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Multi-probe microstructure tracking during heat treatment without an in-situ setup: Case studies on martensitic steel, dual phase steel and β-Ti alloy

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ABSTRACT

In-situ scanning electron microscopy observations of the microstructure evolution during heat treatments are increasingly demanded due to the growing number of alloys with complex microstructures. Post-mortem characterization of the as-processed microstructures rarely provides sufficient insight on the exact route of the microstructure formation. On the other hand, in-situ SEM approaches are often limited due to the arising challenges upon using an in-situ heating setup, e.g. in (i) employing different detectors, (ii) preventing specimen surface degradation, or (iii) controlling and measuring the temperature precisely. Here, we explore and expand the capabilities of the "mid-way" solution by step-wise microstructure tracking, ex-situ, at selected steps of heat treatment. This approach circumvents the limitations above, as it involves an atmosphere and temperature well-controlled dilatometer, and high resolution microstructure characterization (using electron channeling contrast imaging, electron backscatter diffraction, atom probe tomography, etc.). We demonstrate the capabilities of this approach by focusing on three cases: (i) nano-scale carbide precipitation during low-temperature tempering of martensitic steels, (ii) formation of transformation-induced geometrically necessary dislocations in a dual-phase steel during intercritical annealing, and (iii) the partial recrystallization of a metastable β-Ti alloy.

1. Introduction

Current alloy design strategies for expanding the achievable property spectrum often lead to microstructures with increased complexity, e.g. by incorporating additional constituents, grain size distributions, and compositional heterogeneities at different scales. However, regardless of whether an austenitic–martensitic transformation-induced-plasticity (TRIP) steel, an age hardenable Al alloy, an α + β-Ti alloy, or any other alloy that is considered, the microstructure evolution cannot be easily back-tracked from the final microstructure. This is due to the various sequentially or simultaneously occurring micro-processes involved. Thus, there is a growing motivation for the employment of in-situ techniques for the analysis of the microstructure development. In-situ studies in transmission electron microscopy (TEM) [1–3], lab-scale X-ray systems [4,5], and high-energy beamlines [6–9] have enabled unprecedented leaps in the understanding of various micro-phenomena [2,4,7,9]. However, when considering microstructure development during heat treatment, each of these techniques have their specific limitations. Thin TEM foils are affected strongly by surface effects [10], and provide limited possibilities of follow-up analyses by other techniques (e.g. by atom probe tomography (APT)). In-situ X-ray diffraction (XRD), synchrotron X-ray diffraction (SXRD) or neutron diffraction studies access large volumes of integrated microstructural information (e.g. phase fraction, texture), however not allowing for the direct visualization of the microstructure itself. 3D X-ray microscopy [11,12] provides an improvement in this regard, yet still with limited spatial resolution not allowing for studying complex nanostructured alloys.

On the other hand, in-situ heating experiments in the scanning electron microscope (SEM) [13–19] provide an optimal combination of investigated material volume, surface effects, and varieties of imaging modes, resolution and practicality. Yet, the presence of a miniaturized heating stage in the SEM chamber can create certain limitations as well. Employing different detectors (backscattered electrons (BSE) and electron backscatter diffraction (EBSD) [20]) or imaging modes (electron channeling contrast imaging (ECCI) [21]) during heat treatment is limited due to degradation of the resolution and signal/noise ratio (caused by the thermal expansion, radiation and the spurious magnetic or electrical fields [13,22]), or even not possible due to other practical limitations (e.g. space, measurement duration). In fact, even with in-situ setups in chamber, EBSD measurements were often carried out after cooling down to room temperature [17,18] because of the limited applicable temperature and time resolution at high temperatures [14,16,22]. Additionally, precise control and measurement of temperature on the sample is difficult. Often the thermocouple is fixed to the heating stage rather

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than on the sample [14,22]. Achievable heating/cooling rates are usually lower than 100 $K \cdot s^{-1}$, typically from 0.2 to 10 $K \cdot s^{-1}$ [1,13,18,22,23]). Furthermore, even with the advanced vacuum systems of current SEMs, avoiding oxidation at high temperatures can be an important challenge.

In this work, we explore the capabilities of an approach which can be applied without an in-situ setup, that consists of (i) interrupted heat treatments [24] in a dilatometer, and (ii) microstructure characterization in-between by EBSD, ECCI, APT, etc. In what follows we refer to this methodology as "quasi-in-situ" due to the similarity of the data generated in classical in-situ experiments, although the probing process itself is not in-situ. The case studies which focus on three different alloys and heat treatment regimes demonstrate that through careful design of this experimental methodology, this practical approach can deliver significant amount of insight to complex micro- and nano-processes.

2. Methodology and materials

2.1. Quasi-in-situ methodology

The proposed quasi-in-situ methodology is illustrated schematically in Fig. 1. Firstly, a representative microstructural region is selected and characterized on the as-polished surface of the specimen. Heat treatment steps and follow-up microstructure analysis are cyclically repeated on the same sample as many times as required, which is similar to some previous in-situ heat treatment works in the SEM [18,23,25]. The microstructure evolution is not directly observed during heat treatment, but rather tracked back by relocating and imaging the room temperature microstructure after each cycle. Suitable markers are employed in each case study in order to easily detect the target region in the microscope after each heat treatment cycle, which is discussed in detail in chapter 4.

The microstructure characterization of the area of interest is carried out mainly by employing SEM-based BSE imaging, ECCI and EBSD techniques. Since the sample is not constrained to be present within a single analysis chamber, additional analyses can be carried out, which are otherwise not possible during a classical in-situ experiment in the SEM. In this report the use of APT is demonstrated in case study A, but other submicron resolution techniques (e.g. secondary ion mass spectrometry (SIMS), atomic force microscopy (AFM), Auger spectroscopy) can also be incorporated depending on the phenomena of interest. Thus, a multiprobe microstructure characterization approach can be realized at every annealing step complementing the standard SEM-based analysis.

The heat treatments are carried out in a dilatometer, allowing for high heat treatment precision and flexibility through the use of a spiral induction coil fully covering the sample. Nitrogen, helium, hydrogen and argon are the available atmospheres during annealing, while ambient temperature hydrogen or helium is flushed in the chamber for cooling process. The heating frequency and gas flow are close-loop controlled by the instant feedback signal from the spot-welded thermocouple on the sample surface. This ensures the accurate control of the temperature and heating/cooling rate to achieve the respective maximums of 1500 °C and 4000/−2500 $K \cdot s^{-1}$ with an accuracy of 0.05 °C [26]. The heat treatment parameters can therefore be accurately adapted to the kinetics of the investigated micro-phenomena. The chamber atmosphere control helps to limit oxidation of the sample surface. The use of a dilatometer also enables the dilatometric analysis of phase transformations [27], when needed.

2.2. Materials and experimental details

Three materials were investigated here to demonstrate observations of different microstructure phenomena during heat treatments. In case study A, the low-temperature tempering of a low-carbon martensitic steel with composition of Fe–2.51Mn–0.19Si–0.20Cr–0.225C (wt.%) was investigated. The lath martensitic microstructure was obtained after austenitization at 1060 °C for 2 min and quenching. For case study B, the intercritical annealing of a low-carbon ferrite-pearlite steel with composition of Fe–1.68Mn–0.241Si–0.092C (wt.%) was studied. In case study C, the partial recrystallization of a metastable β-Ti alloy with composition of Ti–35.7Nb–1.56Ta–2.83Zr–0.438O (wt.%) was investigated, which was cold rolled to ~80% thickness reduction ($\varepsilon \approx 1.56$) prior to the heat treatment. All samples, which have rectangular prism geometries ($9 \times 4 \times 1\ mm^3$), were wet-ground and polished before conducting the quasi-in-situ methodology. Final polishing was carried out using colloidal silica for steels and a solution of 75% colloidal silica and 25% H_2O_2 for the Ti-alloy.

For microstructure characterization, ECCI and EBSD measurements (of case study A) were carried out in a Zeiss Merlin and a JEOL JSM 6500F FEG-SEM with TSL detector, respectively. APT was conducted with a local electrode atom probe (LEAP™ 3000× HR, Cameca Instruments) under ultra-high vacuum conditions at 70 K sample temperature in voltage mode, with 15% pule fraction and 200 kHz pulse repetition rate. Fine needle shaped specimens were prepared by site-specific lift-out technique [28] using focused ion beam (FIB) milling in a FEI dual-beam HELIOS Nanolab 600i. HV1-hardness measurements were carried out after each heat treatment cycle in order to evaluate the change in mechanical properties. In case studies B and C, the microstructure characterizations were performed using a Zeiss-Crossbeam XB 1540 FIB-SEM instrument equipped with an EDAX/TSL system.

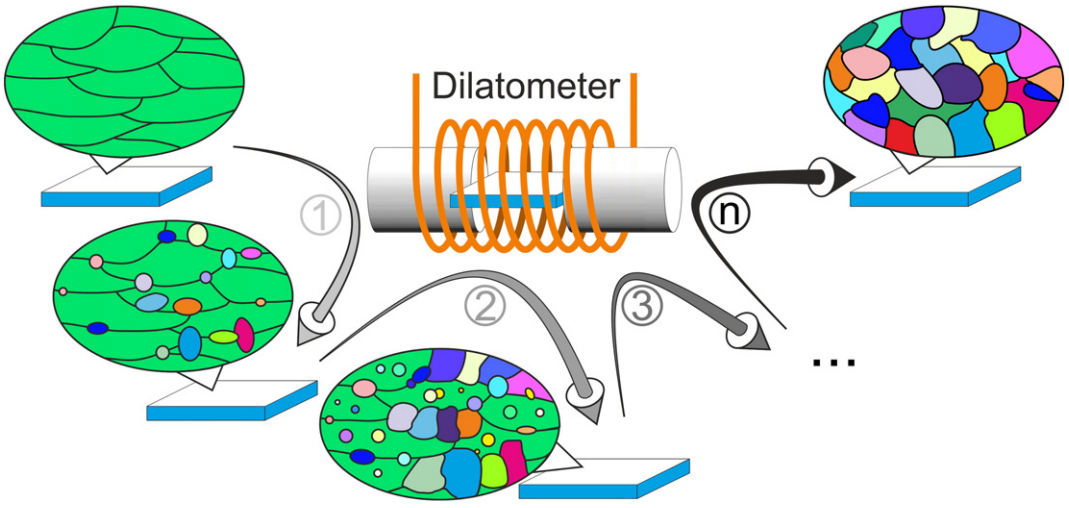

Fig. 1. Schematic illustration of the quasi-in-situ methodology investigating the recrystallization phenomenon. Each number corresponds to a single cycle which contains a dilatometer heat treatment step and microstructure characterization before and after.