STRUCTURE AND GROWTH OF ω-Ti NANOPARTICLES IN β-Ti SINGLE CRYSTALS STUDIED BY ANOMALOUS X-RAY DIFFRACTION

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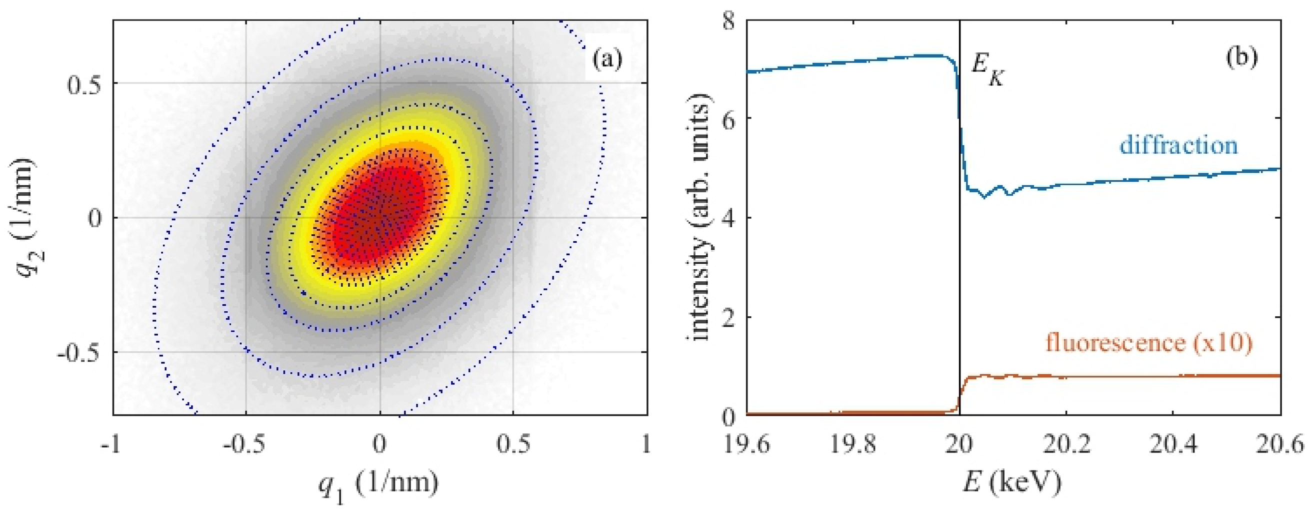
Abstract: Nanoparticles of hexagonal ω phase in bcc-Ti(Mo) single crystals (β phase) occur due to a diffusionless athermal β to ω transformation and they grow during follow-up ageing at elevated temperatures, while the alloying atoms (Mo in our case) are expelled from the nanoparticle volumes. We investigated the Mo content in growing ω nanoparticles by anomalous X-ray diffraction and demonstrate that the Mo expulsion from the ω phase is not full; a thin shell of a nanoparticles where the β to ω transformation is not complete still contains a considerable amount of Mo atoms.

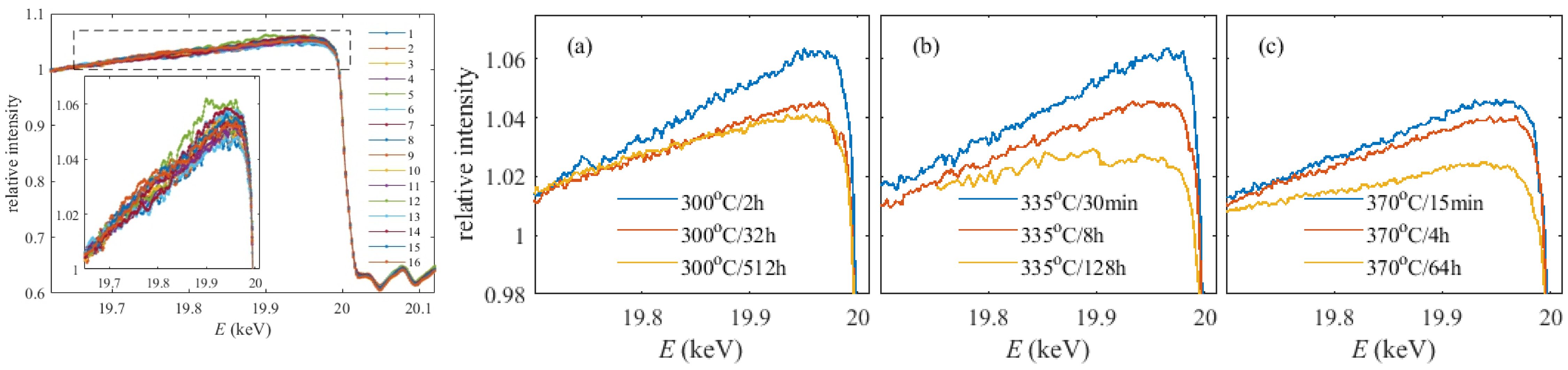
Samples: Single crystals of Ti+8%at.Mo were grown by a float-zone method in an optical furnace, the details of the growth are described in [1,2]. Individual samples were solution-treated (ST) at 860°C for 4 h in an evacuated quartz tube and quenched to water. Subsequently, different heat treated conditions were prepared by ageing in salt baths at temperatures 300°C, 335°C and 370°C for times ranging from 15 min to 512 h. All heat treatments were terminated by water quenching.

AXRD measurement: The anomalous x-ray diffraction (AXRD) experiments were carried out on beamline BM02 at ESRF, Grenoble, France. The measured energies were near the MoK absorption edge (E = 20.0 keV) in the energy range of 19.6 – 20.6 keV. The diffracted intensity was detected by a two-dimensional pixel detector. We acquired the images of the diffraction maximum $(44\bar{8}2)_\omega$ of the hexagonal ω lattice for the energies from the above mentioned range with the energy step of 2 eV, see [3] for details.

Examples of experimental data:



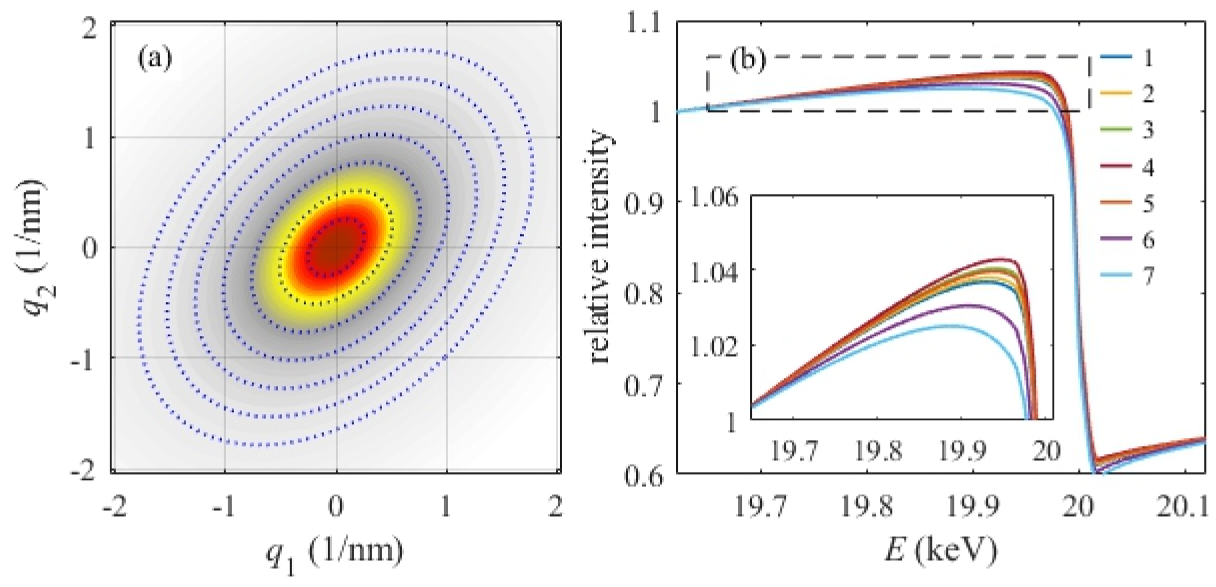
Left panel: One detector frame, the ellipses denote individual ranges of interest (ROIs). Right panel: energy dependence of total diffracted intensity (sum of all ROIs) and the fluorescence signal



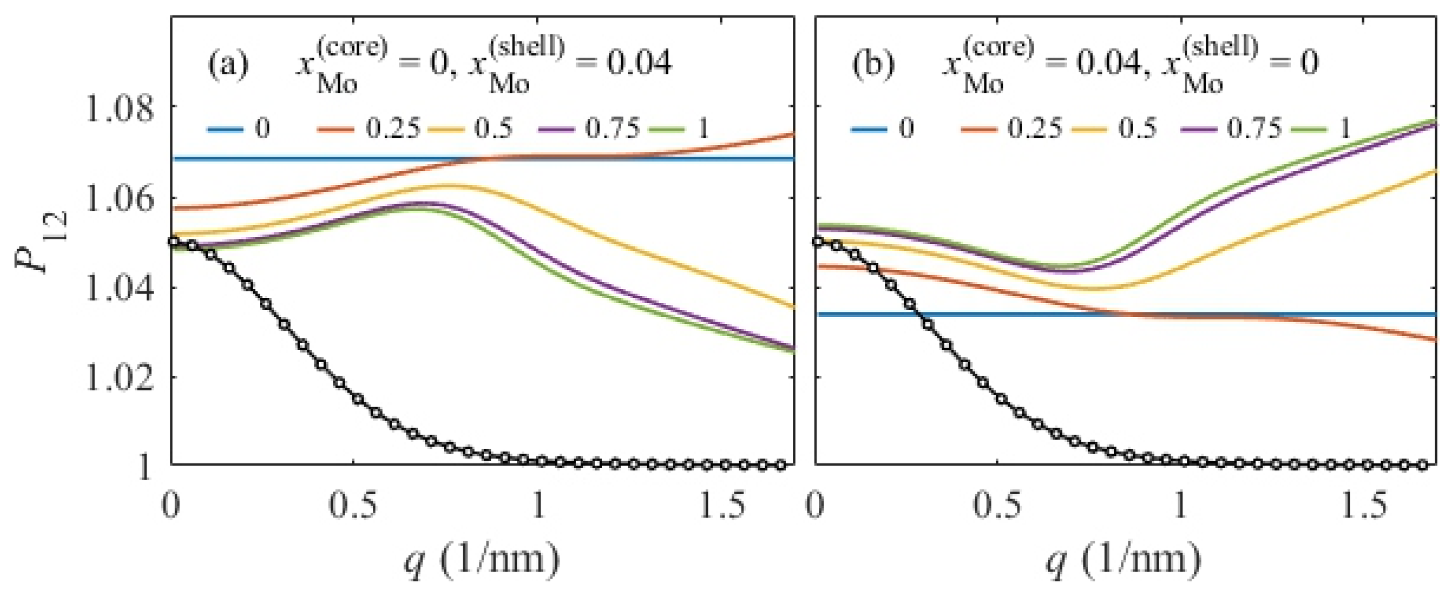
Left: The AXRD spectra of individual ROIS (parameters of the curves), sample aged at 370°C/64 h. The 1st ROI is inside the smallest ellipse, the last ROI is between the largest and second largest ellipses. The dashed rectangular region is enlarged in the inset. Right: The AXRD spectra of intensities integrated over the detector window of the whole sample series, the panels (a), (b), and (c) display the spectra of samples aged at 300°C, 335°C, and 370°C, respectively. The intensities are normalized to their values at 19.6 keV.

Simulations:

Simulation model: the ω-particles are core/shell uniaxial ellipsoids with radii R_L and R_V, the ellipsoid axis is along $\langle 111\rangle_\beta \parallel [0001]_\omega$. The shell thickness is d. The Mo concentrations in core and shell are x_{core}, x_{shell}. The b→ω transformation in the shell is assumed incomplete with the completeness factor $p\in[0,1]$

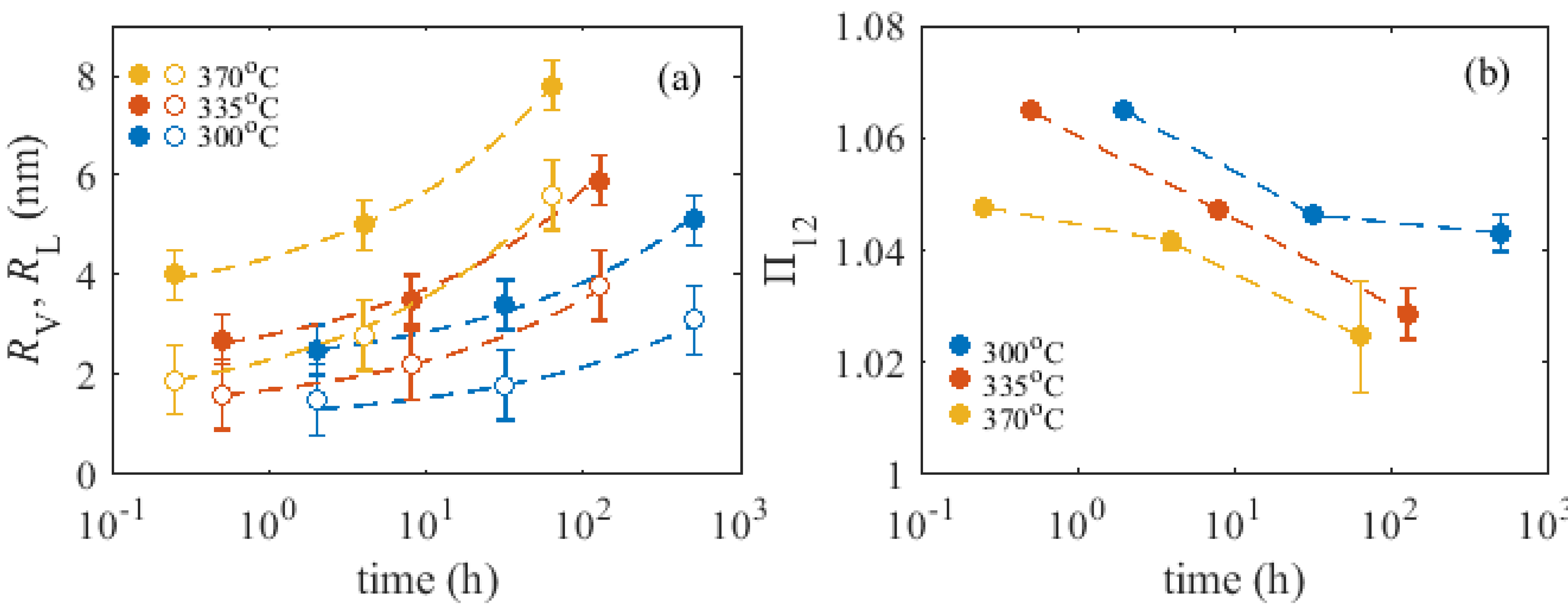


(a) Simulated diffraction maximum $(44\bar{8}2)_\omega$, R_V = 6 nm, R_L = 4 nm, (b) The AXRD spectra of individual ROIS, the AXRD intensities are normalized to their values at 19.6 keV

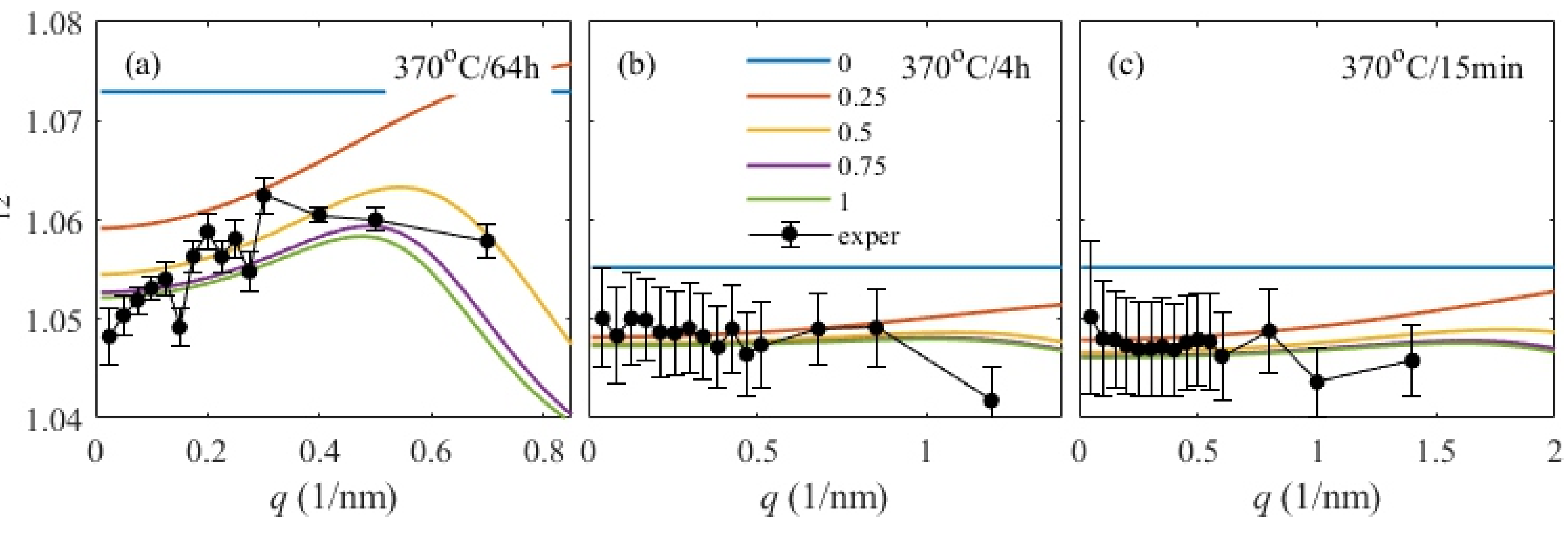


Ratio P_{12} of integrated intensities at energies 19.95 keV and 29.6 keV calculated for Mo atoms present in the shell [panel (a)] and in the core [panel (b)], and various parameters p (parameter of the curves). The black line with dots displays the q-dependence of the diffracted intensity; the vertical axis for this curve is not in scale

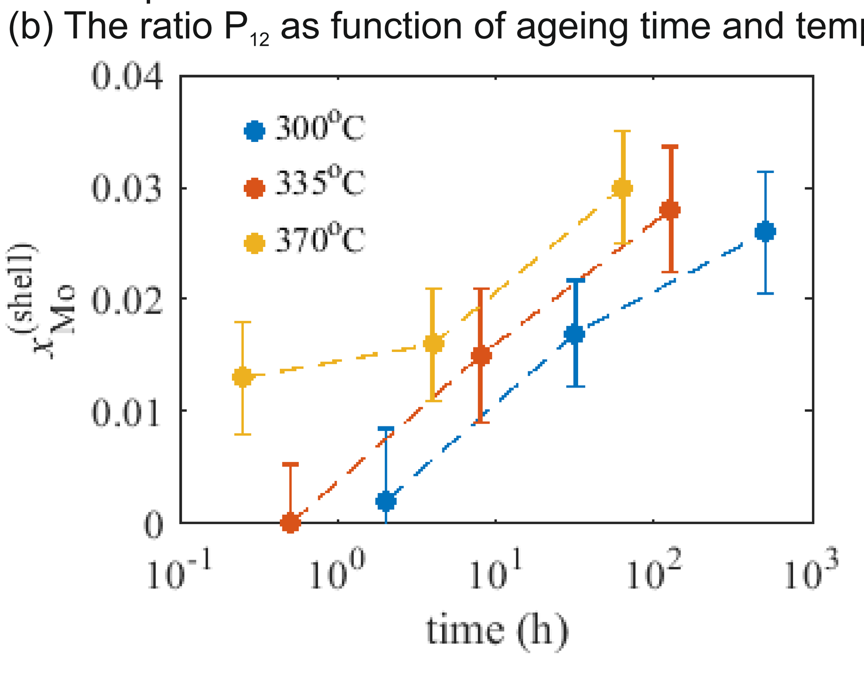
Results:



Vertical (R_V – full) and lateral radii (R_L – open circles) as functions of the ageing time t and temperature. The dashed lines are the $t^{1/3}$ fits following from the LSW model
(b) The ratio P_{12} as function of ageing time and temperature



Measured (black dots) and simulated (color lines) q-dependences of the intensity ratio P_{12} for samples 370°C/64h (a), 370°C/4h (b), and 370°C/15min (c). The parameter of the simulated curves is the completeness factor p



The Mo densities x_{shell} in the particle shells as functions of the ageing time and temperature

Summary:

The Mo density in the shell is larger than in the particle core. During ageing, the thickness of the shell remains almost constant, however the Mo density in the shell increases and it can reach almost the half of the Mo concentration in the β-Ti matrix far from the particles.

[1] Šmilauerová, J.; Harcuba, P.; Pospíšil, J.; Matěj, Z.; Holý, V . Acta Materialia **61**, 6635 (2013).
[2] Šmilauerová, J.; Pospíšil, J.; Harcuba, P.; Holý, V.; Janeček, M. Journal of Crystal Growth **405**, 92 (2014)..
[3] Šmilauerová, J.; Harcuba, P.; Janeček, M.; Holý, V. Crystals, **9**, 440 (2019).

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