the range -2.5 to -4 [6, 7, 13, 18, 19]. Values between -2 and -3 were measured by the stress relaxation technique for some microalloyed steels [6, 20]. Suikkanen et al. [16] obtained a value of -2.1 for p in the case of a 0.2C-2.0Mn-1.48Si-0.6Cr steel. Hence, no distinct dependence on alloying can be ascertained.

Similarly, the slopes of the linear fits in figure 6 show the powers of strain rate (q), estimated as -0.23 and -0.14 for the 2Mn-1Si-1Cr and 3Mn-1.5 Si steels, respectively. The power of strain rate for a 0.1C-1.2Mn-1.15 Si steel (-0.23) was comparable to that of the 2Mn-1.5Si steel [16]. Lang et al. [21]...
determined the SRX kinetics for a 0.21C-1.48Si-2.04Mn-0.6Cr composition, also tested by Suikkanen et al. [16], and found q to be -0.18, i.e. between the values of -0.23 and -0.14. In comparison, relatively low values of q of -0.11 and -0.12 have been reported for C/C-Mn and Ti steels [6, 7, 22]. For Nb and Nb-Ti and also Mo-steels, q has been estimated to be of the order of -0.23 [6, 7]. In summary, all these values of q fall within a small range (-0.11 to -0.23) indicating a weak dependence of SRX on strain rate irrespective of the steel alloying.

3.5. Fractional softening equations for SRX

Taking the power of grain size to be described by the relation $s = 2.13d^{-0.105}$, together with the above values for the other parameters ($Q_{app}$, p and q) in Equation 1 gives the constant A for the two steels. The SRX rate, therefore, can be reasonably described by the following SRX equations:

$$t_{50} = 1.2 \times 10^{-13} e^{-2.8} e^{-0.23} d^{s \exp(225000/RT)}$$

(2)

$$t_{50} = 5.7 \times 10^{-14} e^{-2.35} e^{-0.14} d^{s \exp(241000/RT)}$$

(3)

In order to check the reliability of the equations, a few confirmation experiments were carried out by varying the deformation parameters and/or grain sizes for the two steels as described elsewhere [23]. The results confirmed that the SRX regression equations can be used with reasonable accuracy in designing the thermomechanical processing schedules for the two DQ&P steels.

The $t_{50}$ times for the present steels, estimated with the aid of Equations 2 and 3, are compared with those of other steels elsewhere [23]. The SRX kinetics of the present high-Si steels is quite similar despite the different levels of Si and Mn alloying. The SRX kinetics of high-Si steels have been found to be much slower than C-Mn steels and marginally faster but close to that of 0.03%Nb-microalloyed C-Mn steel as well as an ordinary TWIP steel. An addition of 1–1.5% Si in the high-Si steels seems to have a potent effect, but weaker than that of Nb, in C-Mn steels.

4. Summary and conclusions

The recrystallization characteristics of two experimental high-Si DQ&P steels, viz., 2Mn-1Si-1Cr and 3Mn-1.5Si, have been evaluated using the stress relaxation technique on a Gleeble 3800 thermomechanical simulator. Data analysis resulted in the powers of strain (-2.8 and -2.35) and strain
rate (-0.23 and -0.14) and the apparent (225 and 241 kJ/mol) and recrystallization activation energies (303 and 289 kJ/mol) for the 2Mn-1Si and 3Mn-1.5Si steels, respectively. This suggests that with the increase in the contents of Mn and Si, the SRX kinetics became less sensitive to strain, strain rate and temperature. The differences between the two steels appeared to be small and could be partly due to experimental scatter. Regression equations describing the SRX kinetics of the two steels fit well with the experimental data. Confirmation experiments indicated that the power of grain size suggested earlier for carbon steels was reasonably accurate for these steels, too.

Acknowledgement
The funding of this research activity through the RFCS Grant Agreement RFCSR-CT-2014-00019 is gratefully acknowledged.

References
[1] Speer J G, Edmonds D V, Rizzo F C and Matlock D K 2004 Curr. Opin. Solid State Mater. Sci. 8 219
[2] Li H Y, Lu X W, Li W J and Jin X J 2010 Metall. Mater. Trans. A 41A 1284
[3] Somani M C, Porter D A, Karjalainen L P, Suikkanen P P and Misra D K 2014 Mater. Sci. Forum 783-786 1009
[4] Somani M C, Porter D A, Karjalainen L P, Suikkanen P P and Misra R D K 2015 J. Mater. Today: Proc. 28 631
[5] Somani M C, Porter D A, Kömi J I, Karjalainen L P and Misra D K 2017 Proc. Int. Symp. on New Developments in Advanced High-Strength Sheet Steels (Keystone) (Warrendale: AIST) p 331
[6] Somani M C, Karjalainen L P, Porter D A and Morgridge R A 2003 Proc. Int. Conf. on Thermomechanical Processing: Mechanisms, Microstructure and Control (Sheffield) eds. E J Palmiere, M Mahfouf and C Pinna (Sheffield: The University of Sheffield) p 436
[7] Somani M C and Karjalainen L P 2012 Mater. Sci. Forum 715-716 751
[8] Hamada A S, Somani M C and Karjalainen L P 2007 ISIJ Int. 47 907
[9] Somani M C, Porter D A, Hamada A S and Karjalainen L P 2015 Metall. Mater. Trans. A 46A 5329
[10] Grajcar A and Kuziak R 2011 Adv. Mater. Res. 287-290 330
[11] Grajcar A and Kuziak R 2011 Adv. Mater. Res. 314-316 119
[12] Cabañas N, Akdut N, Penning J and De Cooman B C 2006 Metall. Mater. Trans. A 37A 3305
[13] Somani M C and Karjalainen L P 2004 Mater. Sci. Forum 467-479 355
[14] Sellars C M 1980 Proc. Int. Conf. on Hot Working and Forming Processes (Sheffield) eds. C M Sellars and G J Davies (London: The Metals Society) p 3
[15] Karjalainen L P 1995 Mater. Sci. Technol. 11 557
[16] Suikkanen P P, Lang V T E, Somani M C, Porter D A and Karjalainen L P 2012 ISIJ Int. 52 471
[17] Karjalainen L P, Koskiniemi J A and Liu X D 1995 Proc. 37th MWSP Conf. & Int. Symp. on Recovery and Recrystallization in Steel (Hamilton) XXXIII p 861
[18] Hodgson P D and Gibbs R K 1992 ISIJ Int. 32 1329
[19] Sellars C M 1990 Mater. Sci. Technol. 6 1072
[20] Karjalainen L P, Perttula J, Xu Y and Niu J 1997 Proc. 7th Int. Symp. on Physical Simulation (Tsukuba) p 231
[21] Lang V T E, Suikkanen P P, Somani M C, Porter D A and Karjalainen L P 2011 Proc. Int. Sci. Technol. Conf. on Advanced Metals, Materials and Technologies (St. Petersburg) p 301
[22] Airaksinen K, Karjalainen L P, Porter D and Perttula J 1998 Mater. Sci. Forum 284-286 119
[23] Somani M C, Porter D A, Karjalainen L P, Kantanen P K, Kömi J I and Misra D K 2019 Int. J. Mater. Res. 110 3