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Clustering and precipitation processes in a ferritic titanium-molybdenum microalloyed steel

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ABSTRACT

Atom Probe Microscopy has been performed on a titanium-molybdenum microalloyed steel after simulation of thermomechanical processing. The results reveal the evolution of clustering and precipitation reactions at the austenite/ferrite interphase interface during isothermal heat treatments of durations ranging between 300 s and 100 h. Shorter isothermal ageing times led to extensive solute clustering at the austenite/ferrite interface, which remained free of precipitates. For ageing times beyond 3600 s, high number densities of interphase nano-precipitates were observed along with extensive solute clustering. Beyond 100 h, there was little evidence of solute clustering at these austenite/ferrite microstructural interfaces, which were instead dominated by a dispersion of coarser nano-precipitates. Both the clusters and nano-precipitates formed during isothermal ageing possessed a disc shape morphology and the growth has taken place in the through thickness direction of the disc without much change in the aspect ratio. The size of clusters and nano-precipitates increased from ~2 nm after 300 s to ~15 nm after 100 h of isothermal ageing at 650 °C. The solute clusters contain mainly C, Ti and Mo atoms, while the stoichiometry of the nano-precipitates approached that of MC carbide as their size increased beyond ~4 nm. It is proposed that Mo controls the process of precipitate growth, resulting in a fine and uniform dispersion of nano-precipitates and a high level of tensile strength after just 300 s ageing at 650 °C. Interestingly, the peak strength was achieved during the very early stages of the precipitation process.

1. Introduction

Steel is a major structural material for the transport and construction sectors. For all applications there is a general trend to higher strength while maintaining or improving other properties. This has led to a major increase in research to develop Advanced High Strength Steels (AHSS). These steels provide increased strength with equivalent, or improved, ductility making it possible to reduce the weight of components. AHSS are produced by introducing a mix of phases, such as martensite (α'), bainite, and retained austenite (γ) into the microstructure. However, these multiphase steels often possess poor stretch-flangeability [1]. A single-phase ferrite (α) microstructures mitigates this problem, but the strength levels of ferritic microstructures, even with precipitation hardening, are usually below 600 MPa. However, JFE developed a steel in Japan with a strength of 780 MPa [1] (and more recently a 980 MPa steel) through controlled precipitation hardening. Significantly, the level of the contribution to strength from precipitation was almost twice that obtained in conventional precipitation-hardened low-carbon steels [2]. Fine interphase precipitates were formed during the austenite-to-ferrite phase transformation at the γ/α interface. These serve as highly effective strength enhancers through dispersion strengthening, and have superior properties when compared to larger carbonitride

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precipitates that form in austenite or precipitate as uniform dispersions from supersaturated ferrite [2,3].

The γ/α interphase boundary acts as the preferred nucleation site for precipitation because both strain and surface energy can be minimized. Davenport et al. [4] suggested that the migration of the γ/α interphase boundary is driven by the concentration gradient of carbon in the adjacent austenite, where a para-equilibrium condition exists [5]. Therefore, when the movement of the interphase boundary pauses for precipitation nucleation, only partitioning of carbon takes place, and there is no movement of metallic solutes across the interface boundary. It is likely that the γ/α boundary absorbs metallic solutes atoms during migration, which, in turn, applies a drag force to finally stop the interface boundary when eventually the precipitate nucleation takes place [6]. The growth of the precipitates occurs rapidly because of the easy diffusion of the metallic solute atoms along the interphase boundary. Following formation of interphase precipitates, the carbon concentration ahead of the γ/α interphase becomes favourable and the interphase boundary migrates ahead to a new position. This whole process is repeated during the austenite to ferrite phase transformation.

Thermo-mechanical controlled processing (TMCP) is one of the most simple and cost effective ways to produce AHSS. These are typically produced on a hot strip mill where the steel is rolled above the transformation temperature and then water cooled on the run out table prior to being coiled. Because of the large thermal mass of the coil, the reactions within the coil can be assumed to be isothermal. The strip rolling coiling influences the strength of AHSS. Higher temperatures and longer holding times normally coarsen precipitates and thereby decrease the final mechanical properties of the steel. This knowledge is extremely important from precipitate growth and thereby alloy design point of view. However, the mechanism of interphase precipitates nucleation and growth during isothermal hold is not completely understood. Previous work on AHSS with Nb and Ti or with V addition [7–9], revealed that the relationship between the strength and isothermal holding time in these steels follows a near bell shape pattern i.e the peak strength was attained after a certain holding time (normally from 20 to 60 min) at the isothermal holding temperature. Thereafter, prolonged holding of these AHSS reduced the strength drastically, primarily due to precipitate coarsening. The effect of isothermal time on the interphase row spacing and, whether the interphase precipitate rows still exist after prolong isothermal holding has been previously discussed [7,10]. Early work on high V and Ti steels [10] showed that at a relatively higher ageing temperature (700–800 °C), the interphase precipitate rows disappeared even after a short holding time, whereas, at 600 °C interphase rows were present even after 500 h [10].

It has been reported for AHSS containing Ti and Mo [1,2] that the nucleation of interphase precipitates and coarsening kinetics of TiC particles can be controlled by Mo. The effect of Mo has been studied [1] for Ti-AHSS at 650 °C. It was found that the tensile strength with increasing times at 650 °C decreased in the Ti-AHSS, whereas there was no tensile strength change in the Ti-AHSS with Mo. However, an increase in the Ti/Mo ratio in the steel composition and an increase in the isothermal temperature to 700 °C led to a decrease in strength [1] due to the coarsening of the (Ti, Mo)C precipitates. It was suggested [11] that Mo atoms participate in the precipitation of (Ti,Mo)C carbides during the very early stages of evolution, whereas during coarsening of the interphase precipitates, the substitution of Ti by Mo reduced the equilibrium Ti concentration in the ferrite matrix, which possibly decelerates the coarsening process of (Ti Mo)C precipitation and improves the strength [11]. However, the precise role of Mo in Ti-AHSS is unclear.

The contribution to the strengthening of AHSS due to the formation of interphase precipitates has been studied by several groups [7,9,12–14]. In general, dispersion strengthening can be achieved by deformable, non deformable or other second phase particles in a single phase matrix [15]. Deformable particles are considered to be very small and can be cut by dislocations [15]. The coherency strain and chemical hardening effects are considered to be the main cause of strength improvement [15,7]. Orowan suggested [12] that the increase in strength is due to the dislocation loop formation around non deformable particle, where the stress was inversely proportional to the inter-particle spacing [12]. Later, Ashby modified this concept and showed the role of particle size and volume fraction [13]. A typical increment in the yield strength calculated for different AHSS was found to be 100–150 MPa [3,14], which was in reasonably good agreement with the Ashby-Orowan predictions. However, Kestenbach et al. [14] showed that based on the Ashby-Orowan model, the strength of the microalloyed steels mainly relied upon the volume fraction of the precipitates, rather than the precipitate size [14] i.e. the contribution in strength increment from the coarse precipitates, formed in austenite, is similar to an increase in strength due to the formation of interphase precipitates. It was proposed that as the size of the precipitates increased, the probability of cutting these particles by slip planes also increased which then increased the strength. However, the total strength increment calculation was in agreement with the Ashby-Orowan model. It was suggested by Charleux et al. [7] that the Ashby-Orowan model is based on coarse, non-shearable particles, while the interphase precipitates are fine (~3–5 nm). Hence, a non-linear flow stress model was developed to describe the interphase precipitation strengthening contribution based on the shearable/non-shearable transition diameter of precipitates (5 nm) [15]. The data discussed above showed the importance of the proper measurement of the particle size, the distance between the rows and particles and whether the other microstructural features like clusters were also formed during interphase precipitation.

Thus, in the present work, the effect of isothermal time on the formation of Ti-Mo interphase particles in the thermomechanically processed AHSS was studied using Atom Probe Tomography (APT). The objectives of this paper are

(a) to obtain a fundamental understanding of interphase particles formation during isothermal hold; (b) to perform a quantitative analysis of the shape, size, composition and number density of nano-particles during different isothermal holding times and (c) to describe the evolution of nano precipitates as a function of the isothermal holding time.

2. Experimental methods

In this work, a steel with a composition 0.04C-1.52Mn-0.21Si-0.05Ti-0.22Mo (wt. %), (0.2C-1.5Mn-0.4Si-0.06Ti-0.13Mo (at. %)) was used. The thermomechanical simulation was performed using a Servotest-500 kN machine.

Plate of 40 mm thickness was reduced to 13 mm by hot rolling between 1200 and 1000 °C. Uniaxial compression samples with a height of 15 mm and a diameter of 10 mm were machined with their longitudinal axis along the transverse direction of the plate. During the simulation, the samples were reheated to 1200 °C at a rate of 5° Cs^{-1} and held for 180 s. The precipitation dissolution temperature and equilibrium temperatures (Ae_3 and Ae_1) were calculated using the Thermo-Calc software with the TCFe3 database. The reheating temperature of 1200 °C was selected to be well above the dissolution temperature of the carbides and carbonitrides. Following this, samples were cooled at 10 °Cs^{-1} to 890 °C (Fig. 1), and held for 10 s followed by deformation to a strain of 1 at