The high temperature deformation behaviour of high silicon steels, their continuous cooling to room temperature and the time delay between deformations are important factors for the understanding of their workability. Torsion tests were carried out in two Fe–Si steels with 4.2 and 5.6 wt% Si under continuous cooling to study the influence of the strain and the interpass time on the ordering and non-recrystallisation temperatures by means of the evolution of the mean flow stress (MFS). $^{57}$Fe Mössbauer spectroscopy was used in order to obtain information about the degree and type of ordering in the alloys and to find out its dependence on the applied thermomechanical treatments. Finally, extrapolating the parameters obtained in hot torsion to the rolling mill a suitable schedule for hot rolling was provided in order to guarantee good conditions for further cold rolling.

KEY WORDS: hot workability; torsion test; electrical steel; high silicon steel; Mössbauer spectroscopy; ordering.
rolling can be obtained in order to guarantee good conditions for further cold rolling.\textsuperscript{57}Fe Mössbauer spectroscopy was used to study the type and degree of ordering in the alloys and to find out its dependence on the applied thermo-mechanical treatments.

2. Experimental Procedure

Two experimental alloys with different silicon contents were studied. They were produced on laboratory scale, a third alloy with no Si (ULC-Steel) was used as comparison for some experiments and the chemical composition is shown in Table 1. The Fe–Si alloys were prepared by melting ULC (Ultra Low Carbon) steel and Fe–75\%Si in an induction furnace and after casting they were hot rolled to slabs with 25 mm thickness.

For the hot torsion experiments specimens with a gauge length of \( L=24 \text{ mm} \) in the rolling direction and \( D=2R=6 \text{ mm} \) diameter were prepared from hot rolled slabs. The tests were performed using a computerized torsion machine developed at the Department of Metallurgy and Materials Science at Ghent University, using induction heating. Samples were reheated at 4°C/s until 1 150°C and held at this temperature during 300 s. Multi pass torsion tests were carried out until fracture at a constant strain rate of 0.1 s\(^{-1}\), with interpass times of 5, 10 and 20 s and strains of 0.15, 0.35 and 0.70. After each pass the specimens were cooled at a cooling rate of 4, 2 or 1°C/s, calculated by a constant interpass temperature difference of 20°C and the interpass time. Samples were tested several times to ensure reproducibility of the results, the measured torque (\( T \)) and twist angle (\( \theta \)) were converted to von Misses effective stress (\( \sigma \)) and strain (\( \varepsilon \)) using the equations found in literature\textsuperscript{5}:

\[ \sigma = \frac{3.3T \sqrt{3}}{2\pi R^3} \] ..................................(1)

and

\[ \varepsilon = \frac{\theta R}{L\sqrt{3}} \] ..................................(2)

Once the calculations of \( \sigma \) and \( \varepsilon \) were done it was possible to obtain the MFS to each pass from the flow using the procedure described by Jonas and Borato.\textsuperscript{6}

\[ \text{MFS} = \frac{1}{\Delta F} \int_{\varepsilon}^{\varepsilon_{\text{max}}} \sigma \, d\varepsilon = \frac{\sum_{i=1}^{\text{n}} \sigma_i \Delta \varepsilon}{\varepsilon_{\text{max}} - \varepsilon_{\text{o}}} \] ..................................(3)

Some specimens of both alloys were quenched immediately after the 4th, 9th and 17th pass in order to study the evolution of the microstructure after multi pass torsion in various stages and to perform \textsuperscript{57}Mössbauer spectroscopy to clarify the role of the ordering phenomena on the mechanical behaviour in function of temperature and applied strain.

For the \textsuperscript{57}Mössbauer experiments thin foils were prepared by mechanical grinding down to a thickness of 30–35 \( \mu \text{m} \) in order to obtain good Mössbauer spectra quality. The measurements were carried out in transmission geometry at room temperature calibrating the spectrometer periodically using pure iron foils. A specially designed program was used to fit the spectra. The program introduces 63 Lorentzian-shape sextets corresponding to different configurations for the Fe-atoms in the first and second neighbourhoods denoted by \((x,y)\). \((x,y)\) represents the Fe-atoms with \( x \) and \( y \) Si-atoms on the first and second neighbourhood, respectively. Some relations are imposed between the hyperfine parameters of the different sextets in order to keep consistency. In addition, the site occupancies of the lattice are included as fitting parameters, controlling the areas of the different spectra. From them it is possible to deduce the long range order parameters following the Bragg–Williams model.\textsuperscript{7} Concerning the broadening of the lines, it was supposed to be linear and equal for the 63 sextets. The line area ratios followed a relation \( 3:x:y \), being \( x \) and \( y \) always close to 2 and 1 respectively. The details of the program are published elsewhere.\textsuperscript{8}

All the rolling experiments were physically simulated in the laboratory facilities at Ghent University using a reversible two-high laboratory mill with a maximum rolling force of 3 000 kN and roll diameters of 320 and 345 mm for hot and cold rolling respectively, with a typical sheet width of 250 mm and resistance reheating of slabs. The rolling schedule based on the previous results consists of four steps\textsuperscript{9,10}.

Step I: Reheating of the slabs for 1 h at temperatures between 1 150°C and 1 250°C.

Step II: Hot rolling from 25–30 mm thickness to 1–2 mm thickness in 2, 4 or 7 passes with 30 to 80\% of reduction per pass and finishing at a temperatures higher than 800–900°C.

Step III: Cooling down of the samples after hot rolling in water or at the air, with a cooling rate not less than 400°C/min.\textsuperscript{10}

Step IV: Cold rolling in the deformed state without additional treatment between the hot and cold rolling.

The MFS for rolling were calculated using the equation developed by Maccagno and Jonas\textsuperscript{11}:

\[ \text{MFS} = \frac{P}{(2/\sqrt{3})w w (R (H - h))^{1/2} Q} \] ..................................(4)

Where \( P \) is the rolling load, \( w \) is the width of the sheet, \( R \) is the radius of the rolling mill, \( H \) and \( h \) are the initial and the final thickness, respectively and \( Q \) is a geometrical factor.

\begin{table}[h]
\centering
\caption{Chemical composition (wt\%) of the Fe–Si alloys and ULC steel (balance is Fe).}
\begin{tabular}{cccccccc}
\hline
Alloy & C & Si & Al & Mn & P & S & Ti & N \\
\hline
A & 0.009 & 4.20 & 0.003 & 0.048 & 0.003 & 0.006 & 0.005 & -0.0008 \\
B & 0.004 & 5.63 & 0.009 & 0.089 & 0.003 & 0.006 & 0.013 & - \\
ULC & 0.0019 & - & 0.02 & 0.072 & 0.012 & 0.003 & - & - \\
\hline
\end{tabular}
\end{table}
3. Results and Discussion

3.1. Hot Torsion Tests

Figure 1 shows the flow curves plotted vs. strain for complete multi pass torsion tests of ULC steel, steel A (4.2 wt% Si) and B (5.6 wt% Si) in order to compare the behaviour of an alloy with allotropic transformation and alloys where only order-disorder transformation can be present. When each torsion pass is considered as a separate element of the test, the following observations can be made. At the beginning of each deformation step, a yield point phenomenon is observed: the plastic deformation starts at a slightly higher level than the plateau at which the rest of the deformation occurs. This phenomenon is most probably the consequence of a high strain-rate sensitivity of the steels A and B (4.2 and 5.6 wt% Si, respectively) and is not visible for the ULC material. Absolute and relative values of the peak stress observed in each pass were calculated. The relative values of the peaks remain constant while the absolute values of the peaks follow the increase of the mean flow stress as they are shown in Fig. 2(a). Lakso et al. describe a similar behaviour in compressive stress–strain tests of Fe–Si (0 to 25 at% Si) alloys, linking it with the generation of superlattice dislocations and its interaction due to insufficient thermal energy available at temperatures lower than $T_{ord}$. In our tests the peaks were also observed at temperatures above $T_{ord}$ but with a smaller absolute value.

Comparing the complete torsion test for the Si-steels A and B and the ULC steel, some observations can be made. The strain hardening during each step of the torsion test is much more important for the ULC-steel, especially above the Ar3-temperature, when the steel has the fcc-structure. Below Ar1, the steel is bcc and presents less strain hardening, which is in agreement with the basic concepts of mechanical metallurgy. The steel samples with a high Si-content (4.2 to 5.6 wt% Si) do not present any stress increase in our tests for the torsion deformation mode for temperatures above 1000°C. This was also observed in the hot rolling tests reported later in this paper: the MFS during rolling remain almost constant until a temperature of some 950°C.

A typical dependence of the MFS on inverse temperature is shown in Fig. 2(b) as a plot of the MFS values vs. $1000/T$ (K$^{-1}$). A linear regression makes clear that three different regions can be recognized. The first change in slope can be associated with the temperature for disorder-order transition and is called $T_{ord}$. In region I above this temperature no order is present in the material, there is a random substitutional solid solution (called A2 in literature). When the temperature is lowered under $T_{ord}$, order starts to appear as small regions with a B2- or D0$_3$-type of order. The degree of order may increase at lower temperatures, depending on chemical composition and cooling rate.

The second change of slope does not correspond to a phase transformation, because the chemical composition of both alloys, in which the Si is higher than 1.8 wt% Si, ensures the stability of the ferritic phase and the total absence of phase transformation (first order transformation) in the complete processing temperature range, only order/disorder transformations (second order transformation) could be present. The mentioned second change of slope is associated with the retardation of recrystallisation process: no static recrystallisation of the deformed structure occurs for a given interpass time at temperatures below $T_{nr}$ (the so-
called non-recrystallisation temperature).

For both high-Si steels, the $T_{ord}$ measured by torsion testing is in the range 1 000–1 100°C. According to the equilibrium phase diagram of the Fe–Si-system, this temperature range should be 700–800°C. The range measured by torsion testing would hence be too high for the occurrence of ordered structures of the type B2 or D0$_3$. Nevertheless, it is important to emphasize the fact that the experiments described in this paper are not performed under equilibrium conditions and that the equilibrium phase diagram is not completely applicable. Furthermore, previous experiments in hot rolling gave temperatures in acceptable agreement with the $T_{ord}$ determined by torsion testing.

In Fig. 2(b), region I corresponds to high-temperature deformation in which there is no stress accumulation and the MFS remains almost constant. Furthermore, the yield point phenomenon in this region is weak and full static recrystallisation takes place after the deformation step, with a well developed subgrain microstructure and a grain refinement as shown in Fig. 3(a).

In region II a stress accumulation from pass to pass is observed with decreasing temperature and the MFS increases. The yield point phenomenon starts to appear clearly at the beginning of each deformation step. In this region, the increase of the MFS is not only caused by the temperature decrease, but the gradual appearance of order also interferes with the workability, being this more acute as the Si increases. Similar to region I, the microstructure is characterised by recrystallised grains with a well developed subgrain microstructure. Due to the quenching of the torsion sample from high temperature, some cracks may appear through the grains in the regions I and II (Fig. 3(b)).

Region III corresponds to deformation below $T_{nr}$ and the MFS values increase more steeply when lowering the temperature than in region II. Microstructural analysis shows a highly developed subgrain microstructure with partial or no recrystallisation at all. The recrystallisation delay in the alloy B (higher Si) is more pronounced than in the alloy A (lower Si): the higher degree of order due to the higher Si content retards recrystallisation, on top of the silicon in solid solution. Increasing the silicon content is considered to retard the progress of the recrystallisation at lower temperatures than $T_{ord}$ because of the interaction between the ordering and the recrystallisation processes. Previous work of Ros-Yanez et al. in hot rolled samples of material A and B showed that for annealing at temperatures below $T_{ord}$ the recrystallisation was 300 times slower than for annealing at temperatures above $T_{ord}$. Samples of material B (5.6 wt% Si) can show a recrystallisation 55 times slower than samples of material A (4.2 wt% Si) at 800°C.

Figure 4 shows data of the MFS vs. 1 000/T regarding the effect of the interpass time in the alloy B (5.6 wt% Si). Shorter interpass times correspond to higher cooling rates between the deformation steps (see the experimental procedure). $T_{nr}$ decreases with increasing interpass time, while $T_{ord}$ increases but in a lower extent. The steep increase of the MFS below $T_{nr}$ is not only associated with the temperature decrease, but also with the retardation of recrystallisation due to the appearance of ordering.

The MFS curves for the studied alloys, see Fig. 4, are not especially affected by the interpass time for temperatures above $T_{nr}$. In region I and II the MFS-values are almost coincident and hence the slopes of the MFS vs. 1 000/T curves, for each region remain the same, while for tempera-

![Fig. 3. Evolution of the microstructure of alloy B (5.6 wt% Si) after multipass torsion at various stages. (a) After the 4th step at 1 090°C, (b) after the 9th step at 990°C and (c) after the 17th step at 830°C, cooling rate 4°C/s (5 s interpass time) and pass strain 0.15.](Image)

![Fig. 4. Effect of interpass time on the MFS/inverse pass temperature dependence of steel B (5.6 wt% Si). Strain $\varepsilon=0.15$/pass, strain rate of 0.1 s$^{-1}$. Curves are shifted with respect to the MFS-axis for clarity: 0 MPa for 5 s, 20 MPa for 10 s and 40 MPa for 20 s.](Image)
tures below the $T_{\text{ord}}$ (region III) an increase of the MFS as a function of the increasing interpass time (lower cooling rate) is appreciated, therefore the slope of the MFS vs. 1000/$T$ curves in that region is increased as the cooling rate is decreased.

Figure 5(a) shows how the $T_{\text{ord}}$ for the steel A (4.2 wt% Si) decreases for increasing cooling rates for each of the given strains per pass. The lowest curve is found for a strain of 0.15 per pass. The manifestation of ordering is delayed to lower temperatures because the ordering reaction is diffusion controlled and some undercooling occurs at higher cooling rates. Considering the effect of the strain in each pass, strains higher or lower than 0.35 shift the $T_{\text{ord}}$ to lower values, although at strains of 0.70 the $T_{\text{ord}}$ remains above the value obtained for strains of 0.15, without a systematic variation. Nevertheless, a larger deformation step seems to favour the development of ordering at higher temperatures. Although early works reported the ordering in high Si alloys is very stable in relation with heat treatments,2,10) it has also been observed that ordering effects do not depend only on temperature, but also on the cooling rate and the strain deformation. The higher deformation strain may increase the mobility of the atoms and move the start temperature for ordering to higher temperatures.

Figure 5(b) shows similar graphs for the steel B (5.6 wt% Si), only slightly shifted to higher temperatures: steel with a higher Si-content has a higher tendency to produce order. Similar remarks can be made concerning the effect of the cooling rate and strain per pass as for steel A, but in this case the $T_{\text{ord}}$ at strains per pass of 0.35 and 0.70 are the same.

Figure 6 shows the effect of the interpass time on the $T_{\text{ord}}$ in steel A and B at a strain of 0.15. The $T_{\text{ord}}$ values are always higher for the alloy B with more Si content and the reason is associated to the influence of the higher degree of order in the retardation of the recrystallisation phenomena.3) At the same time the $T_{\text{nr}}$ is lower when a higher interpass time is applied, associated with a lower cooling rate.

3.2. Mössbauer Spectroscopy
In order to study the degree of ordering of the samples during the hot torsion test a water quench was performed after the fourth, ninth and seventeenth pass at temperatures of 1 090, 990 and 830°C, respectively. Figure 7 shows the spectra with their corresponding fittings for the alloy B with 5.6 wt% Si. From the Mössbauer spectra analysis long range order parameters were directly obtained as explained in the experimental procedure (Sec. 2).

The Mössbauer analysis shows that there is always some order present in the samples since no spectra with random distribution of the atoms could be fitted. Besides, both alloys appear to have a mixture of Fe$_{15}$Si and D0$_3$ orderings, which is in agreement with literature.14) The results of the Fe$_{15}$Si and D0$_3$ LROP's (long-range order parameters) are shown in Fig. 8 as a function of the quenching temperature.
For comparison, the values for the as-cast material (starting material) are also shown and the hot rolled material with slow cooling in the oven at 0.5°C/min (high degree of order). Although changes in degree of order seem to be slight, there is an apparent increase as the temperature decreases, especially for the D0₃ LROP.

The Fe₁₅Si LROP increases at 990°C while at 830°C it decreases slightly, probably because the influence of the deformation. Further research has to be done to throw light on this deviation from the linear behaviour, although this parameter is less significant for the processing of the alloy, compared to the D0₃ one.

It can be concluded that the increase of ordering during cooling appears at high temperatures between 1 000 and 1 100°C, which is in agreement with the experimental hot torsion results and from previous publications. Nevertheless the deformation state was not the same in all samples with a consequent additional effect on the spectra, which were measured in the longitudinal section, indicating that there is a gradient in deformation depending on the distance from the centre to the edge.

3.3. General Procedure for Hot Rolling of High Si

Results of the hot torsion tests, previous work and literature show that decreasing the Si-content and increasing the pass strain and strain rate decreases $T_{nr}$, while increasing the cooling rate between deformations increases the $T_{nr}$. Decreasing the cooling rate and the Si-content increases the ordering temperature $T_{ord}$, while increasing the pass strain up to 0.35 increases $T_{ord}$, although higher strains seem to reduce $T_{ord}$.

Regarding the above mentioned, apart from initial microstructure and chemical composition, the cooling rate between deformations can be taken as the most important parameter in processing high Si. The cooling rate during rolling will depend on numerous factors as, type of rolling, initial slab dimensions, preheating temperature, reduction per pass, etc.

The data of $T_{ord}$ and $T_{nr}$ from the torsion test were expressed as a function of the logarithm of the cooling rate versus $1000/T$. Linear regressions on these data allowed an extrapolation of $T_{ord}$ values for hot rolling, ranging between 952 and 1 016°C for alloy A and between 995 and 1 030°C for alloy B. Unfortunately during the hot rolling experiments the change in the behaviour of MFS vs. $1000/T$ plot was not observed. On the other hand $T_{nr}$ was estimated to be between 840 and 864°C for alloy A and between 927 and 1 027°C for alloy B. In order to corroborate the results of the torsion test a hot rolling was performed applying 0.15 strain per pass at a cooling rate between passes of 8°C/s. These results were compared with the torsion data revealing that the values of $T_{nr}$ calculated for alloy B were in good agreement, but a high deviation was found for the $T_{ord}$ values of alloy A. Experimental values and the recalculated from the linear regression, are shown in Table 2.

The following hot rolling procedure for these alloys was studied:

**Step I:** Reheating temperatures of 1 150°C offer better initial conditions for the hot rolling because of the extremely high degree of oxidation at 1 250°C.

**Step II:** The hot rolling can be applied in 7, 4 or 2 passes with 30 to 80% of reduction per pass, see Table 2, but finishing at temperatures higher than 950°C is desired, in

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**Table 2.** Rolling schedules for alloy A (4.2 wt% Si) and B (5.6 wt% Si), with: $h_0$, initial thickness, $h_f$, final thickness in hot rolling (in mm), IRT, initial rolling temperature, FRT, final rolling temperature, $T_{nr}$, no-recrystallisation temperatures experimental and calculated, respectively.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>$h_0$ (mm)</th>
<th>$h_f$ (mm)</th>
<th>IRT (°C)</th>
<th>Inter pass cooling rate (°C/sec)</th>
<th>FRT (°C)</th>
<th>$T_{nr}$ Exp. (°C)</th>
<th>$T_{nr}$ Calc. (°C)</th>
<th>Cooling rate from Hot Rolling (°C/min)</th>
<th>Remarks</th>
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<tbody>
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<td>A</td>
<td>30-30-30-30-30-30-30</td>
<td>24</td>
<td>1140</td>
<td>11</td>
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<td>950</td>
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<td>830</td>
<td>980</td>
<td>1013</td>
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<td></td>
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<td>1150</td>
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<td>940</td>
<td>959</td>
<td>963</td>
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<td>790</td>
<td>939</td>
<td>940</td>
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<td>0.38</td>
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<tr>
<td>B</td>
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<td>20</td>
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<td>970</td>
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<td>982</td>
<td>1.7</td>
<td>2100</td>
</tr>
</tbody>
</table>

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and where the increase of the MFS is significant, as it is in some cases the time delay prior to cold rolling. Processing seem to be the cooling rate after hot rolling and cooling kinetics and the changes in the state of order during the rolling of passes will increase the MFS. Increasing the total reduction and/or reducing the number of rolling above 950°C: both curves are almost coincident. Obviously, the conditions there is only a very slight effect of the Si-content torsion experiences), see Table 2. For each set of rolling conditions is presented in Figs. 9(a) and 9(b). The curves correspond to different rolling conditions in which the hot rolling has been conducted in different number of passes, then different inter pass cooling rates, and finished at temperatures below or higher than the \( T_{nr} \) (estimated by the hot torsion experiences), see Table 2. For each set of rolling conditions there is only a very slight effect of the Si-content above 950°C: both curves are almost coincident. Obviously, increasing the total reduction and/or reducing the number of passes will increase the MFS.

Steps III and IV: The parameters which control the ordering kinetics and the changes in the state of order during the processing seem to be the cooling rate after hot rolling and in some cases the time delay prior to cold rolling. For a set of experiments concerning the effect of the cooling rate during the processing, samples were hot rolled in 2 passes until 1.7 mm with 93% of total reduction, see Table 2. The finishing temperature of the hot rolling was between 940 and 970°C and the samples were cooled down from this temperature to room temperature using three different cooling rates, 1 000°C/min for water quenching, 450°C/min in quiet air at room temperature and 0.38°C/min for furnace cooling simulating the cooling of a coil after hot rolling. Samples quenched in water after hot rolling were cold rolled in a large series of passes from 1.70 mm down to 0.50 mm with 71% of total reduction without experiencing edge cracks. In samples cooled at the air after hot rolling and immediately cold rolled (without any time delay), starting with 1.40 mm, a final thickness of 0.50 mm could be obtained for both materials without cracking problems. However, the water quenched samples exhibited a higher ductility in cold rolling, while smaller reduction (64% for alloy B) could only be achieved in samples air cooled after hot rolling and in some occasion edge cracks appeared. Samples cooled down very slowly in the furnace were impossible to cold roll because of their brittle behaviour. This is most probably due to a high ordering degree achieved during cooling after hot rolling.

Fast cooling after hot rolling (\( >400°C/min \)) made the material sufficiently ductile for cold rolling, but in certain conditions only during a limited period of time. The cold rolling in the deformed state can be done without additional treatment between the hot and cold rolling. Total reductions of 70% can be achieved during cold rolling without cracks experiences.

4. Conclusions

Thermomechanical processing of high silicon steel (\( >4 wt\% Si \)) is possible whenever adequate temperatures and rolling schedules are used. Finishing the hot rolling at temperatures not lower than 950°C and fast cooling rates (\( >400°C/min \)) are recommended in order to allow cold rolling without cracking problems until reductions of 70%. Hot torsion tests have shown that deformation is easy at temperatures above the ordering temperature, which is estimated between 1 000 and 1 100°C, depending on the Si-content and followed thermomechanical procedure. Ordering appearing during cooling below \( T_{nr} \) causes the MFS to increase, while a further increase of MFS occurs for \( T< T_{nr} \) (non recrystallisation). Steel with a higher Si-content is slightly more sensitive to the temperature variation in terms of MFS and consequently more difficult to deform. The increase of the MFS below \( T_{nr} \) is not only associated with the cooling but also with the retardation of recrystallisation by the appearance of ordering. \( T_{ord} \) and \( T_{nr} \) are also influenced by the cooling rate, the Si-content and the deformation degree in each step.

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