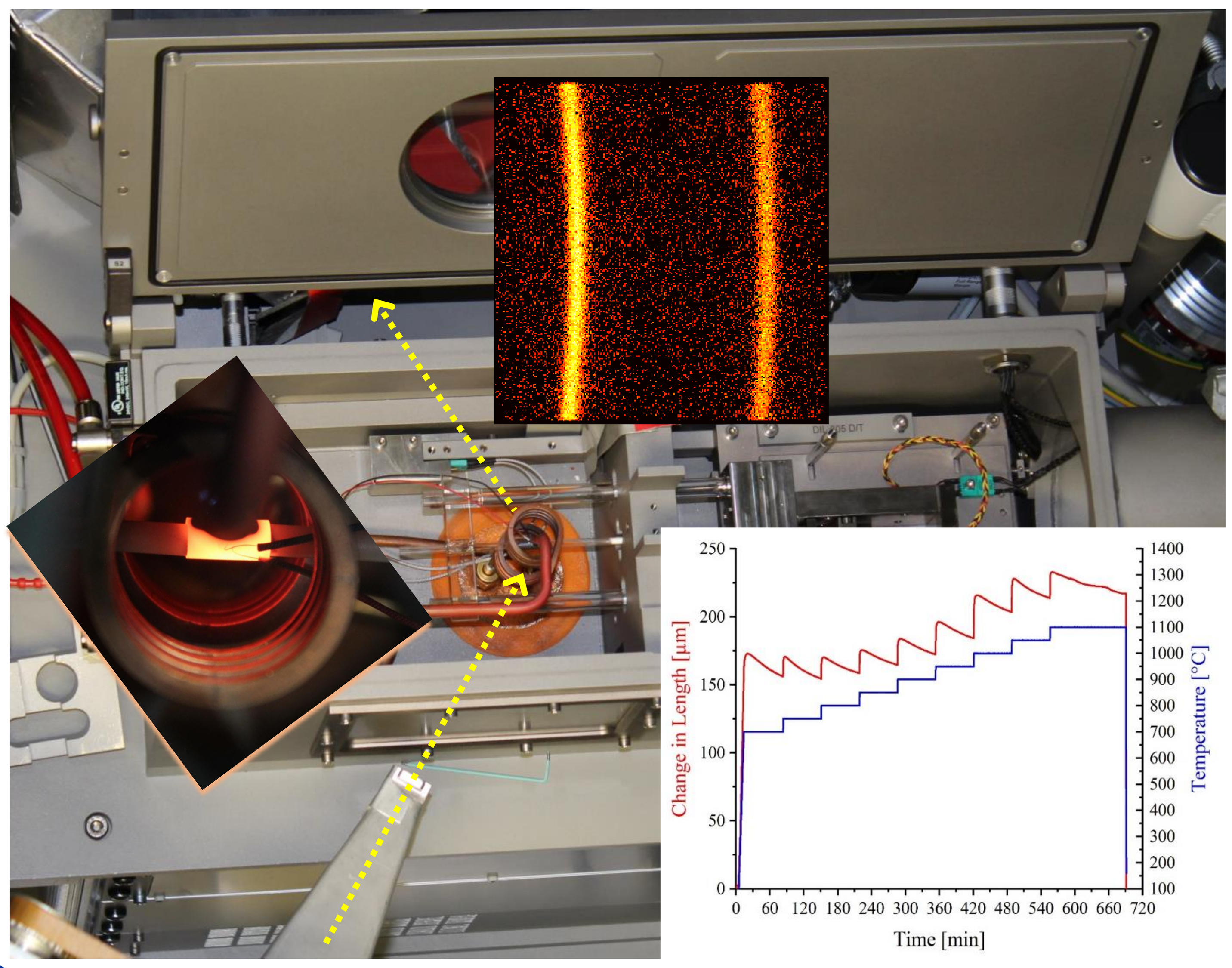
New Quenching and Deformation Dilatometer at FRM II

X.H. Li[1], C. Solis[2], M. Reiberg[3], M. Landesberger[1], M. Hofmann[1], W.M. Gan[2]

[1]Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, Garching, Germany
[2]German Engineering Materials Science Centre (GEMS) at MLZ, Helmholtz-Zentrum Hereon, Garching, Germany
[3]Institute of Materials Science and Mechanics of Materials, Technical University of Munich, Boltzmannstr. 15, 85748, Garching, Germany

Combination of dilatometer DIL 805A/D/T with neutron diffraction at FRM II (STRESS-SPEC)



Neutron beam path and experimental signal during the in-situ measurement

Technical details

	Alpha mode	Compression mode	Tension mode
Temperature Range	50˚C to 1500˚C With special designed heating coil up to 1450˚C at STRESS-SPEC and 1100˚C at SANS-1		
T. Resolution	0.05 ˚C		
Heating rate Cooling rate	4000 ˚C/s 2500 ˚C/s	100 ˚C/s 100 ˚C/s	100 ˚C/s 100 ˚C/s
Length change	10 nm	50 nm	50 nm
Deformation force		Up to 20 kN	Up to 8 kN
Deformation rate		0.01 – 200 mm/s	0.01 – 20 mm/s
Strain rate		0.001 – 20 s^{-1}	0.001 – 20 s^{-1}
Min. Gauge length		3 mm	3 mm

STRESS-SPEC — Alpha Quenching; Compression Tension
SANS-1 — Queching for sheet sample
Standard

Different types of heating coils

Publication:
X. H. Li, M. Hofmann, M. Landesberger, M. Reiberg, X. Zhang, E. Werner, W. M. Gan,: A unique quenching and deformation dilatometer for combined in situ neutron diffraction analysis of engineering materials; *Adv. Eng. Mater.* (2021) 2100163; **DOI:** 10.1002/adem.202100163

Recent Examples

High-temperature Phase Characterization in AlCrFeNiTi alloys

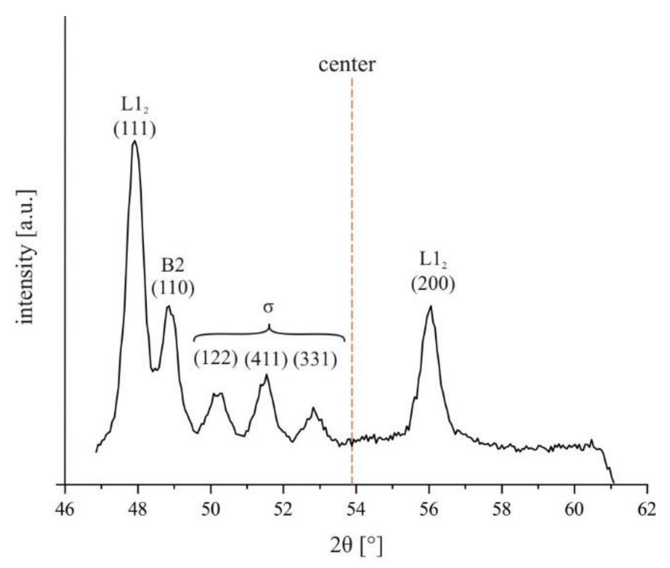


Fig.1 Neutron-diffractogram of composition Al10Ti10 as-HIP at 900 ºC. Tagged peaks are used for determination of phase-amounts of $L1_2$, B2 and σ.

$$I_{(hkl)\alpha} = \frac{C|B_{(hkl)\alpha}|^2 V_\alpha}{\mu_s}$$

C is a constant related to an experimental setup and remains the same in a single experiment, *B*(hkl)*α* is the structure factor of reflection (hkl) of phase *α*, *Vα* is the volume fraction of phase *α* and *μ*s is the mass absorption coefficient of the specimen.

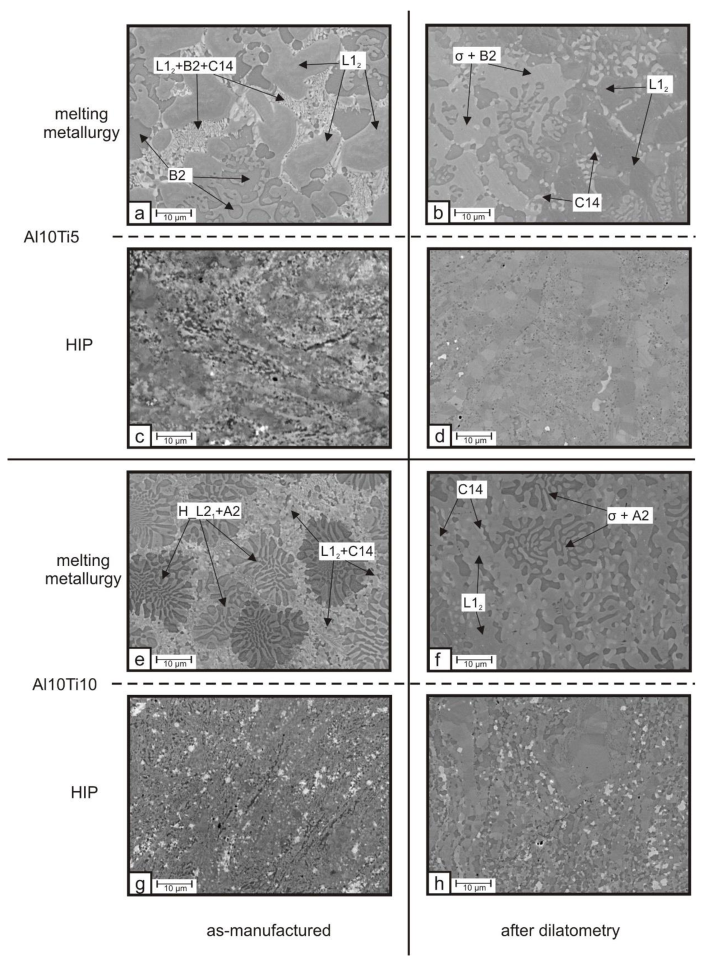


Fig.2 SEM images of compositions Al10Ti5 (a–d) and Al10Ti10 (e–h); Phases are labeled in the images of the samples produced by melting metallurgy.

Quantitative Phase Analysis

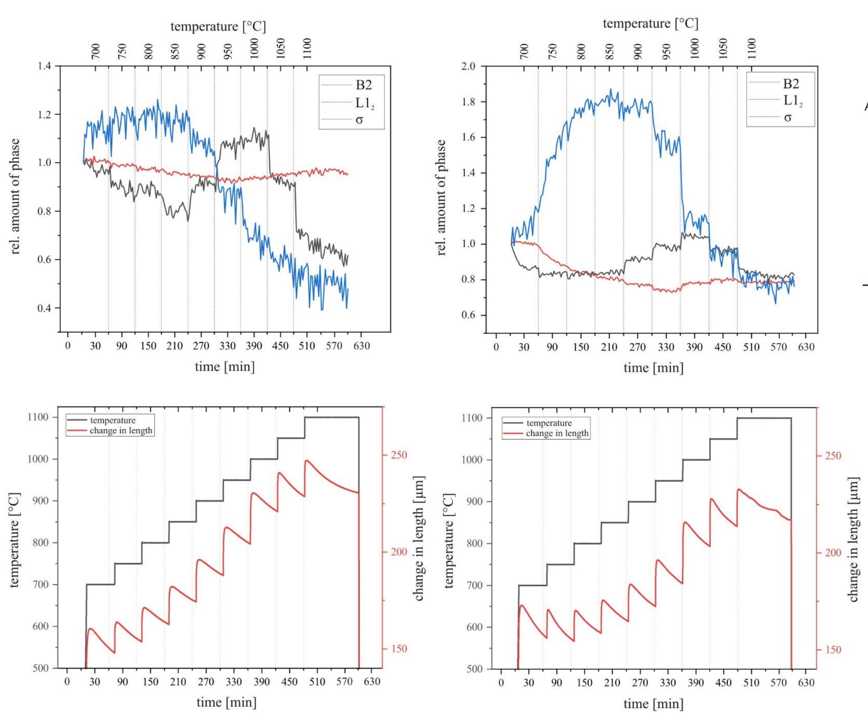


Fig. 3 Overview of the relative amount of phases determined by neutron-diffraction and the change in length and temperature plotted over time. Left: as-HIP Al10Ti5, right: as-HIP Al10Ti10. Since phase fractions differ significantly and the development of the phases are shown in the same diagram, the phase fractions are considered separately and are normalized to their respective values at 700 ºC.

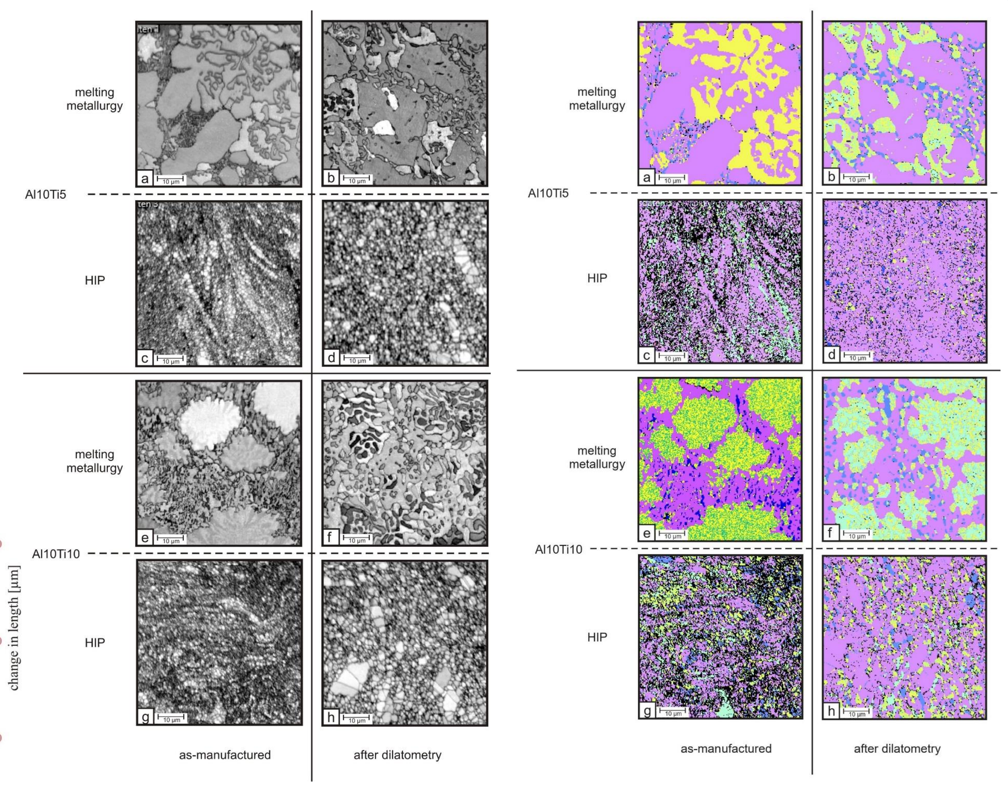


Fig.4 EBSD band contrast images (left) and images with colors of compositions Al10Ti5 (a–d) and Al10Ti10 (e–h); Detected phases are L_{12} (purple), A2/B2 (yellow), σ (turquoise), C14_Laves (blue), Full Heusler (green).

M. Reiberg, C. Duan, X. H. Li, E. Werner: High-temperature phase characterization of AlCrFeNiTi compositionally complex alloys; *MATER. CHEM. PHYS.* (2021) 2100163; DOI:10. 1016/j.matchemphys.2021.125272

Phase Transition Kinetics in Austempered Ductile Iron (ADI) with Regard to Mo Content

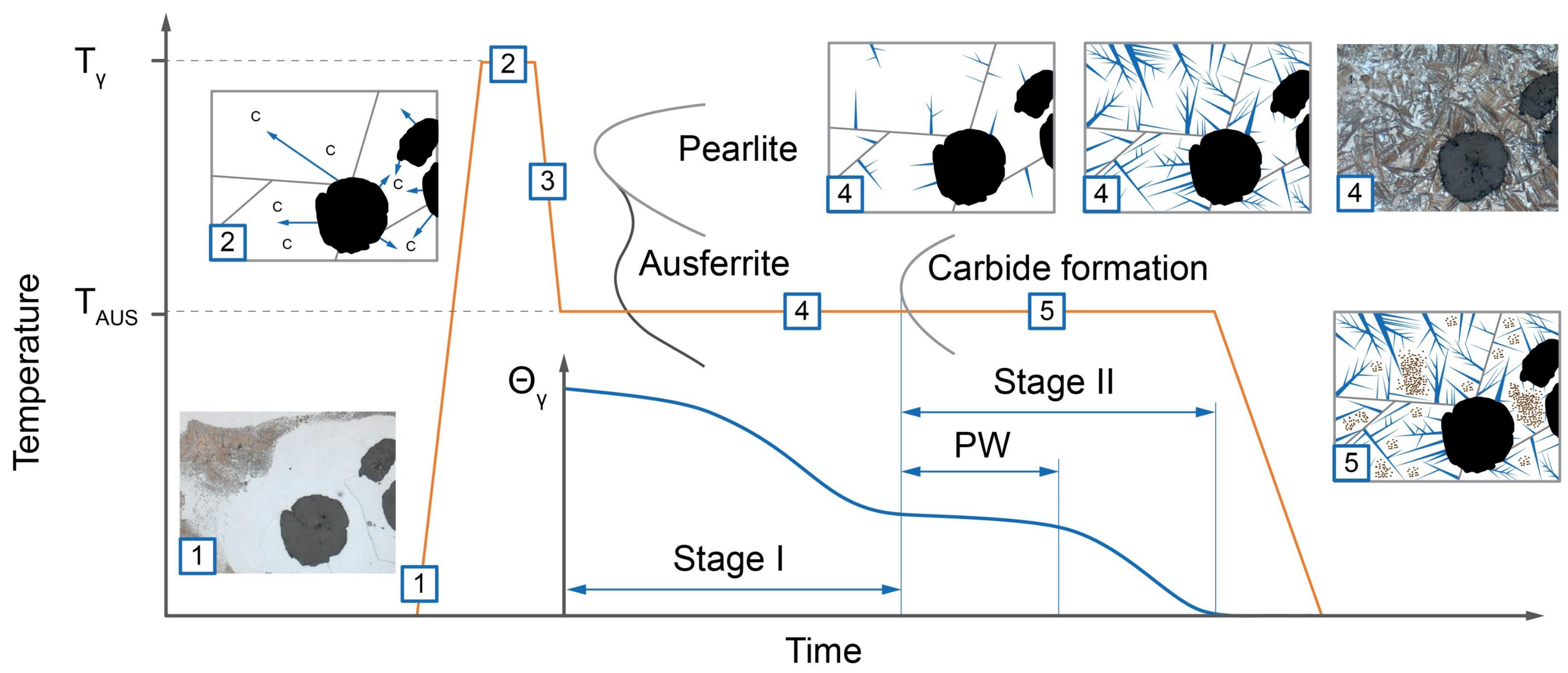


Fig. 5 The course of an ADI heat treatment is exemplarily shown. Starting from the initial state at (1) over heating to austenitization temperature *Tɣ* (2), followed by quenching (3) to annealing at austempering temperature *Taus* and subsequent cooling to room temperature. The evolution of austenite phase fraction ϕɣ during austempering is schematically depicted. Ausferritic microstructure is formed in the Stage I reaction (4) and followed by the decomposition of retained austenite (5) into carbides and ferrite (Stage II). As long as the loss in austenite fraction is small, the process window (PW) for industrial ADI heat treatmentis defined.

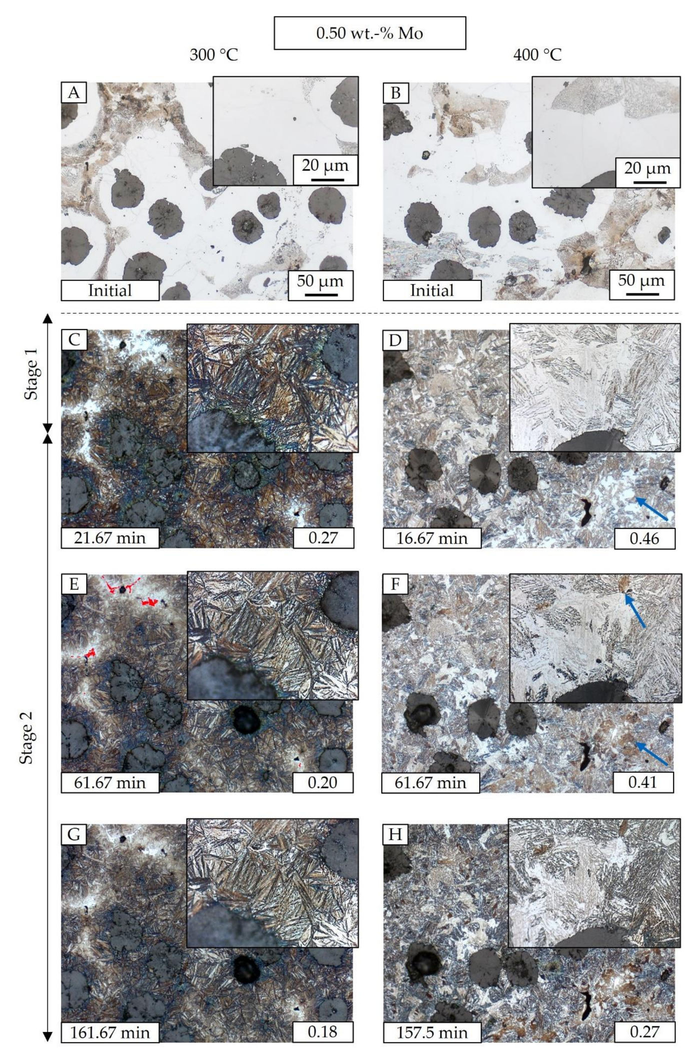


Fig. 6 The evolution of austenite decomposition over time for ADI with 0.5 mass% Mo is shown by Nital etched LOM images. The austempering temperatures are 300 ºC (c,e,g) and 400 ºC (d,f,h). The initial material state is depicted in (a,b). Carbides in (e) are colored in red.

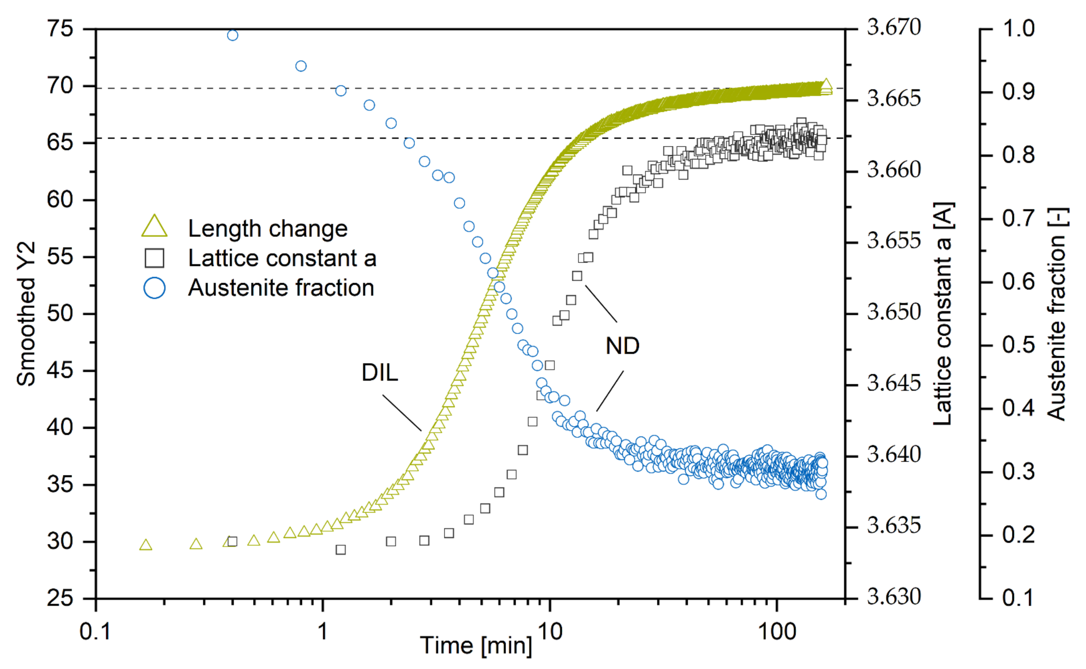


Fig. 7 The austempering temperature is 350 ºC. The evolution of retained austenite decomposition over time for ADI with 0.5 mass% Mo is analyzed with neutron diffraction and dilatometry. The macroscopic length change can be followed as well as the peak position shift due to C uptake. Neutron diffraction (ND) and dilatometry data (DIL) are marked, respectively.

M. Landesberger, R. Koos, M. Hofmann, X. H. Li, T. Boll, W. Petry and W. Volk: Phase Transition Kinetics in Austempered Ductile Iron (ADI) with Regard to Mo Content; *Materials* (2020), 13, 5266; doi:10.3390/ma13225266

Contact: Cecilia Solis
E-mail: cecilia.solis@hereon.de
Phone: +49.89.158860882

Weimin Gan
E-mail: weimin.gan@hereon.de
Phone: +49.89.158860766

FRM II
Forschungs-Neutronenquelle
Heinz Maier-Leibnitz