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Quenching and partitioning (Q&P) processing of fully austenitic stainless steels

M. Wendler [a, *], C. Ullrich [b], M. Hauser [a], L. Krüger [c], O. Volkova [a], A. Weiß [a], J. Mola [a]

[a] TU Bergakademie Freiberg, Institute of Iron and Steel Technology, Leipziger St. 34, 09599 Freiberg, Germany
[b] TU Bergakademie Freiberg, Institute of Materials Science, Gustav-Zeuner St. 5, 09599 Freiberg, Germany
[c] TU Bergakademie Freiberg, Institute of Materials Engineering, Gustav-Zeuner St. 5, 09599 Freiberg, Germany

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ABSTRACT

A novel quenching and partitioning (Q&P) processing was applied to the steel Fe-15Cr-3Ni-3Mn-0.5Si-0.12N-0.16C (wt.-%) with an almost fully austenitic microstructure in the solution annealed condition. The Q&P processing consisted of a subzero cooling step and a partitioning step at 450 °C. The fraction of α'-martensite after the Q&P processing was nearly 58 vol%. During partitioning, M_3C precipitates with an average size of 20 nm formed inside the α'-martensite. Concurrently, the interstitial content of austenite was increased by an average of almost 0.1 wt.-%. After Q&P processing, the stainless steel exhibited outstanding mechanical properties including a yield strength of 1050 MPa, an ultimate tensile strength of 1550 MPa, and a tensile elongation of 22% at room temperature. Reduction in the tensile test temperature from 100 °C to −40 °C facilitated the strain-induced α'-martensite formation and led to the simultaneous enhancement of strength and ductility. The enhancement of tensile ductility in spite of the strain-induced α'-martensite formation (α'-transformation-induced plasticity effect) was attributed to differences in the stability of austenite mainly caused by its non-uniform interstitial enrichment.

1. Introduction

In recent years, many researches have focused on the Quenching and Partitioning (Q&P) processing of martensitic low-alloy and stainless steels for the further enhancement of strength and ductility [1–5]. Q&P processing aims to generate the mechanical properties needed for the 3rd generation Advanced High Strength Steel (AHSS) applications. Such applications require ultimate tensile strength (UTS) levels above 1200/1300 MPa and total elongations (TE) in excess of 12% [6–8]. Q&P processing consists of an austenitization step followed by rapid cooling (quenching) to a temperature between martensite start (M_s) and martensite finish (M_f) temperatures known as the quench temperature. Low-alloy and stainless steels typically used for the Q&P processing have M_s temperatures well above room temperature (RT) so that direct quenching to RT results in only small volume fractions of austenite. Therefore, the desired martensite volume fraction has to be adjusted by quenching to a temperature above RT. After quenching, a partitioning step has to be performed in order to raise the stability of austenite. The partitioning step enables the diffusion of interstitial elements from supersaturated martensite to the untransformed austenite [9–11]. To delay or suppress the formation of transition carbides and M_3C in the martensitic phase, Si is added to steels [12,13]. The enrichment of austenite by C and N and its increased stability inhibits the formation of as-quenched martensite in the final cooling step so that all of the austenite existing at the partitioning temperature is retained at RT. Because of its ductility-enhancing effect, the volume fraction of retained austenite plays a very important role in Q&P steels. The formability of the austenitic phase is controlled by the glide of dislocations which could be wavy or planar [14–16]. In particular, twinning-induced plasticity (TWIP) and transformation-induced plasticity (ε- and/or α'-TRIP) mechanisms as byproducts of glide planarity have profound effects on the formability [17–23]. To achieve high elongation levels in metastable high-alloy austenitic steels, stacking fault energies (SFEs) in the range of 15–40 mJm^{-2} are required [24–27]. The SFE is mainly controlled by temperature [28–31] and chemical composition [32–36]. Ideally, the interstitial enrichment of austenite during Q&P processing must increase the SFE to within the preceding SFE range.

The aim of the present study is to demonstrate how a fully austenitic metastable stainless steel can be made ultra-high

* Corresponding author.
E-mail address: Marco.Wendler@iest.tu-freiberg.de (M. Wendler).

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strength by means of a novel Q&P processing consisting of subzero cooling in order to obtain a partially martensitic microstructure followed by partitioning. The main focus is placed on the microstructure formation processes during heat treatment steps and plasticity mechanisms during tensile tests at various temperatures.

2. Experimental

2.1. Ingot casting

The steel Cr15NC12.16 used in this study was produced in a VIM12 vacuum induction melting and casting facility from ALD Vacuum Technologies GmbH. An argon partial pressure of 350 mbar was applied in the melting step. Nitrogen gas with a partial pressure of 250 mbar was later introduced for nitriding. The steel was finally cast into a water-cooled copper mould placed in the furnace chamber. To avoid pore formation in the ingots, the nitrogen partial pressure was raised to 1500 mbar during casting. The chemical composition of the austenitic as-cast steel and its SFE at RT according to the empirical relationship proposed by Dai et al. [37] are given in Table 1.

2.2. Tensile tests

For tensile testing, round specimens with a gauge diameter of 6 mm and a gauge length of 30 mm were prepared according to the DIN 50125 standard. The heat-treated specimens were tensile tested in a Zwick 1476 universal testing machine. The crosshead velocity was set to 1 mm min^{-1} corresponding to an initial strain rate of 4×10^{-4} s^{-1}. Strain was measured using a clip-on extensometer. The adjustment of different temperatures in the range of -40 °C to 100 °C was done with the aid of a thermal chamber which surrounded the tensile specimen and its fixtures.

2.3. Magnetic measurements

For the quantification of the ferromagnetic martensite, a Metis MSAT magnetization device equipped with a Lake Shore 480 model fluxmeter was used. The equipment measures the magnetization in an external magnetic field of over 300 kA m^{-1}. The ferromagnetic phase content is quantified based on the measured magnetization after corrections for the effect of alloying elements on the magnetization of pure iron. The relative measurement error is below 1%. The device used returns the average magnetization of bulk specimens rather than that at surface which might be higher due to the transformation to martensite in the prior thermal and mechanical treatment steps [38,39].

2.4. Microstructure analysis

In addition to light optical microscopy (LOM), the microstructure of the steels was studied using a Zeiss LEO-1530 GEMINI-type field emission scanning electron microscope (FESEM) at an acceleration voltage of 20 kV. The HKL Channel 5 software was used for the analysis of the electron backscatter diffraction (EBSD) data. The step size during EBSD measurements was set to 0.1–0.2 μm. Further examination of the microstructure and lattice defects was done by means of electron channelling contrast imaging (ECCI). The analysis of precipitates by Selected Area Diffraction (SAD) and Fast-Fourier Transformation (FFT) analyses of high-resolution images was done in a Jeol JEM-2200FS transmission electron microscope (TEM) operated at an acceleration voltage of 200 kV.

To quantify the enrichment of austenite by interstitials during partitioning, the lattice parameter of austenite was determined by X-ray diffraction (XRD). Measurements were done using CuK_α radiation in a Seifert-FPM RD7 diffractometer. The sample preparation for XRD consisted of hot grinding to minimize the formation of surface martensite during sample preparation followed by electropolishing.

3. Results and discussion

3.1. Design of Q&P processing

In austenitic stainless steels with M_s temperatures equal to or slightly below RT, the martensite prerequisite for the application of Q&P processing can be introduced by further cooling. The Q&P processing of metastable austenitic stainless steels exhibiting athermal martensite formation below RT is shown schematically in Fig. 1. The formation of athermal martensite in metastable austenitic stainless steels can come to a standstill during cooling to cryogenic temperatures [40,41]. In such cases, the $\gamma \rightarrow \alpha'$ transformation remains incomplete. This characteristic behavior can be explained by a decrease in the chemical driving force $\Delta G^{\gamma \rightarrow \alpha'}$ originating from the paramagnetic to antiferromagnetic transition of austenite at Néel temperature (T_N) which in turn influences the physical and mechanical properties [40,42–44]. Consequently, the maximum martensite fraction is generated in the vicinity of T_N without further α'-martensite formation at lower temperatures (see Fig. 1). T_N depends on the chemical composition of the steel; whereas Mn increases T_N, other common alloying elements in stainless steels such as Cr, Ni, Si, C, and N decrease it [43,45–47]. Designing the alloy to ensure an M_s in the vicinity of RT and a T_N far lower than RT allows to generate controlled austenite and martensite fractions in the microstructure by quenching to cryogenic temperatures. These characteristics simplify the quenching step in comparison to low-alloy and conventional martensitic stainless steels since the initial quench temperature (prior to cryogenic cooling) could be set equal to RT. Moreover, the partitioning step does not have to be carried out immediately after quenching because the remaining austenite is thermally stable at RT.

In austenitic-martensitic stainless steels, an accelerated precipitation of carbides is expected inside the α'-martensite phase. It is well known that tempering at temperatures up to about 450 °C only leads to the formation of paraequilibrium M_3C-type carbides (M denotes Fe and substitutional alloying elements) [12,48,49]. During the Q&P processing, the formation of carbides in the partitioning step has to be prevented since it can lead to an insufficient interstitial enrichment of austenite and favor its further transformation to α'-martensite during the final cooling to RT. In view of the strength, however, the formation of nano-sized carbides could be beneficial as it increases the yield strength [38]. To ensure a good ductility in multiphase steels consisting of austenite, α'-martensite, and carbides, the austenite fraction should be elevated in comparison to conventional Q&P-steels. In this study, by means of a

Table 1
Chemical composition in wt.-% and the calculated SFE value at RT.

Alloy	C	N	Cr	Ni	Mn	Si	Fe + others	SFE at RT [mJm^{-2}] [37]
Cr15NC12.16	0.155	0.122	14.90	2.91	2.97	0.53	bal.	18