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AFM AND MÖSSBAUER SPECTROMETRY INVESTIGATION OF CRYSTALLIZATION PROCESS IN Fe-Mo-Cu-B ALLOY

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Aim

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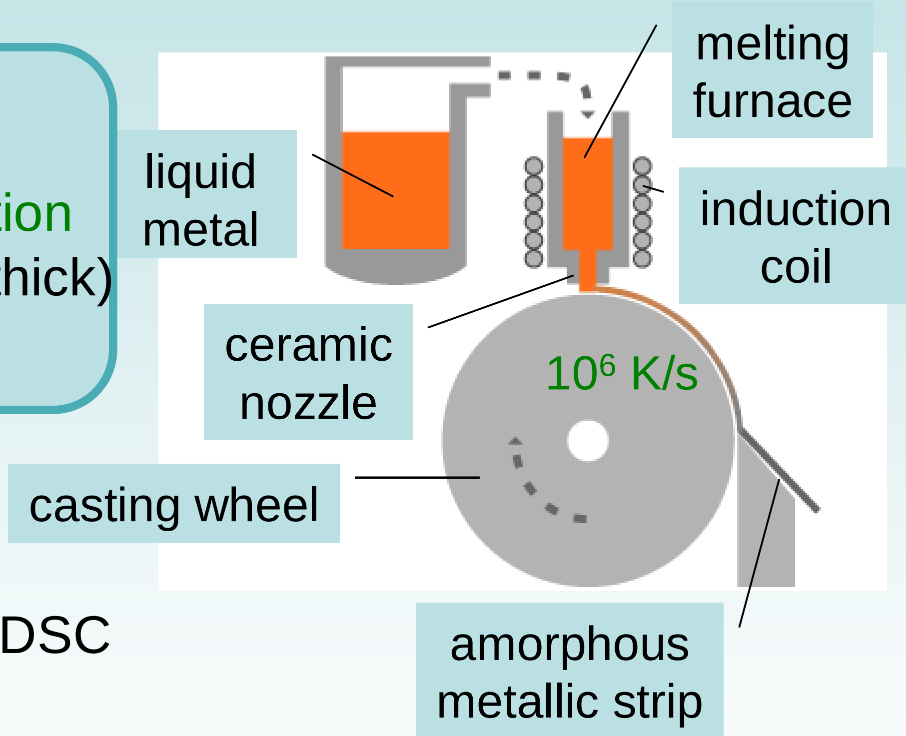
- evolution of surface crystallization of amorphous metallic glass

Sample: $Fe_{79}Mo_8Cu_1B_{12}$

- planar-flow casting rapid solidification (ribbon: 10 mm wide, **20 – 22 μm** thick)
- controlled annealing in vacuum



Methods of investigation:

- differential scanning calorimetry – DSC
- X-ray diffraction – XRD
- atomic force microscopy – AFM
- conversion electron Mössbauer spectroscopy – CEMS
- transmission Mössbauer spectroscopy – TMS

Nanocrystalline alloys

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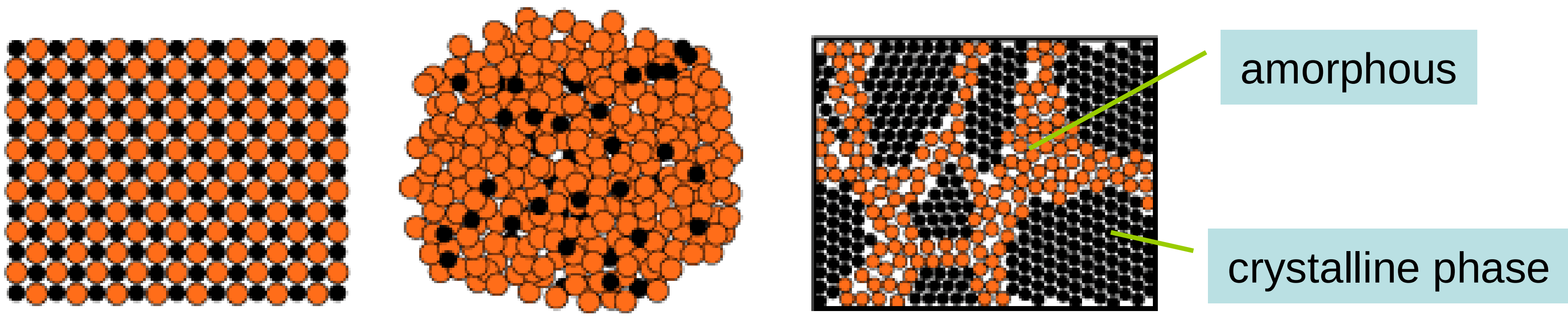
Features:

- two-phase structural and magnetic behaviour
- high saturation magnetization and permeability

- nanocrystalline grains
  - origin of soft magnetic properties
  - thermal stabilization of the structure

Technical applications:

- power transformer, magnetic heads, sensors, magnetic shielding

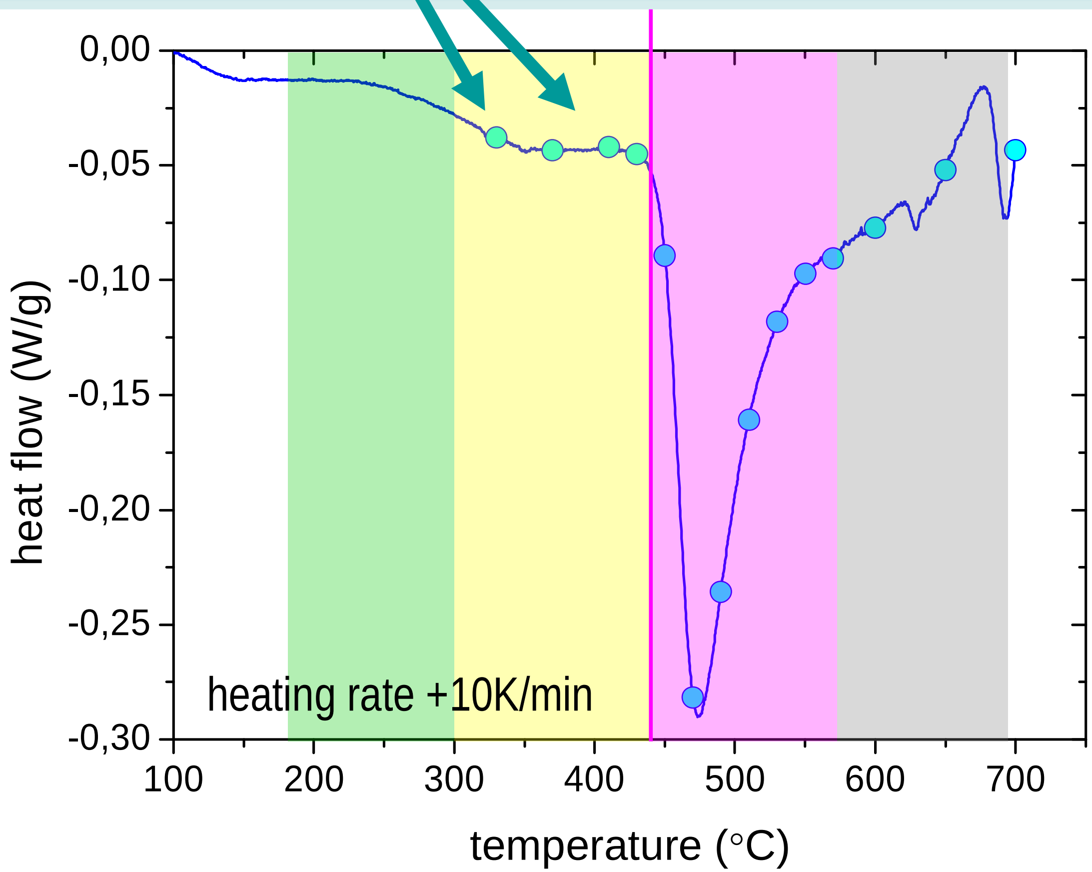


Conventional crystalline microstructure, amorphous structure, nanocrystalline structure

Preparation of samples

- annealing in vacuum
- annealing temperatures: according to DSC

$T_{x1}^{*} \approx 445$ °C



structural relaxation

diffusion-like precrystallization effects

normal grain-growth-like formation of α-Fe nanocrystallites in amorphous matrix

diffusion controlled grain-growth and nucleation of further crystalline phases

*E. Illeková *et al.*, J. Magn. Magn. Mater. **304** (2006) e636–e638.

XRD ($Fe_{79}Mo_8Cu_1B_{12}$) Top/Bottom

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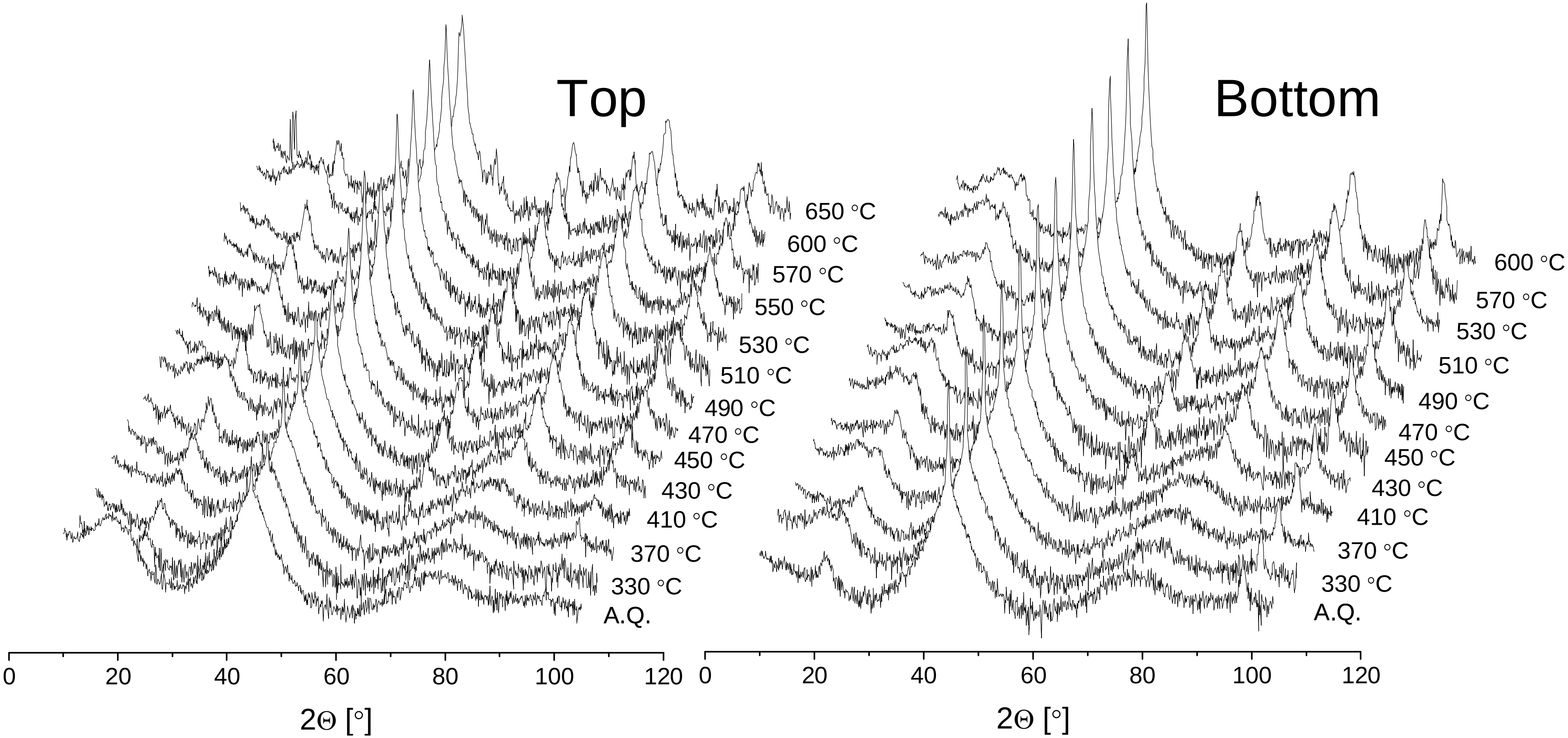
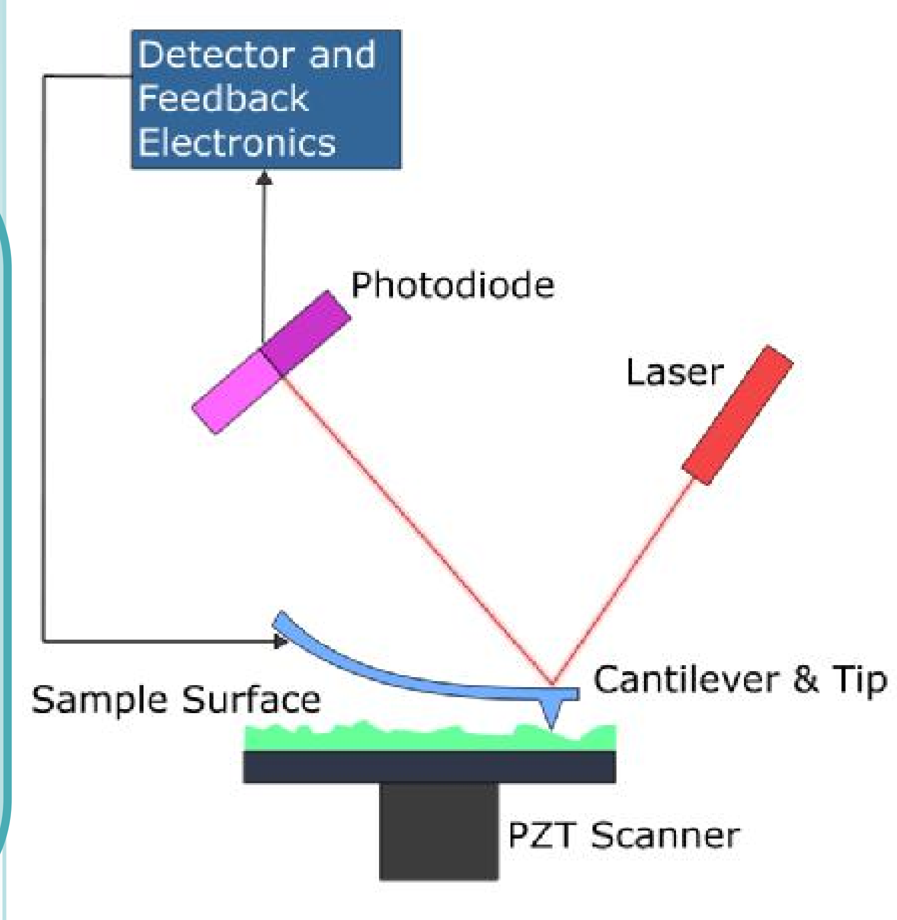
- crystallization at the bottom side of the ribbon is more developed as on the opposite side
- T_{x1} approx. 410 – 430 °C
- sample is not fully amorphous in A.Q. state



Atomic force microscopy

Principle:

- an atomically fine tip scans the surface of the sample
- the attractive or repulsive force between the tip and the sample leads to a deflection of the cantilever
- a laser beam measures the displaced of the probe tip
- the feedback system works to keep the tip-sample force steady



Atomic force microscopy

The AFM can operate in three different ways:

Contact mode

- An extremely low force (~10^{-9} N) is maintained on the cantilever, thereby pushing the tip against the sample as it rasters. The contact force causes the cantilever to bend to accommodate changes in topography.

Non-contact mode

- The cantilever oscillates above the sample's surface and is affected by surface/tip (van der Waals) forces.

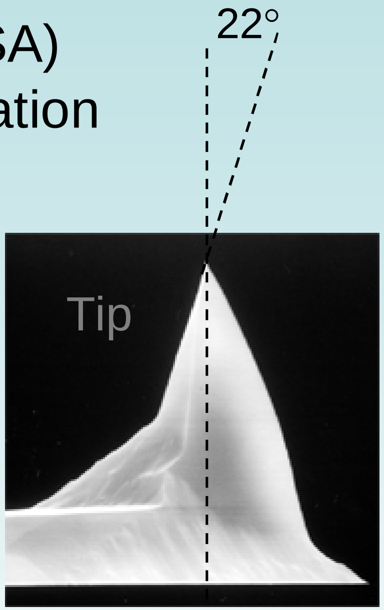
Tapping mode

- The AFM tip taps the sample surface during the closest point of approach of an oscillation cycle. The interaction with the sample surface changes the vibration frequency.

AFM - Experimental details

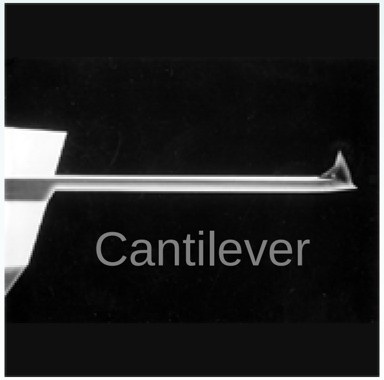
Microscope: AFM Explorer (ThermoMicroscopes, USA)

- the microscope was placed on a pneumatic antivibration desk, under a damping cover



Probes: Veeco 1650-00

- Material: Si
- Tip Height: 10 – 15 μm
- Tip Radius: **<10 nm**



Measurements:

- in air and at room temperature
- in a Non-contact mode

- the size of the scanned area was 2 x 2 μm and the resolution of image was 300 x 300 pixels

AFM

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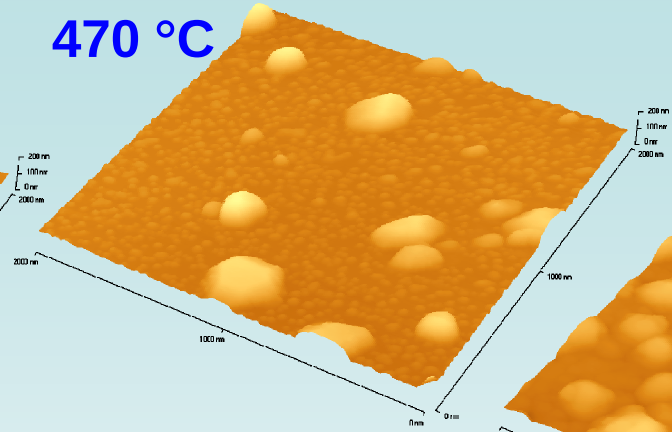
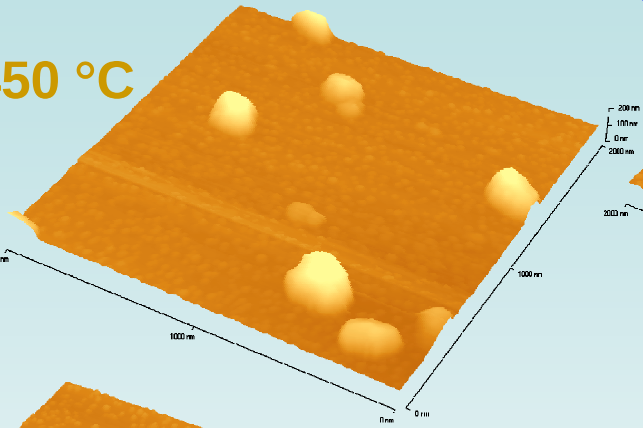
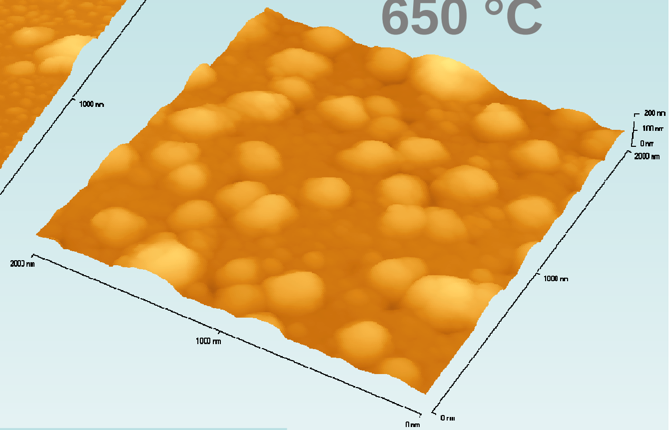
470 °C



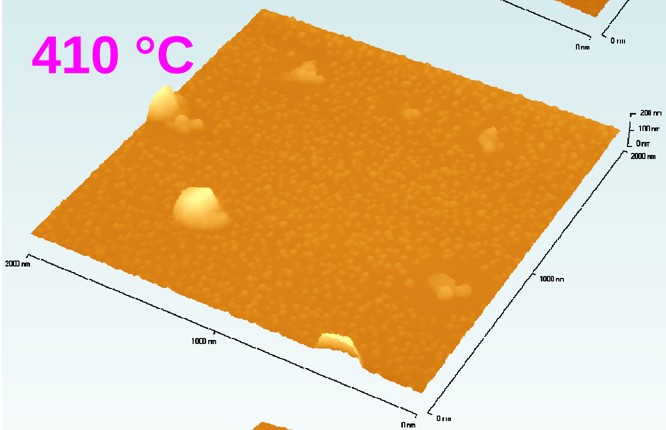
450 °C



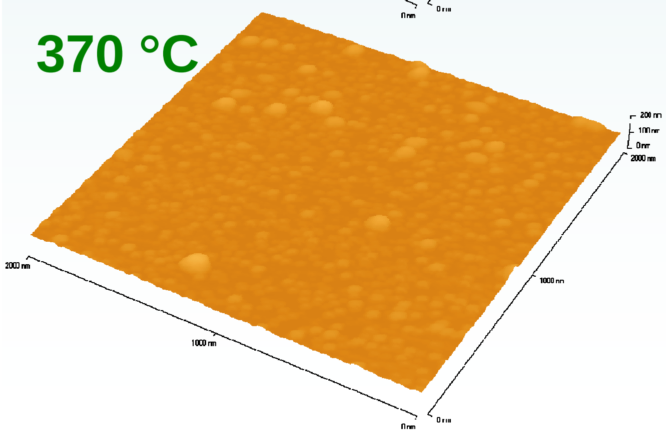
650 °C

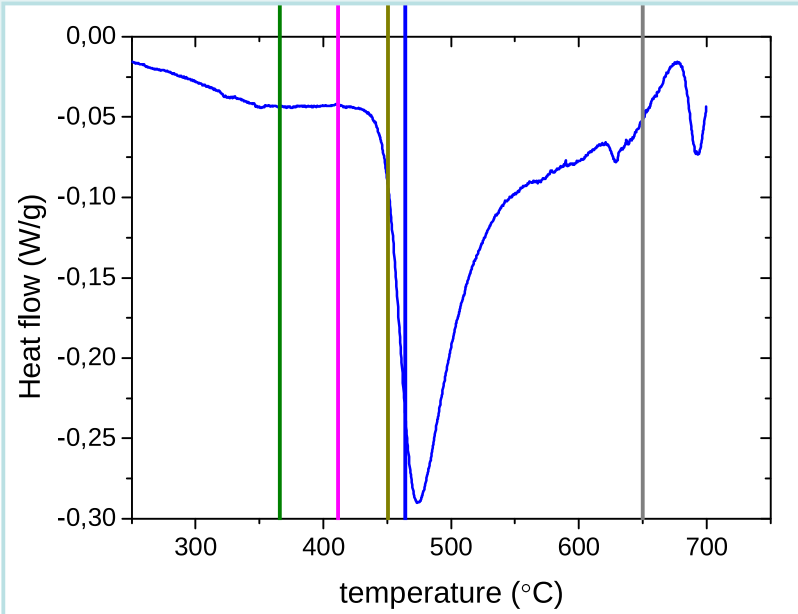


410 °C



370 °C





Differential Scanning Calorimetry

AFM ($Fe_{79}Mo_8Cu_1B_{12}$) A.Q., Top

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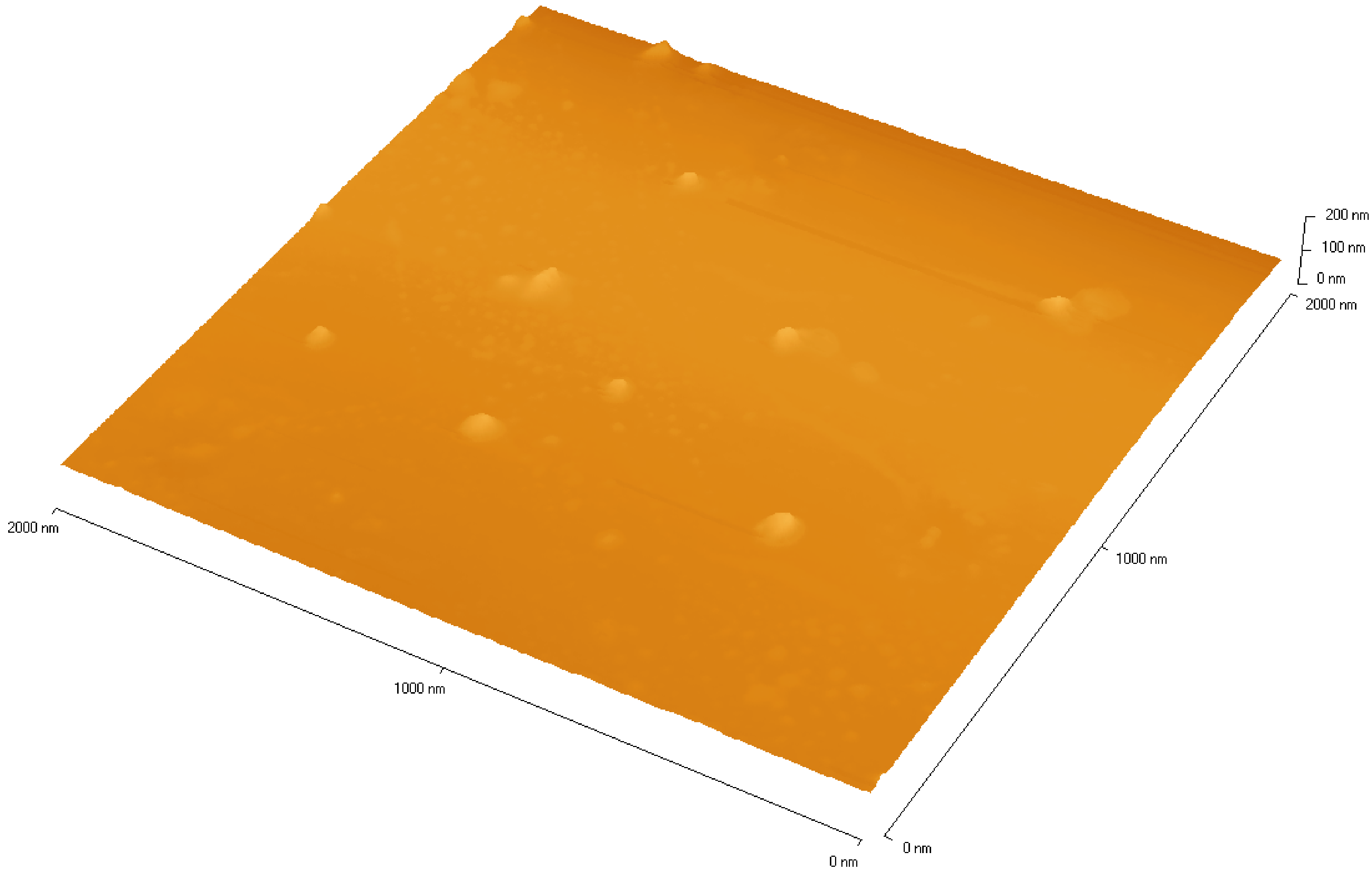

AFM ($Fe_{79}Mo_8Cu_1B_{12}$) 370 °C

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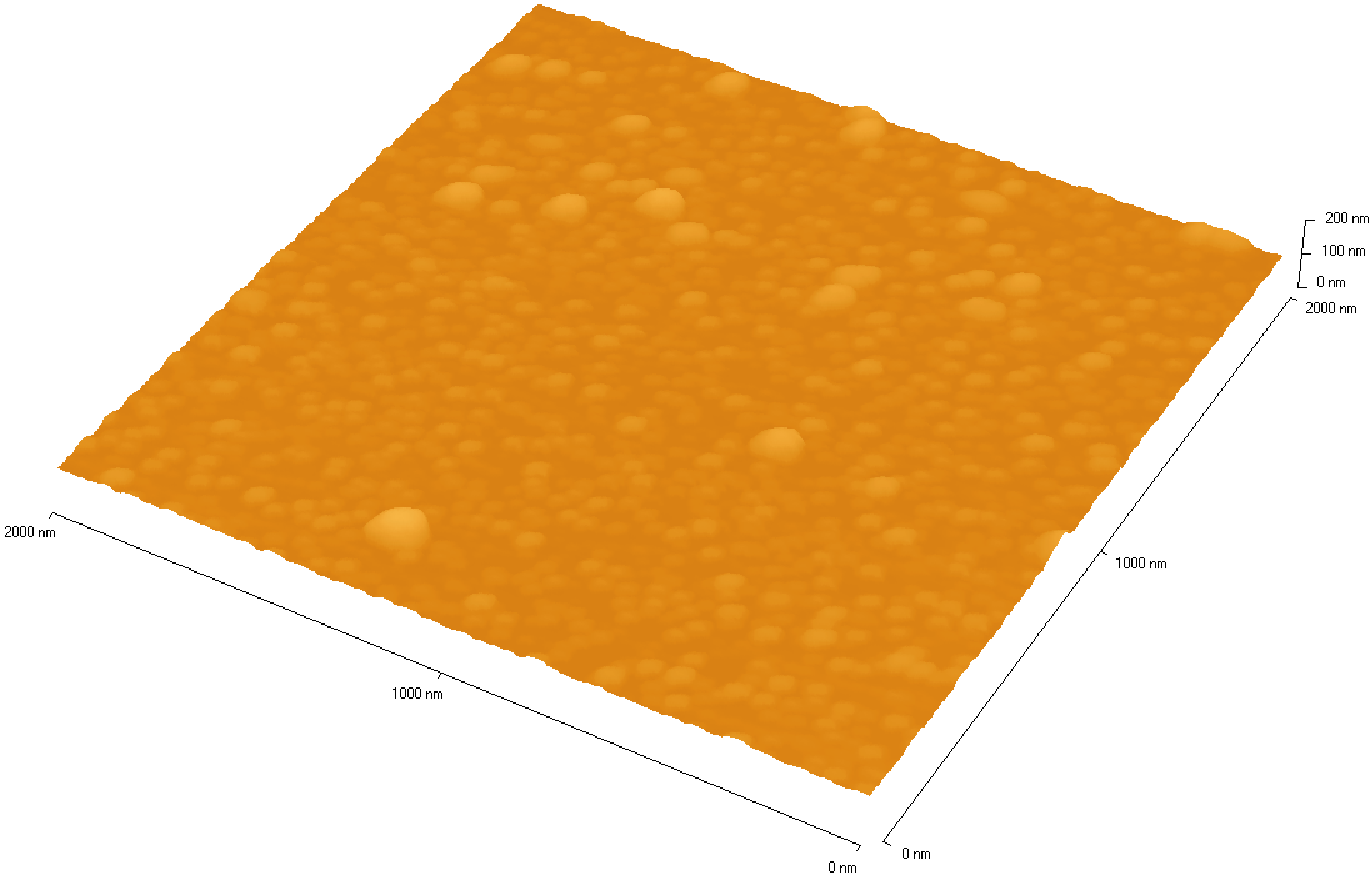

AFM ($Fe_{79}Mo_8Cu_1B_{12}$) 410 °C

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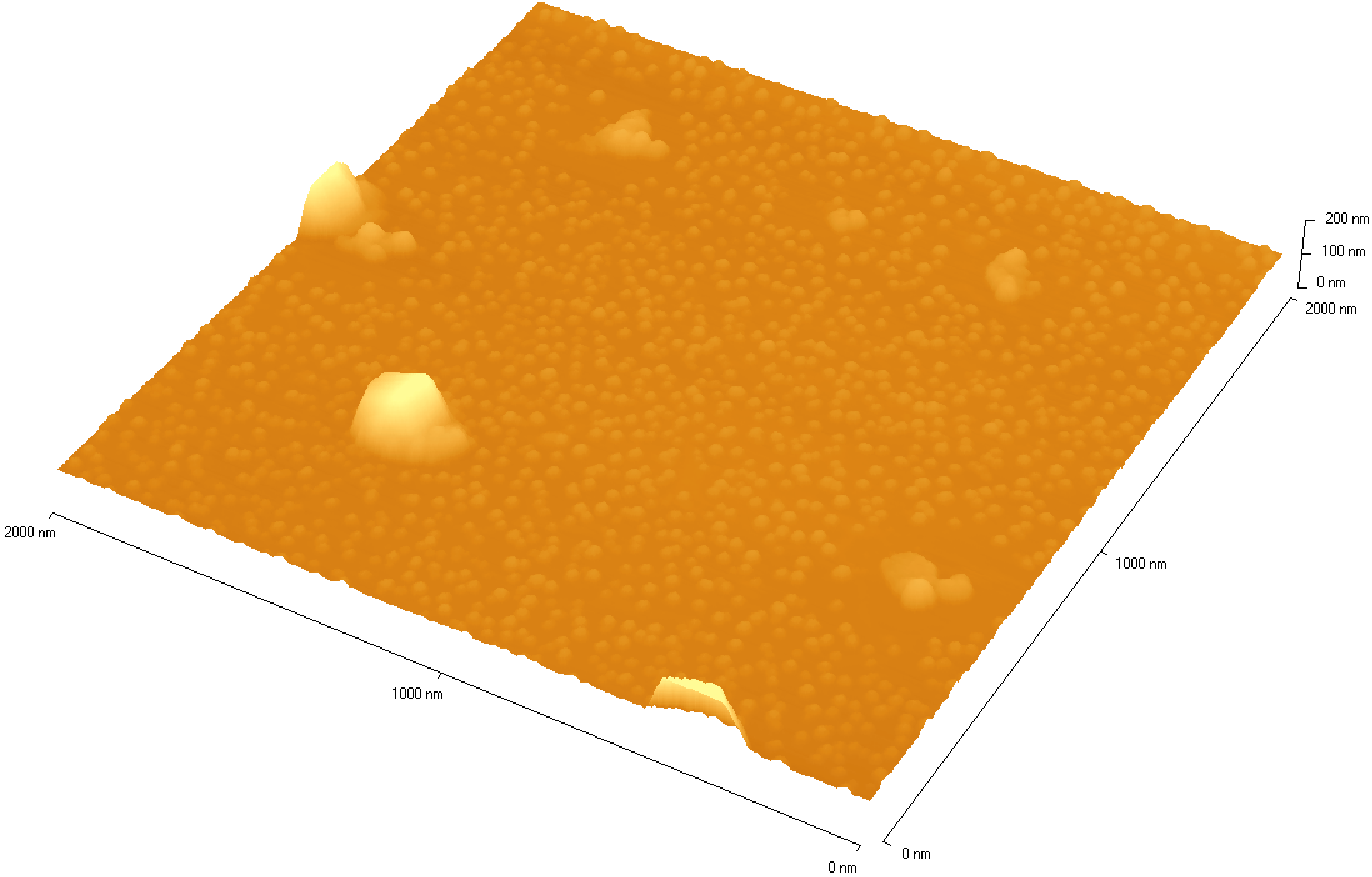
avg. height of o ~ 13 nm; O ~ 26 – 119 nm



AFM ($Fe_{79}Mo_8Cu_1B_{12}$) 450 °C

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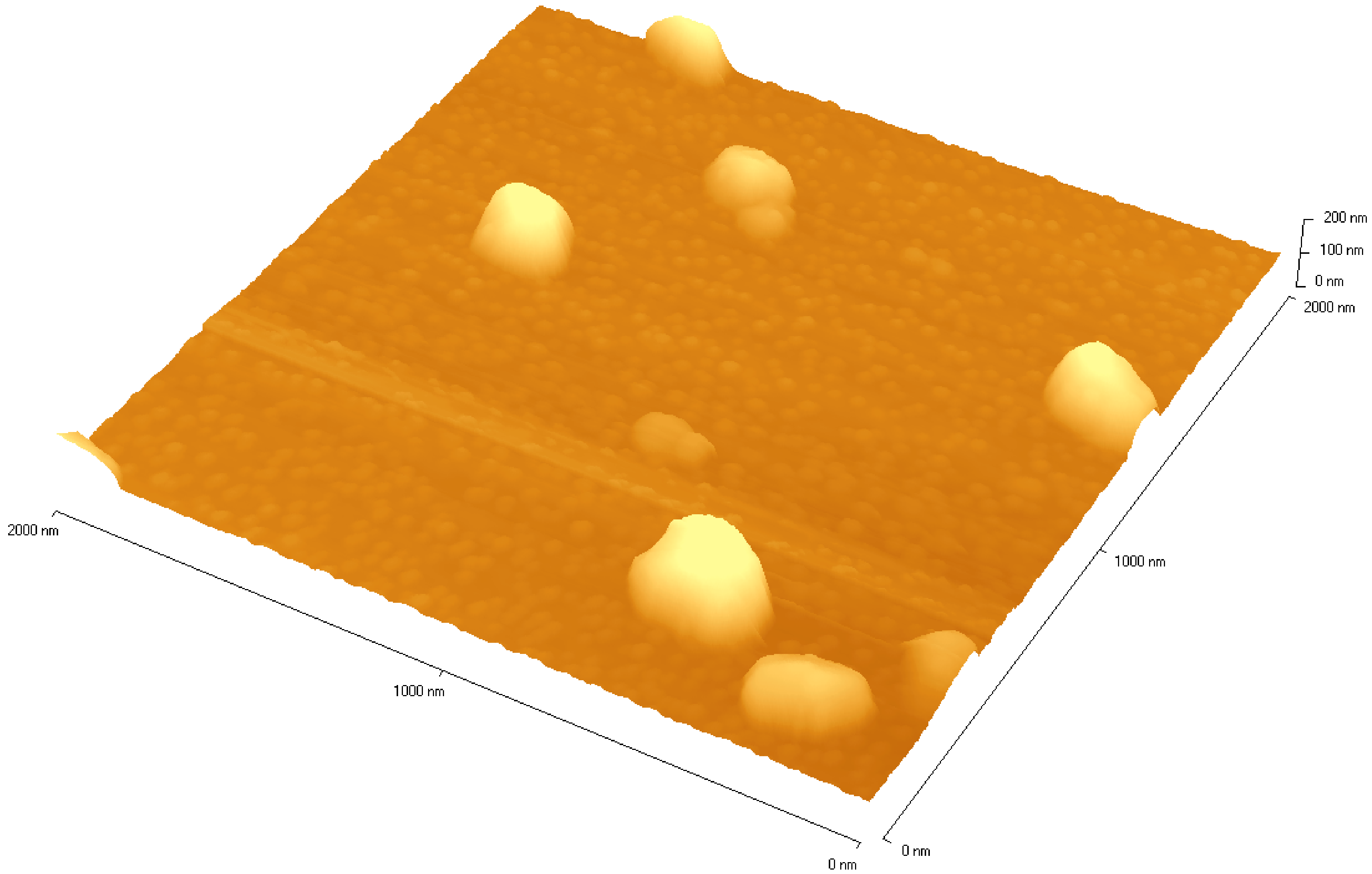
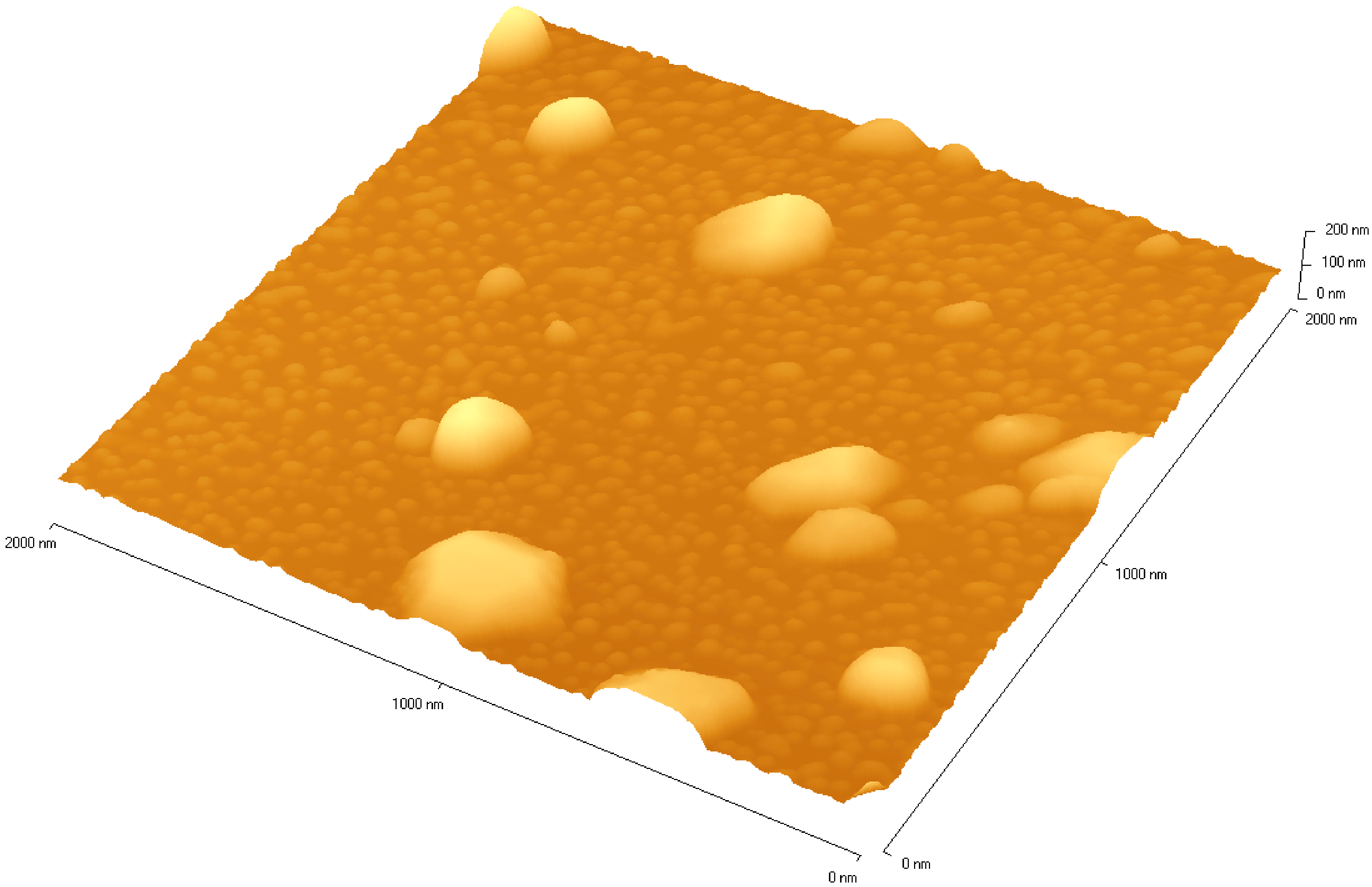


AFM ($Fe_{79}Mo_8Cu_1B_{12}$) 470 °C



AFM ($Fe_{79}Mo_8Cu_1B_{12}$) 650 °C

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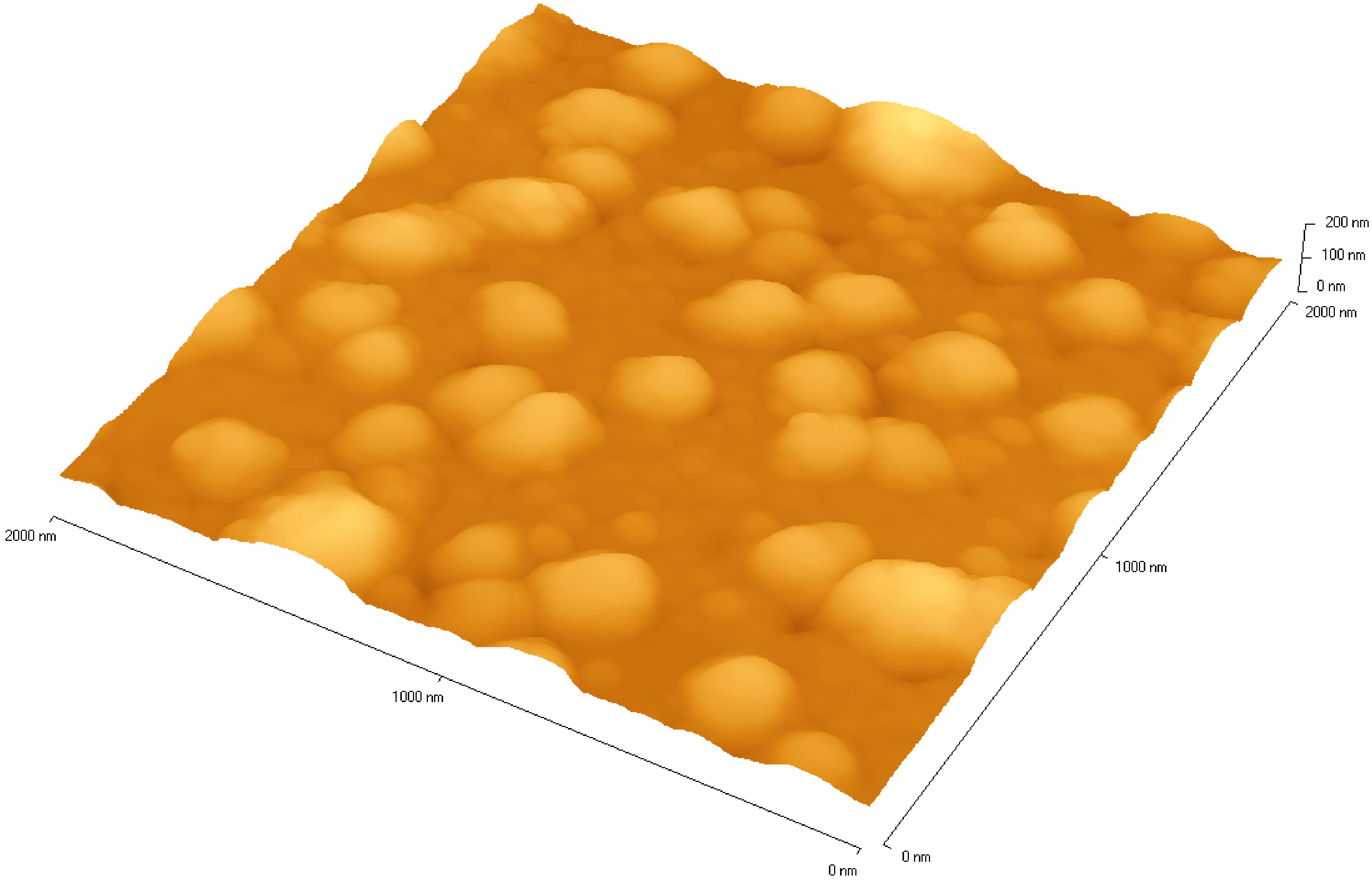
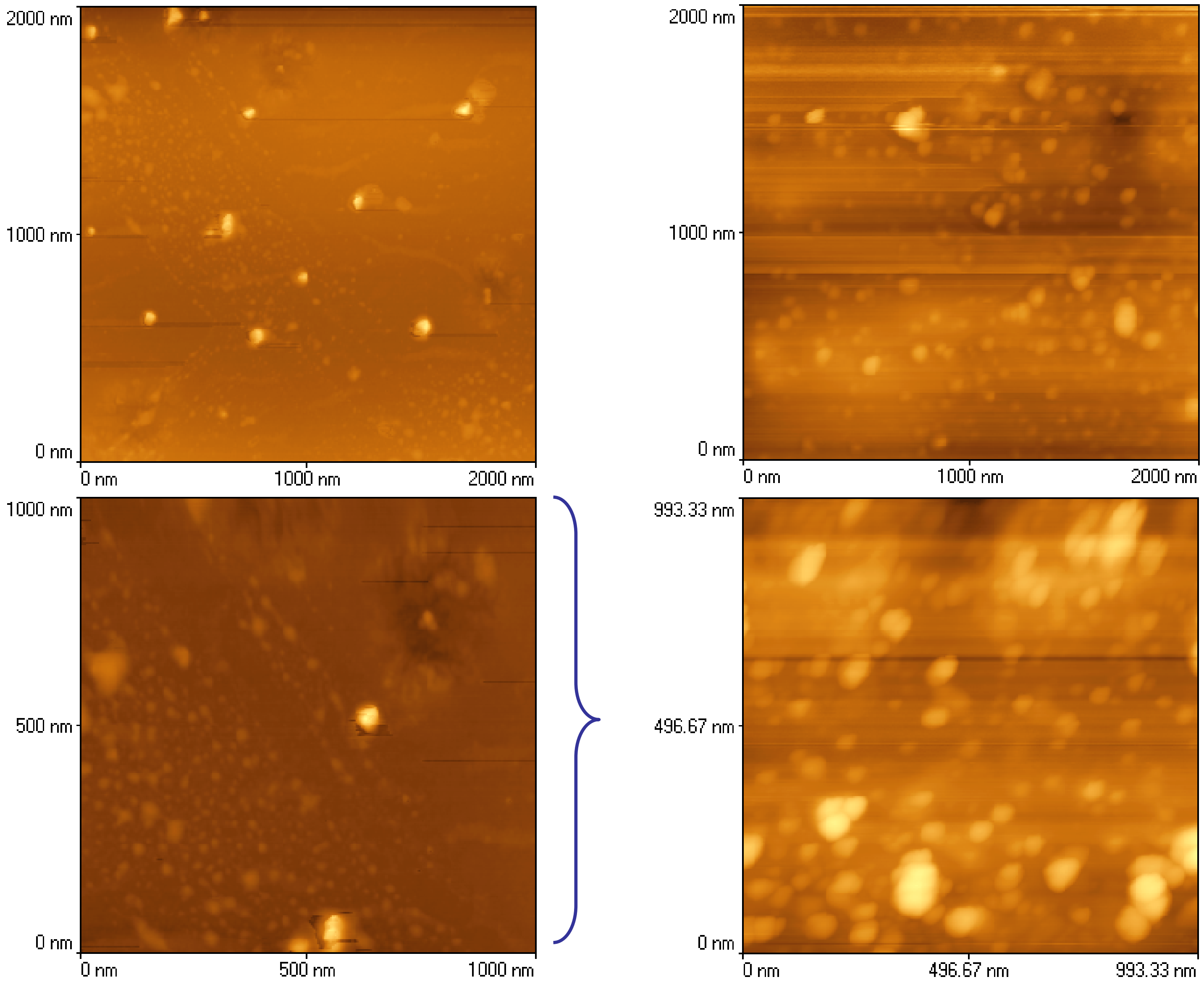


AFM ($Fe_{79}Mo_8Cu_1B_{12}$) A.Q., Top/Bottom

air side | wheel side



Zoom
1x1 μm

XRD, CEMS ($Fe_{79}Mo_8Cu_1B_{12}$) A.Q .

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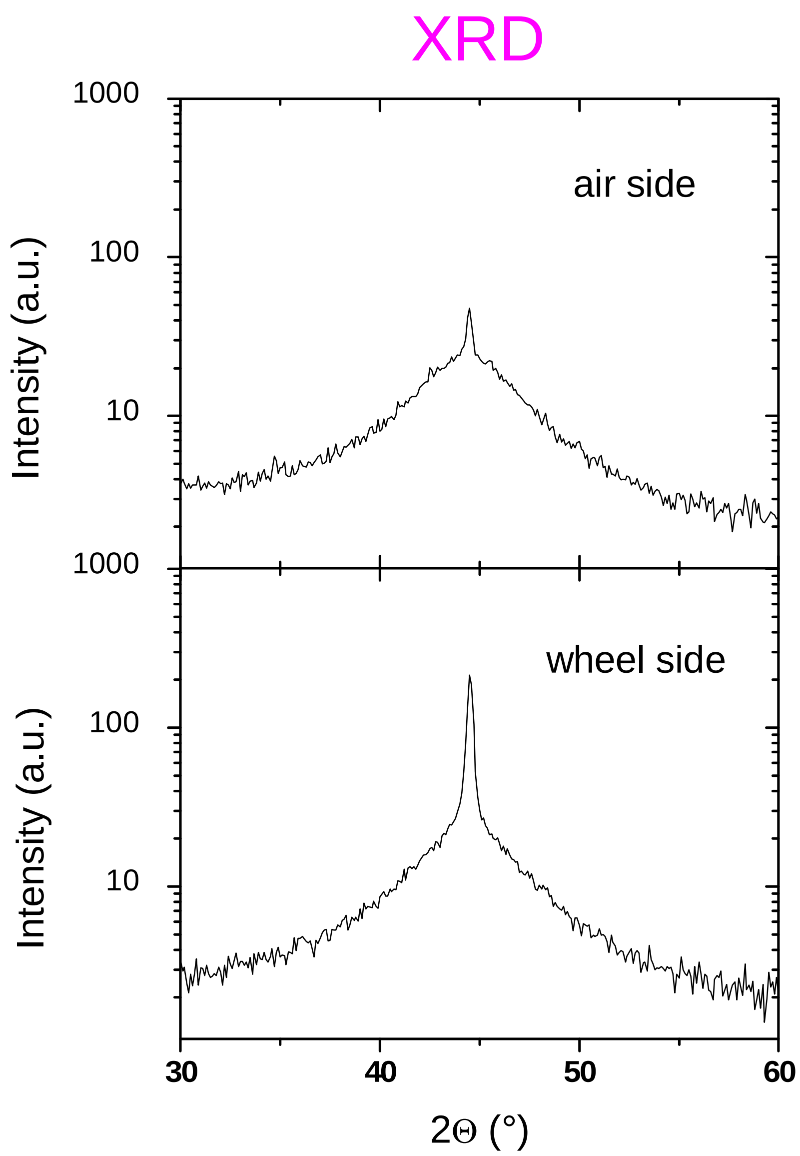
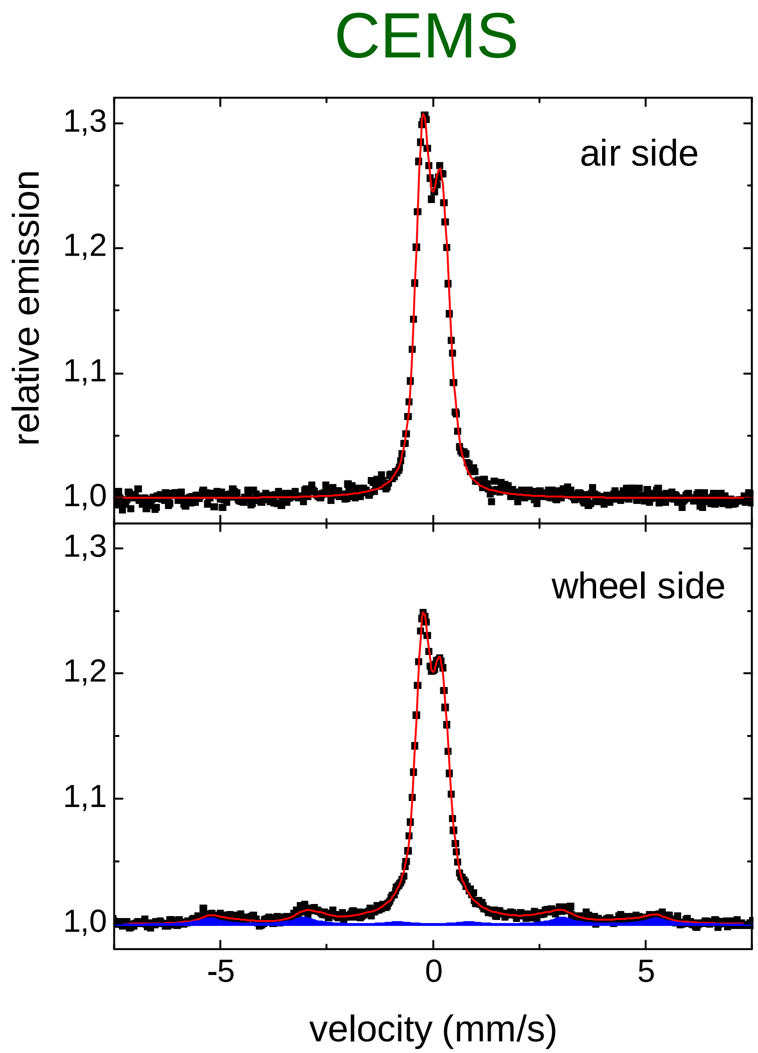
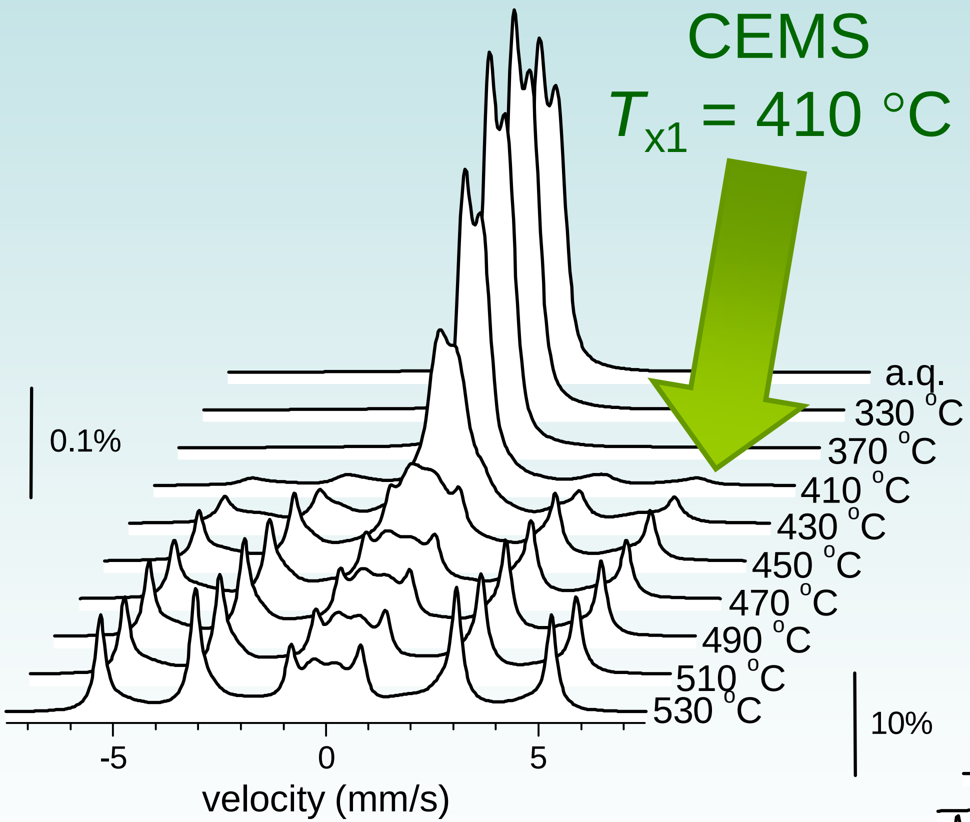
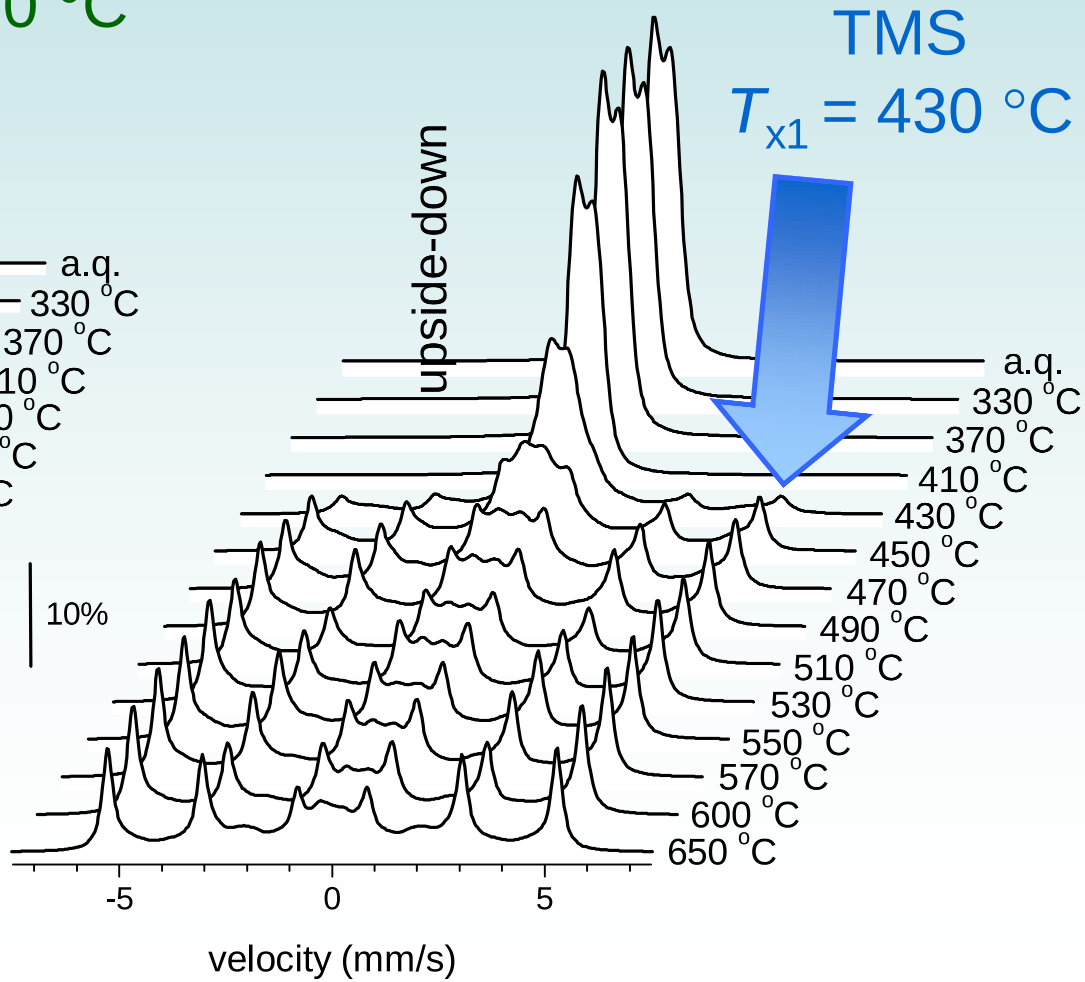


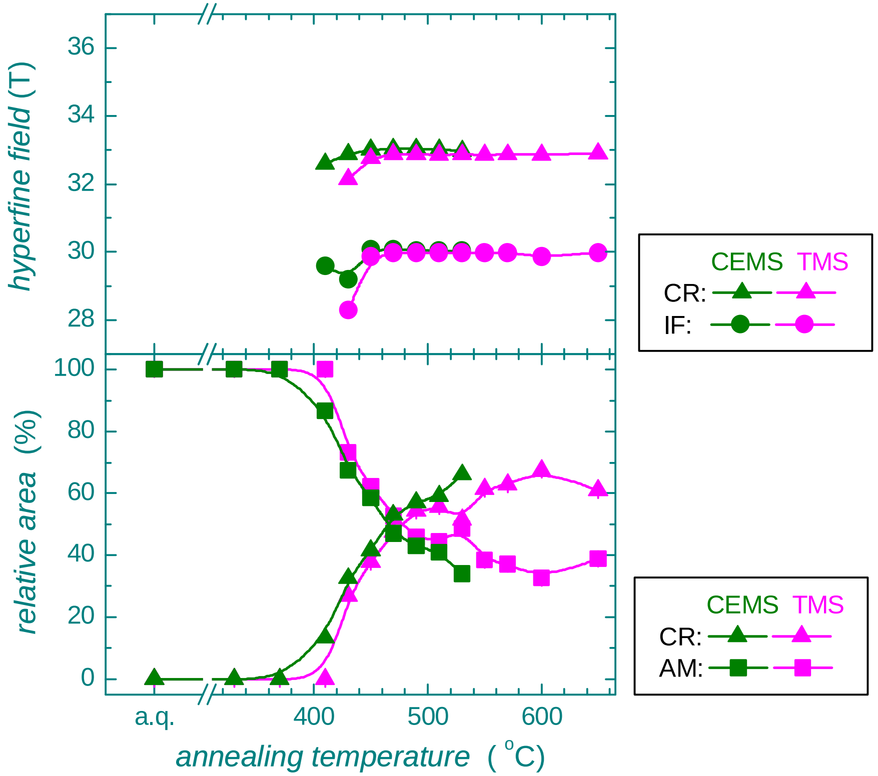


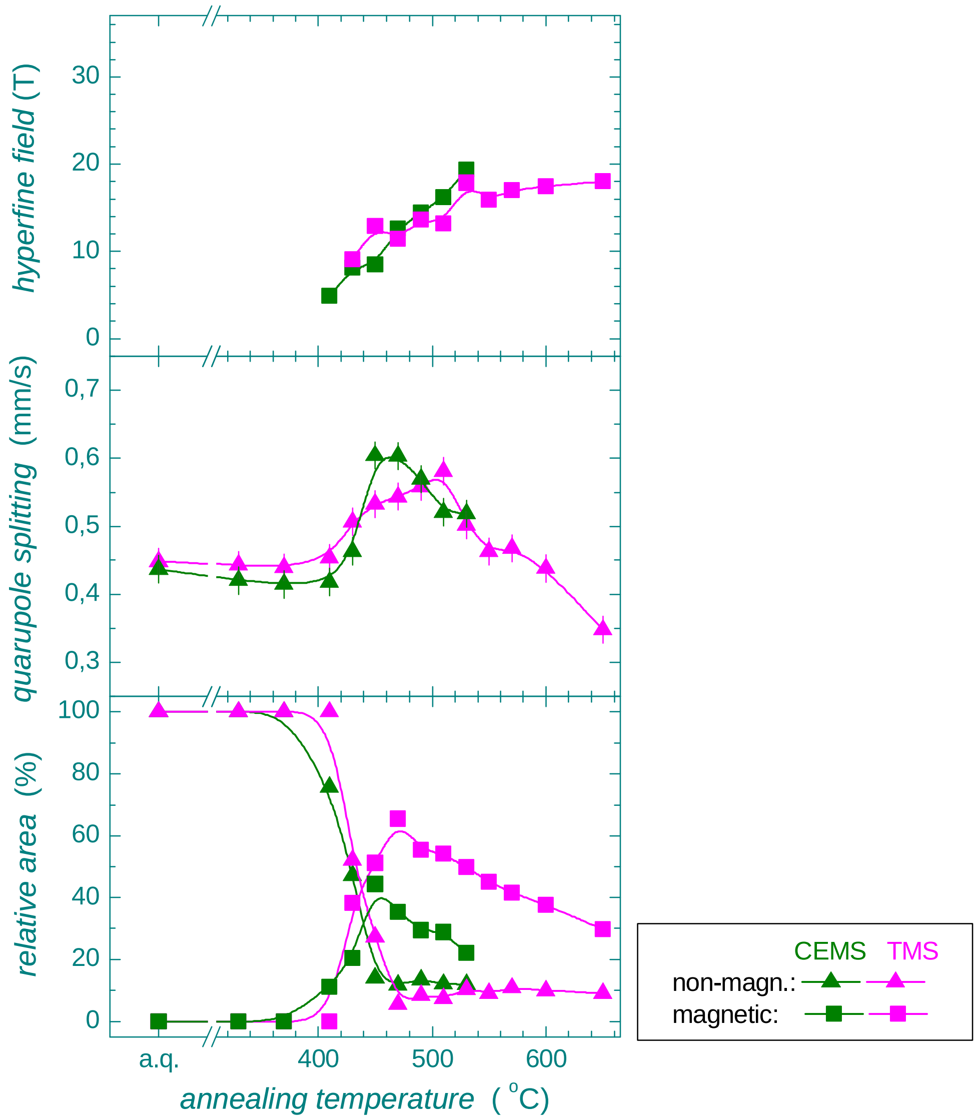
CEMS, TMS: Structure evolution





CEMS, TMS ($Fe_{79}Mo_8Cu_1B_{12}$) CR/AM





Conclusions:

- Sample is not fully amorphous even in A.Q. state
- Crystallization at the bottom (wheel) side of the ribbon is more developed as on the opposite (air) side
- Crystallization starts earlier in the surface areas of the ribbon than in the bulk region
- Onset of crystallization on surface ~ 410 °C
- as-quenched (amorphous) and nanocrystalline $Fe_{79}Mo_8Cu_1B_{12}$
  - combined hyperfine interactions:
    - non-magnetic regions
    - magnetic regions
  - combined structure:
    - (nano) crystallites
    - residual amorphous matrix

Future objectives:

- Calculate the grain size at different temperatures of annealing from X-ray diffraction images and compare it with AFM observations

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