



**Supplementary Materials** 

## **Comparative Study of the Tempering Behavior of Different Martensitic Steels by Means of In-Situ Diffractometry and Dilatometry**

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## **1. Diffraction Patterns**

## *1.1. The Diffraction Patterns for SAE 52100 Is Shown in the Main Text*

Exemplarily, the diffraction patterns for 20 °C, 300 °C, and 500 °C are shown in Figure S1. At 20 °C mainly martensite and a small amount of 5 ma.% austenite could be detected. The austenite peak vanished at 300 °C. At 500 °C, a peak of about 3 ma.% cementite occurred. Thermal expansion shifted the martensite peaks to smaller angles. To further elucidate the effects from the dilatometric measurements, they are compared to the results from the in-situ XRD tempering experiments, where lattice parameters of martensite, its tetragonality and the phase contents of cementite, martensite and retained austenite were evaluated.



**Figure S1.** Diffraction patterns at 20 °C, 300 °C, and 500 °C from in-situ X-ray diffraction for SAE 4140.

The diffraction patterns for 20 °C, 300 °C, and 500 °C are shown in Figure S2. At each temperature, mainly martensite and a small amount of 5 ma.% austenite could be detected. Thermal expansion shifted the martensite peaks smaller angles.



**Figure S2.** Diffraction patterns at 20 °C, 300 °C, and 500 °C from in-situ X-ray diffraction for SAE H13.

## *1.2. Metallography*

The microstructure of as-quenched SAE 4140 consists of full martensite, see Figure S3a. Partially, some martensite laths can be identified. Figure S3b shows micrographs of the quenched and tempered microstructure.



**Figure S3.** Micrographs of SAE 4140: **a**) as quenched, **b**) quenched and tempered up to 600 °C.

Figure S4a shows the micrograph of as quenched SAE 52100 (etchant: Nital). For the austenitizing condition applied, a significant amount of cementite is undissolved. This undissolved cementite can be identified by white-etched, spherical precipitations. Further, brown etched martensite plates and brighter austenite can be identified. Quenched and tempered SAE 52100 is shown in Figure S4b. The microstructure consists of a typical high annealed microstructure of SAE 52100.



**Figure S4.** Micrographs of SAE 52100: **a**) as quenched, **b**) quenched and tempered up to 500 °C.

An SEM micrograph of as-quenched SAE H13 is shown in Figure S5a. The microstructure is a typical martensite with some undissolved, spherical carbides. Figure S5b shows the quenched and tempered state. The secondary electron images (SE) were acquired by using a Helios Nanolab DualBeam microscope of type G3 CX with an accelerating voltage of 5 kV and a beam current of 0.17 nA.



**Figure S5.** SEM micrographs of SAE H13: **a**) as quenched, **b**) quenched and tempered up to 650 °C.



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