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Formation of Widmanstätten ferrite at very high temperatures in the austenite phase field

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ABSTRACT

Compression tests were carried out on a 0.06wt%C-0.3wt%Mn-0.01wt%Si steel at temperatures high in the austenite phase field. Eight deformation temperatures were selected in the range from 1000 to 1350 °C at 50 °C intervals. The quenched samples were examined using optical microscopy and EBSD techniques. It was observed that dynamic transformation took place and that the volume fraction of transformed ferrite first decreased with temperature (up to 1050 °C) and then increased as the delta ferrite temperature domain was approached. The EBSD results revealed the presence of Widmanstätten ferrite plates under all testing conditions, right up to 1350 °C.

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1. Introduction

The transformation of austenite to ferrite during deformation above the Ae_3 temperature was first studied by Yada and co-workers in the 1980's [1,2]. In their pioneering work, Yada et al. showed that DT ferrite could be formed as high as 166 °C above the Ae_3. They also demonstrated the occurrence of this unusual phenomenon in real time by performing in-situ X-ray examinations while simultaneously deforming by torsion testing [3]. Follow-up work by Chen and Chen in 2000 [4] showed that DT ferrite retransformed back into austenite on prolonged holding, 30 °C above the Ae_3. More recently, researchers in various countries have reported on other aspects of the dynamic transformation of austenite [5–10].

The dislocation density introduced by working was initially taken to be the driving force for dynamic transformation. For example, Sun et al. [11] in 2008, calculated the Gibbs energy increase in the deformed system to be 22.3 J mol^{-1} and concluded that this was responsible for DT. Somewhat similar results were obtained by Hanlon et al. [12], who justified the presence of dynamically transformed ferrite in this way at temperatures up to 10 °C above the Ae_3. Ghosh et al. [13] extended the temperature range proposed by Hanlon and co-workers [12] by allowing for the distribution of dislocations to be inhomogeneous in nature. By increasing the estimated local stored energy to 197 J mol^{-1}, they were able to extend the predicted temperature range for the occurrence of this phenomenon up to 100 °C above the Ae_3.

One limitation of the inhomogeneous distribution model is that it is unable to explain how strains as low as 10% can lead to the initiation of DT. This difficulty was resolved by introducing the concept of mechanical activation by the applied stress, which can more readily account for the displacive nature of the forward transformation [14–16]. In the most recent version of this approach, Aranas et al. [17–19] proposed that the driving force for the transformation is the flow stress difference between the strain hardened austenite and the yield stress of Widmanstätten ferrite that takes its place. As will be shown below, such softening can provoke dynamic transformation at temperatures right up to the upper limits of the austenite phase field.

The objective of the present investigation was, therefore, to determine the characteristics of dynamic transformation at temperatures very high in the austenite phase field (up to 480 °C above the $Ae_{3\,(p)}$ in the present case). For this purpose, compression tests were performed on a 0.06wt%C-0.3wt%Mn-0.01wt%Si steel using a Gleeble 3800 thermomechanical system. The tests were conducted in the temperature range 1000–1350 °C (with intervals of 50°).

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Strains of 0.3 and 0.65 were employed in the tests and the strain rate was held constant at 1 s^{-1}. After water quenching, the specimens were polished and etched for examination by means of optical and electron back scattered microscopy. Double differentiation was also employed to identify the critical strains necessary for the initiation of dynamic transformation. The present observations indicate that the Fe–C phase diagram must be modified so as to apply to the case of dynamic loading.

2. Experimental procedure

2.1. Compression testing

The plain C–Mn steel was received in the form of hot rolled plates with thicknesses of 12.5 mm. Cylindrical compression samples with dimensions of 15 mm × 10 mm were machined from these plates with the cylinder axes parallel to the rolling direction. The composition of this alloy is shown in Table 1 together with the respective paraequilibrium and orthoequilibrium Ae_3 temperatures [13]. These were calculated using the FSStel database of the FactSage [20] and were validated using the Thermo-Calc thermodynamic software [21].

The compression tests were carried out in a vacuum and the Gleeble 3800 (Dynamic Systems Inc. USA) thermomechanical simulator was interfaced with QuikSim software that controlled the heating rate, cooling rate, quenching medium, strain, strain rate and temperature. The thermomechanical schedule employed is displayed in Fig. 1. Here the specimens were heated at the rate of 4.5 °C s^{-1} to test temperatures in the range 1000 °C– 1350 °C and held for 3 min in order to fully austenize the microstructure before deformation. These were water quenched immediately after straining. Note that, a K-type thermocouple was used for the experiments carried out over the range 1000 °C– 1250 °C. For the compression tests above 1250 °C, an R-type thermocouple was employed. The data were later processed into stress vs strain curves obtained directly from the above software.

The first set of compression tests involved the imposition of a strain of 0.3 applied at the rate of 1 s^{-1} over the temperature range 1200–1350 °C. In the second set, a higher strain of 0.65 was applied using the same parameters, but over the wider temperature range 1000–1350 °C. Note that these temperatures are high in the austenite phase field and approach the delta ferrite formation temperature of approximately at 1472 °C.

2.2. Metallography

For microstructural examination, central cross-sections perpendicular to the longitudinal axis were cut from the compressed specimens. The latter were then mounted in a phenolic hot mounting resin. The mounted specimens were polished using SiC papers from 400 to 1200 grit. Finally, these were subjected to 3 and 1 μm diamond polishing. Thereafter, the specimens were etched with 2% nital for optical microscopy. For determination of the volume fraction of DT ferrite in the austenite, etching was also carried out using a 10% sodium metabisulfite ($Na_2S_2O_5$) solution. MIP microstructure analysis software was then used to calculate the volume fractions.

Table 1
Chemical composition (weight %) and equilibrium transformation temperatures (°C).

C	Mn	Si	Orthoequilibrium Ae_3	Paraequilibrium Ae_3
0.06	0.30	0.01	877 °C	870 °C

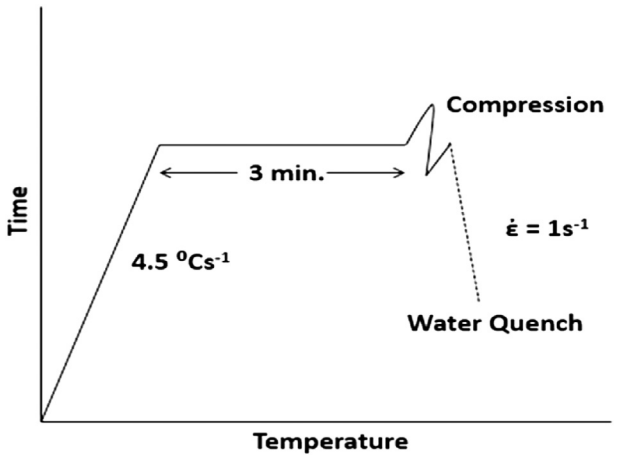

Fig. 1. Thermomechanical schedule of the compression tests carried out to strains of i) 0.3 at temperatures in the range from 1200 to 1350 °C and ii) 0.65 in the range 1000–1350 °C. The strain rate was 1 s^{-1} in both series of tests.

For the EBSD analysis, the specimens from each temperature were polished in the same manner as was employed for optical microscopy. Later, the specimens were subjected to final polishing using 0.05 μm silica in a vibromet for 6–8 h.

3. Results

The stress–strain curves associated with deformation over the temperature range 1000–1350 °C are displayed in Fig. 2. Here the samples were strained to 0.65 at the rate of 1 s^{-1}. All the curves display softening after the peak stress is attained. As shown by earlier workers [6,14] this is due to the concurrent operation of dynamic transformation and dynamic recrystallization. Steady state flow was attained at a strain of about 0.4. An interesting feature of these curves is their somewhat "flattened" shape prior to the peak. This is an indication of the initiation of dynamic transformation, which reduces the net rate of strain hardening once it is underway. The transformation softening process makes it possible to detect its initiation by means of the double differentiation technique, as will be shown in more detail below.

3.1. Mean flow stress

The areas under the flow curves of Fig. 2 were determined by

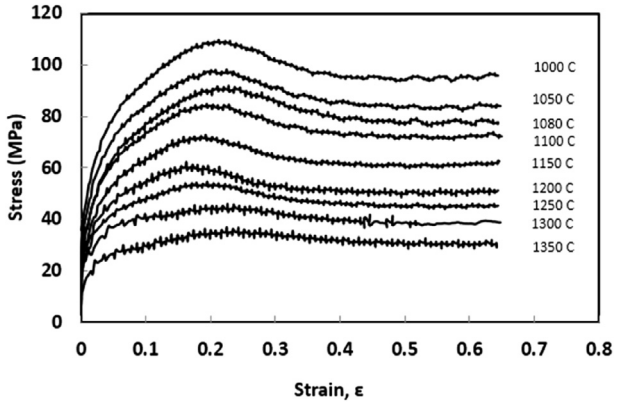

Fig. 2. Stress vs strain curves of the 0.06wt% C- 0.3wt% Mn - 0.01wt% Si steel compressed to a strain of 0.65 at 1 s^{-1} over the temperature range 1000–1350 °C. Flow softening of about 10% is generally observed after the peak stress is attained.

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