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Morphology change of retained austenite during austempering of carbide-free bainitic steel

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ABSTRACT

A change in the mechanical properties of a carbide-free bainitic steel was observed during prolonged holding at austempering temperature after termination of the bainitic transformation. To determine the origin of the property change, the microstructure was investigated by correlative electron microscopy. Although the retained austenite content remains the same during prolonged holding, its morphology changes from thin films separating the individual bainitic sub-units to a more globular structure. Since films of austenite contain a higher C concentration, the blocky austenite becomes gradually enriched in C during this morphology change. The more homogeneous distribution of the C after prolonged austempering leads to higher deformability as a result of a more pronounced TRIP effect.

1. Introduction

Increasing demands on safety and fuel efficiency in the automotive industry have led to the development of advanced high-strength steels (AHSS). Currently, the third generation of AHSS is the subject of ongoing steel research. There, the advantages of the multi-phase microstructure of the first generation of AHSS and increased fractions of meta-stable retained austenite, which increases strain hardening by the transformation-induced plasticity (TRIP) effect, of the second generation are combined to obtain improved and balanced strength and ductility combinations. The high strength is realized by a hard, fine-grained martensitic or bainitic matrix, whereas the enhanced ductility is achieved by stabilizing considerable amounts of retained austenite [1–4]. Economic benefits of the third generation of AHSS result from the lower amount of alloying elements and also their processability is superior compared to the second generation.

These high requirements are addressed by several different approaches. For example, medium Mn steels with a decreased Mn content compared to the second generation show improved processability and economy, and thus are topic of current research [5]. Speer et al. [4] developed the quenching-and-partitioning (Q&P) process, where low-alloyed steels are quenched to temperatures between the martensite start (M_s) temperature and the martensite finish (M_f) temperature. Subsequently, the C is allowed to partition into the remaining austenite which, as a result, is stabilized. Carbide-free bainitic steels, which are the topic of the present study, use a similar approach, but they are produced via austempering in the bainitic temperature range [6].

The cost-effective alloying concept of carbide-free bainitic steels is mainly based on an increased amount of Si which prevents cementite precipitation and, therefore, leaves the C available for austenite stabilization [7,8]. By means of the T_0 curve [9], which is the locus of all points on a temperature versus C concentration plot where austenite and ferrite of the same chemical composition have the same free energy, the phase fractions and C concentration of bainitic ferrite and retained austenite can be estimated after transformation at a particular isothermal holding temperature. Retained austenite with a very low stability transforms early during plastic straining and, consequently, its influence on the mechanical properties is limited. On the other hand, if the stability of the austenite is too high, it does not transform to martensite even under high loads and the potential of the TRIP effect remains unused [10,11]. Since the amount and stability of the austenite are of fundamental importance in these steels, many studies have focused on means to control these microstructural features. In carbide-free bainitic steels there are two types of austenite. In between sub-units which share the same crystallographic orientation, the austenite is present in form of nanoscale

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films, whereas in between sheaves of bainite the austenite exhibits a more blocky morphology [7,12]. Caballero et al. [12,13] found that the films of austenite contain a higher amount of C compared to the blocky austenite. The M_s strongly depends on the chemical composition, whereby C has the greatest influence [14]. Therefore, increasing amounts of C in the retained austenite increases its chemical, and hence, also its mechanical stability. However, not only the chemical composition but also the austenite grain size contributes to the stability since smaller grains provide less potential nucleation sites for martensite and, thus, are more resistant to transformation [15].

The bainitic matrix influences the mechanical properties of the steel through the bainitic grain size and, additionally, through its ability to constrain the transformation of the retained austenite as it is more stable if the austenite is surrounded and shielded by a hard matrix [11,16,17]. In summary, the austenite stability depends on chemical composition, mainly the C concentration, morphology, size, and distribution within the matrix.

In the investigated low-alloyed bainitic steel the microstructure evolution is not well understood yet. Especially a change in the mechanical properties during prolonged holding times after the bainitic transformation, which is accompanied by a change in austenite morphology, has not been addressed in detail. Therefore, the aim of the current study was to correlate the observed mechanical properties to the microstructure with special focus on the retained austenite. The amount of austenite as determined magnetically was compared to the amount observed by electron backscatter diffraction (EBSD). Higher resolution investigations of the morphology and size were carried out by means of a correlative microscopy approach applying transmission electron microscopy (TEM) and transmission Kikuchi diffraction (TKD) [18]. The recently developed TKD technique [19,20] is based on the conventional EBSD hardware available in a scanning electron microscope (SEM). However, not the backscattered, but the electrons transmitted through a very thin sample are collected. Owing to the significantly smaller interaction volume, the spatial resolution is greatly improved. Therefore, this new technique facilitates the investigation of the nanostructure of AHSS. Additionally, the chemical stability of the retained austenite was assessed by atom probe tomography (APT). The results from the microstructural investigation explain the observed mechanical properties and thus extend the understanding of the microstructure-property relationship in complex steels.

2. Experimental

The chemical composition of the investigated low-alloyed steel is given in Table 1. Increased amounts of Si prevent cementite precipitation and the elevated Mn content ensures proper hardenability. The material was produced on an industrial scale in form of cold-rolled sheets with a thickness of 1.2 mm. Samples of $10 \times 10 \times 1.2$ mm^3 were inductively heat treated in a dilatometer DIL805A from TA Instruments (Germany) for the microstructural investigations. The temperature was controlled by a type S thermocouple, which was spot welded to the sample surface. Austenitization at 900 °C for 1 min was followed by quenching with 100 °C/s in He gas to different isothermal holding temperatures (350 °C, 375 °C, 400 °C), where the bainitic transformation could proceed for different holding times (1000 s and 3600 s). Finally, a second quenching step with 100 °C/s in He gas to room temperature followed.

Table 1
Chemical composition of the investigated steel in mass% and at%.

	Fe	Mn	Si	C
Mass%	Bal.	2.47	1.51	0.22
At%	Bal.	2.45	2.93	1.00

The austenite phase fraction was determined magnetically by the Joch-Isthmus method [21] where the investigated samples are magnetized to saturation and compared to a fully ferritic sample of the same chemical composition. From the difference of the magnetization the fraction of retained austenite can be calculated.

The austenite phase fraction, its morphology and the bainitic matrix were investigated by EBSD. Therefore, after conventional grinding and polishing the samples were electropolished on a Lectro-Pol V from Struers (Germany) using the commercially available A2 electrolyte (a mixture of 65–85% Ethanol, 10–15% 2-Butoxy-ethanol and 5–15% water and 60% perchloric acid in a ratio of 12:1). A voltage of 40 V and a flux of 10 at room temperature were applied. The resulting surface relief was subsequently removed by manually polishing using OP-U as proposed in [22]. Attention must particularly be paid to prevent a mechanically-induced transformation of meta-stable retained austenite to martensite during preparation.

SEM and EBSD investigations were carried out in a dual-beam SEM/ focused ion beam (FIB) microscope VERSA 3D from FEI (USA), equipped with a field emission gun and a Hikari XP EBSD system from EDAX (USA). The analyses were performed at a working distance of 15 mm with an acceleration voltage of 20 kV, 6×6 binning and a step size of 100 nm.

For TKD and TEM investigations heat treated samples were mechanically thinned to a thickness of 100 μm. Subsequently, disks of a diameter of 3 mm were punched out and electropolished on a TenuPol-5 twin-jet polisher from Struers (Germany) using a solution of 4 vol% perchloric acid ($HClO_4$) and 96 vol% acetic acid (CH_3COOH), applying a voltage of 15 V with a flow rate of 10 at room temperature. TKD experiments were carried out under an effective tilt angle of $-20°$ to the incident electron beam at a working distance of 5 mm with an acceleration voltage of 30 kV, 4×4 binning and step sizes between 20 nm and 50 nm. EBSD and TKD data evaluation was always performed with the TSL OIM Analysis 7 software. For TKD on the TEM samples a grain dilatation clean-up was applied. TEM investigations were conducted on a Tecnai F20 from FEI (USA) operated at 200 kV at the University Service Center for Transmission Electron Microscopy, Vienna University of Technology (Austria).

APT specimens were electropolished by the standard double-layer and micropolishing methods as reported in [23]. Subsequently, the combined TKD/FIB technique as described in [24] was applied to obtain the retained austenite in the first 100–200 nm of the final APT tip. For this, a voltage of 30 kV, 4×4 binning and a step size of 10 nm were used. The atom probe tips were analyzed using a local electrode atom probe LEAP 3000X HR from Cameca (USA) in the voltage mode with a pulse repetition rate of 200 kHz, a pulse fraction of 0.2 and a sample temperature of 60 K. Imago Visualization and Analysis Software (IVAS) version 3.6 was used for all analyses.

For the mechanical tests, cold-rolled sheets were heat treated using the multi-purpose annealing simulator MULTIPAS from vatron (Austria). The same heat treatment schema as for the dilatometry samples was conducted. Heating was carried out via electric resistance and cooling via pressured air by gas jet. After the heat treatment tinder was removed by pickling in diluted sulfuric acid (H_2SO_4). Flat tensile test samples with an initial length of 250 mm were used to determine the mechanical properties on the tensile testing machine BETA 250 from Messphysik (Austria) in accordance with EN 100002. The measurement accuracy was 6%.