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A novel high-strain-rate ferrite dynamic softening mechanism facilitated by the interphase in the austenite/ferrite microstructure

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ABSTRACT

The ferrite softening mechanisms in the austenite/ferrite microstructure have been comprehensively examined as a function of the strain rate at a high deformation temperature. For this purpose, a 23Cr-6Ni-3Mo duplex stainless steel was used having the microstructure specifically designed to contain an extremely low fraction of the pre-existing ferrite/ferrite high-angle boundaries, which are generally expected to provide preferential discontinuous dynamic recrystallization (DDRX) nucleation sites. The deformation was performed in uniaxial compression at 1000 °C using strain rates of 0.1 and 10 s⁻¹ and a detailed microstructural analysis was conducted, including the determination of dislocation Burgers and line vectors, dislocation density and stored energy. The softening mechanism within ferrite at the low strain rate used has been classified as continuous dynamic recrystallization (CDRX), characterised by a progressive conversion of low-misoriented subgrains into (sub)grains delineated partly by low-angle and partly by high-angle boundaries. In contrast to the current widespread view, it has been revealed that a marked increase in the strain rate leads to a transition in the softening mechanism from CDRX towards a novel mechanism analogous to DDRX. The latter mechanism involves the formation of new grains through the growth of the highly-misoriented subgrains, preferentially formed in the ferrite/austenite interphase mantle regions.

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1. Introduction

The ferrite dynamic restoration mechanisms, in particular in the two-phase microstructures, have long been the subject of intense debate among different research groups around the world. Specifically, there have been contradictory views on the effect of the Zener-Hollomon parameter Z (i.e. the temperature compensated strain rate [1]) on the dominant ferrite softening mechanism, namely dynamic recovery and continuous, discontinuous or geometric dynamic recrystallization [1–26]. It has been widely accepted that the ferrite phase, characterised by relatively high stacking fault energy (SFE) values, tends to soften during hot deformation through intense dynamic recovery (DRV) [1]. This process might gradually evolve with increasing strain into continuous dynamic recrystallization (CDRX), also termed "extended" DRV. The above mechanism has been reported to operate in both single-phase ferrite steels [1–12] and dual-phase steels [2,13–18] under a wide range of hot deformation conditions. Gourdet et al.

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[19,20] have proposed that the CDRX process is not associated with the formation of new grains through nucleation and growth. Instead, low-angle boundaries (LABs) created by DRV at the early stages of deformation become progressively converted during straining into high-angle boundaries (HABs) through continuous absorption of dislocations. It should be noted, though, that some authors have questioned the operation of the CDRX mechanism in high SFE metals and, instead, have suggested that the presence of HABs might arise from geometric dynamic recrystallization (GDRX) [1,21].

There have also been suggestions that ferrite in both singlephase [8,10,12,22–25] and dual-phase [26] steels might, under certain hot deformation conditions, soften through discontinuous dynamic recrystallization (DDRX). However, the actual conditions that would favour the above softening mechanism are currently a matter of intense debate. There have been suggestions [8,12,22–24] that DDRX tends to operate within ferrite when the Z parameter falls below a certain critical limit (i.e. at high deformation temperatures and low strain rates), whereas DRV (or CDRX) dominates when Z exceeds the above critical threshold. By contrast, Castan et al. [10] have recently reported that DDRX in Fe-8% Al low-density







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steel subjected to hot torsion is favoured over CDRX when the strain rate is increased in the temperature range between 900 and 1100 °C (i.e. with an increase in the Z value). Furthermore, it should be highlighted that, to date, the actual formation mechanism of ferrite DDRX grains and their dislocation substructure characteristics have not been detailed even in the case of single-phase steels. Thus, more detailed experimental work is urgently required to clarify the above matters.

In analogy to austenite [1], the ferrite grain boundaries might be expected to serve as preferential DDRX nucleation sites. In the twophase austenite/ferrite microstructures, the content of these boundaries can be varied, as it is determined by the phase ratio and the morphological characteristics of the phases. The above characteristics, together with the character of the interphase and the deformation conditions, also govern the strain partitioning between the austenite and ferrite phases. It has been reported that strain tends to preferentially partition into comparatively softer ferrite during hot working of duplex steels [27–29]. Thus, as there is a need to accommodate the stress/strain incompatibilities across the interphase boundaries, deformation inhomogeneities might be expected to preferentially accumulate in the interphase mantle regions within ferrite, which might also impact on the character of ferrite softening. In the above context, it would be of interest to elucidate the nature of the ferrite restoration processes in a duplex microstructure, characterised by a limited presence of the preexisting ferrite/ferrite HABs, and the role the interphase might play in these processes.

The objective of the present work was to perform a detailed examination of the microstructure evolution and softening processes within ferrite during hot deformation of the austenite/ferrite microstructure, having an extremely low content of ferrite/ferrite grain boundaries, as a function of the strain rate. A specially heat treated 23Cr-6Ni-3Mo duplex stainless steel, containing approximately equal austenite and ferrite fractions, was employed in the investigation. The above steel does not undergo a phase transformation during quenching from hot deformation temperatures, which makes it possible to perform a detailed microstructural investigation of the high-temperature deformation microstructure. Taking into account the industrial importance of the duplex stainless steels [30], the data obtained can be expected to have both fundamental and practical significance.

2. Experimental procedures

The experimental material was a 2205 duplex stainless steel with the chemical composition of 0.036 C, 0.321 Si, 1.82 Mn, 0.013 P, 23.2 Cr, 2.90 Mo, 5.6 Ni, 0.034 Co, 0.153 Cu, 0.018 Nb, 0.065 V, 0.025 W, 0.245 N (in wt. %) and remainder Fe. The as-received material was in a form of a hot rolled plate with a thickness of 20 mm. The plate sections were reheated at 1370 °C and held for 40 min in a muffle furnace in an argon atmosphere, which resulted in the full dissolution of austenite and the formation of a single phase ferritic microstructure. The sections were then slowly cooled in the furnace from 1370 °C to 970 °C within 48 h and, to avoid the formation of brittle precipitates, they were water-quenched from 970 °C. This heat treatment resulted in the diffusional transformation of delta ferrite to austenite, forming a microstructure containing about 50% austenite with a morphology of roughly equiaxed islands distributed in the ferritic matrix. Importantly, the original ferrite grain boundaries were almost fully occupied by austenite islands, which provided an extremely low content of the pre-existing ferrite/ferrite HABs.

The heat treated material was then machined to make cylindrical compression samples, having a length of 15 mm and a diameter of 10 mm, with the compression axis parallel to the plate rolling direction. The hot deformation was performed in uniaxial compression using a computer-controlled servo-hydraulic deformation simulator (Servotest TMTS machine manufactured by the Servotest Company, UK). The samples were first heated to 1000 °C at a rate of 10 °C/sec, and held for 120 s to obtain uniform temperature distribution throughout the sample cross-section. They were then subjected to single-pass deformation performed at strain rates of 0.1 and 10 s⁻¹ to true strains of 0.2, 0.4, 0.6, 0.8, and 1.0. The deformation was followed by immediate water quenching to preserve the obtained microstructure.

Microstructural examination was performed in the central regions of the hot compressed samples on a plane containing the compression axis (CA) using the electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) techniques. The sample preparation methods for the above techniques are detailed elsewhere [18]. EBSD study was carried out using Zeiss LEO 1530 FEG SEM and FEI Quanta 3D FEG SEM/FIB instruments, both operated at 20 kV. The former instrument was equipped with the HKL Technology (now Oxford Instruments) EBSD attachment. The data acquisition was performed using the Oxford Instruments Aztec software, while the HKL Channel 5 software was used for data post processing. This included the careful wild spike removal followed by noise reduction and the application of the edge preserving modified Kuwahara filter routine for orientation averaging [31]. The latter routine was based on a 7×7 pixel grid, which would enhance the precision of misorientation detection to below 0.5°. The FEI Quanta instrument included the TexSEM Laboratories (TSL OIM) EBSD hardware and software for data acquisition and postprocessing. For the substructure statistical analysis, 8 EBSD maps with the size of ~200 \times 200 μ m² were collected for each deformation condition using a step size of 0.2 μ m. An area of ~6 \times 4 mm² was scanned at each condition to investigate the evolution of crystallographic texture using the step size of 2 µm. The DDRX grains were separated from the deformed matrix manually using the crop routine in the TSL OIM software. A TEM examination of thin foils was performed using a JEOL JEM 2100F microscope operated at 200 kV. Local crystallographic orientations and misorientations were determined using convergent-beam Kikuchi patterns [32]. The density of dislocations was determined according to the Ham method [33] with the foil thickness being estimated using convergent-beam electron diffraction (CBED) [34]. The dislocation density determination was performed in 8-10 ferrite locations (grains) in several thin foils for strain levels of 0.2, 0.6 and 1.0. The statistical error in the experimental data was, as widely accepted, estimated as a standard deviation of the measured population divided by a square root of the number of measurements. Furthermore, the "reading error" was also taken into consideration, limiting the accuracy of the measurement of misorientation and sub/grain size to 0.5° and the step size, respectively. In the following, terms "subgrains", "(sub)grains", and "grains" relate to the microstructure units delineated fully by LABs, by both LABs and HABs, and fully by HABs, respectively. Furthermore, in view of the complex nature of misorientation arrangements encountered in the present work, it has been decided to use a single term "misorientation" for all the boundaries throughout the text, instead of using a separate term "disorientation" for the case of high-angle boundaries.

3. Experimental results

3.1. Starting material, flow behaviour and phase structure

Fig. 1a—c shows the microstructure of the starting material, obtained after the heat treatment of the original hot rolled plate described in Section 2. It is seen that the microstructure contained

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