The origin of clinopyroxene – titanomagnetite clustering during crystallization of synthetic trachybasalt

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References
Hammer et al. 2010 https://doi.org/10.1130/G30601.1
Pontesilli et al. 2019 https://doi.org/10.1016/j.chemgeo.2019.02.015

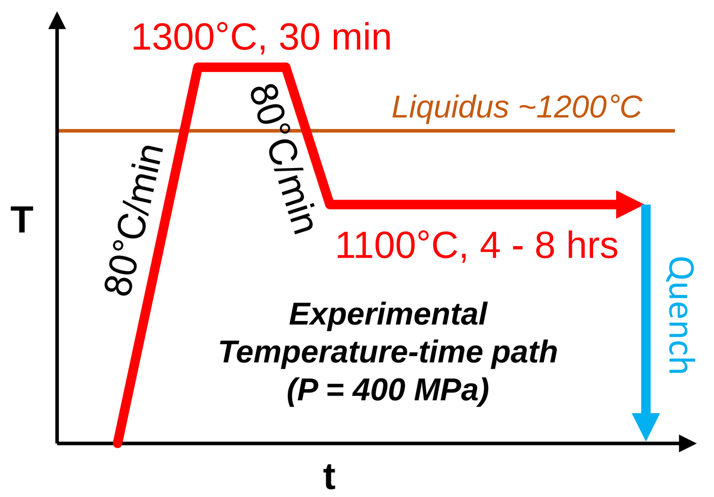
FWF Der Wissenschaftsfonds.

Introduction

Crystal clustering impacts rheology and differentiation in magmatic systems, and also offers insights into nucleation processes. Electron backscatter diffraction (EBSD) is ideal for studying interactions between crystals at interfaces via their crystallographic orientation relationships (CORs). Clustering between Clinopyroxene (Cpx) and titanomagnetite (Timt) is well known in natural and experimental samples and has been attributed to heterogeneous nucleation *(Hammer et al. 2010)*. Clusters formed in time series experiments on synthetic trachybasaltic melt were studied using EBSD to understand the cause of clustering and investigate the effect of water content and annealing time at constant T on cluster formation and properties.

Experimental details

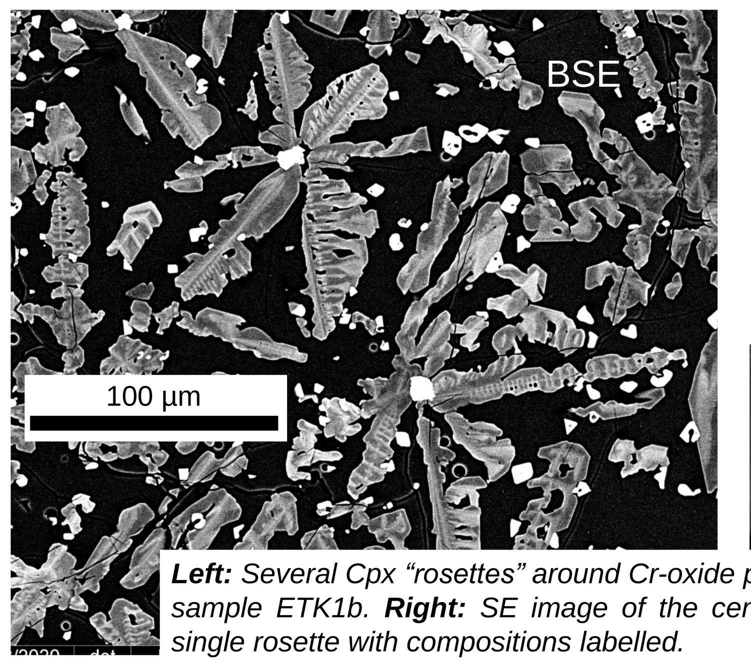
Starting glass was synthesized from synthetic oxides and carbonates. The experimental composition is trachybasaltic, chosen to correspond to one of the most primitive compositions erupted by Mt. Etna, Sicily. Ca. 2g of homogenized powdered glass was placed into Pt-capsules in a non-end-loaded piston cylinder apparatus using standard ¾" talc-pyrex-graphite-MgO assemblies, yielding an oxygen fugacity close to NNO+2. Capsules were either "dry" (drying for 48h at 110°C) or "wet" (added 2wt% H2O). Experimental pressure was 400 MPa, following the temperature-time path shown below, followed by isobaric quenching at 100°C/s. After initial crystallization the dendritic Cpx microstructure evolves through rearrangement of material at approximately constant crystallinity *(Pontesilli et al. 2019)*.

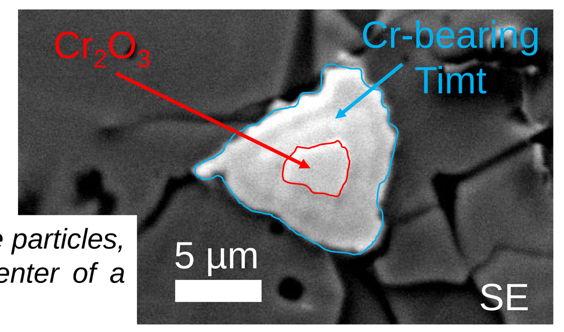


Sample microstructure

All experiments crystallize dendritic Cpx and isometric euhedral to hopper-shaped Timt. Greater water content and dwell time (sample ETK1b, center-right column) leads to more euhedral Cpx, obscuring original dendrites. Infrequent Cr-oxide crystals (solid impurities?) are surrounded by polycrystalline Cr-bearing Timt rims. Cpx dendrite "rosettes" radiate from the polycrystalline rims, but many dendrites do not belong to rosettes, at least in 2D. Individual Timt crystals (Cr-free) are strongly associated with the sides and tips of Cpx dendrites. ~ 75% of Timt grains are in contact with Cpx in 2D.

Cpx-Timt interfaces are often irregular and Timt is often attached only by thin necks. Timts are weakly clustered (R = 0.87 – 0.95, 1 = random).

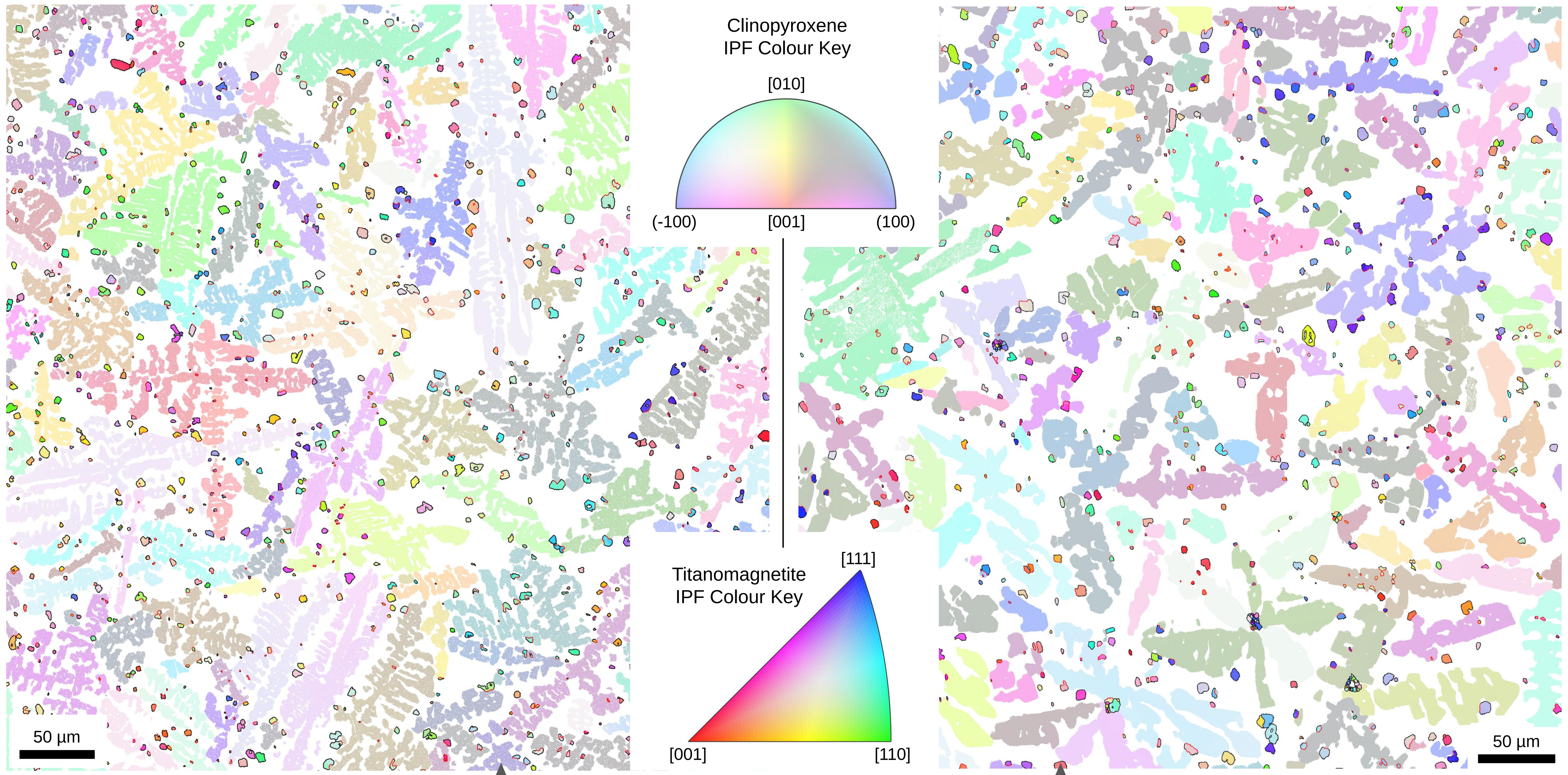




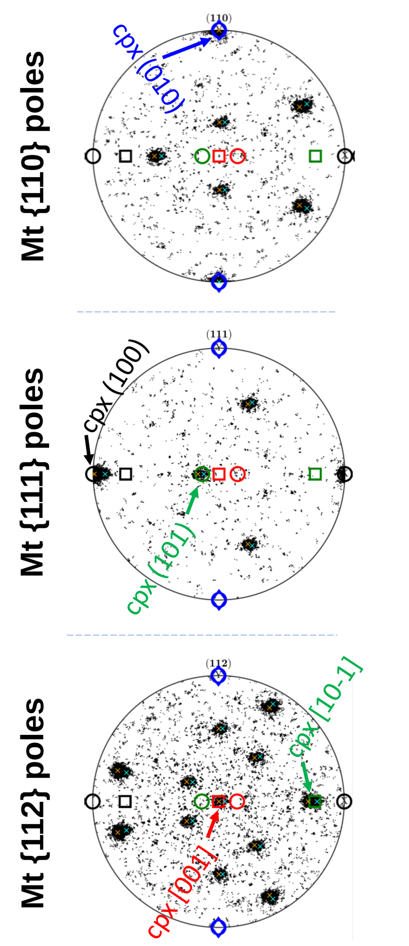
Left: *Several Cpx "rosettes" around Cr-oxide particles, sample ETK1b.* ***Right:*** *SE image of the center of a single rosette with compositions labelled.*

ETK2a	ΔT (initial)	:	**120°C**
	Added H_2O	:	**0 wt%**
	Dwell time	:	**4 hours**

ETK1b	ΔT (initial)	:	**80°C**
	Added H_2O	:	**2 wt%**
	Dwell time	:	**8 hours**

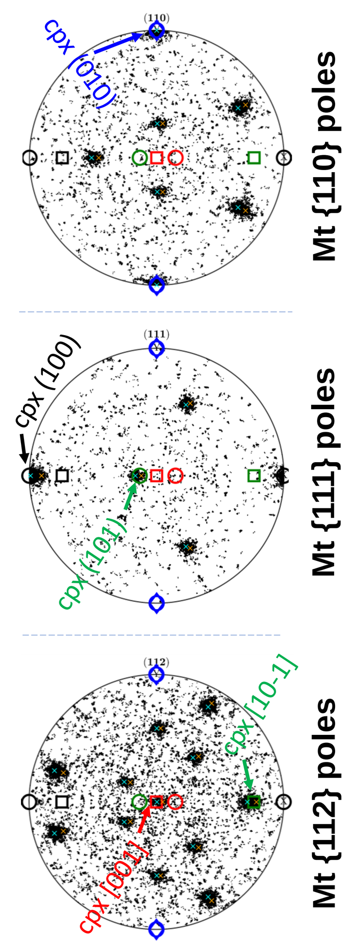


EBSD maps of Cpx and Timt in 2 samples, IPF color coding || *z*
*White fields = glass, Timt grain boundaries = **black lines***
*Boundary segments with Timt-Cpx COR = **red lines***



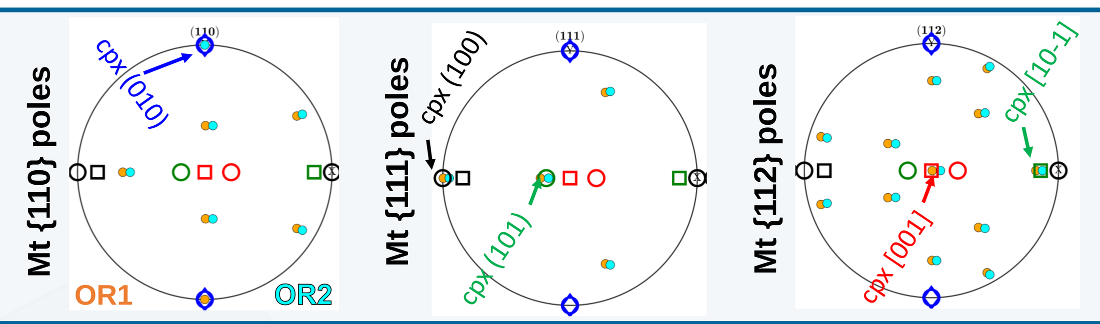
Timt orientation normalized relative to Cpx for all Cpx-Timt boundary segments in the above map (ETK2a).

Timt orientation normalized relative to Cpx for all Cpx-Timt boundary segments in the above map (ETK1b)



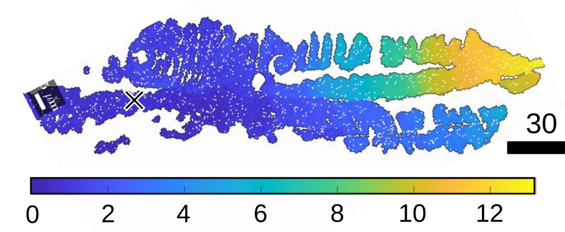
EBSD mapping reveals that the majority of Timt grains follow a clear crystallographic orientation relationship (COR) with touching Cpx crystals in both samples. This can already be seen in the uniform IPF color of all Timt adjacent to a single Cpx grain (EBSD maps above, 1 map per sample). ETK2a is "dry" with 4hrs dwell time, ETK1b "wet" with 8hrs dwell time. The orientation of Timt relative to Cpx can be plotted for every Timt-Cpx boundary segment in both maps (equal angle pole figures, left = ETK2a, right = ETK1b). Clearly for most segments the relative orientation of Timt and Cpx is very similar. In detail, Cpx-Timt boundary misorientations cluster around two separate CORs, described in the blue box below and indicated by cyan and orange crosses in the pole figures. However, the distribution of misorientations is continuous with no gap between OR1 and OR2. 75 – 89% of all Timt in contact with Cpx are misoriented <6° from the mean of OR1 and OR2. All boundary segments fulfilling this criterion are highlighted in red in the maps above. Within this larger category, most boundary segments can be assigned to either OR1 or OR2 within a tolerance angle of 2.6° (chosen as it is half the misorientation between the two CORs).

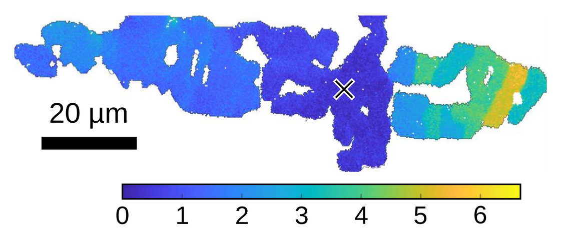
OR1:	**OR2:**
cpx [010] || tmt {110}	cpx [010] || tmt {110}
cpx (100) || tmt {111}	cpx (101) || tmt {111}
cpx [001] || tmt {112}	cpx [10-1] || tmt {112}



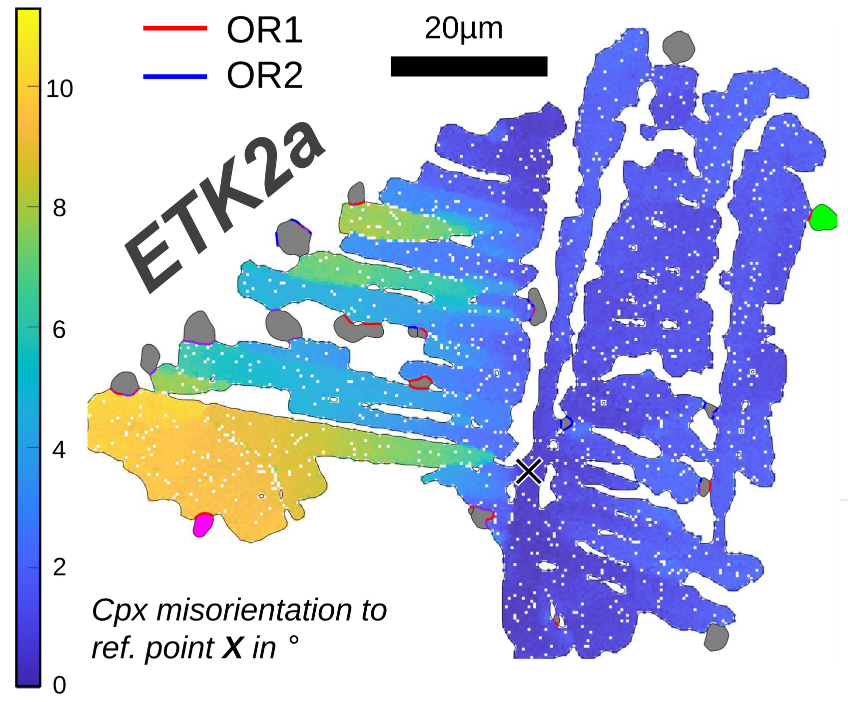
68 – 77% of all Timt in contact with Cpx are within 2.6° of one of these two CORs. OR1 and OR2 are misoriented by ca. 5.3°

Dendrites in the dry, 4hr annealed sample show continuous bending up to 15° and no subgrain boundaries.



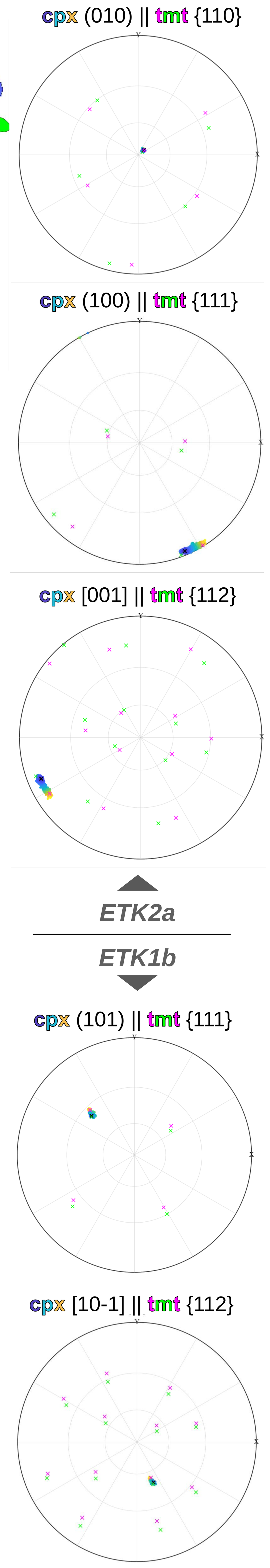


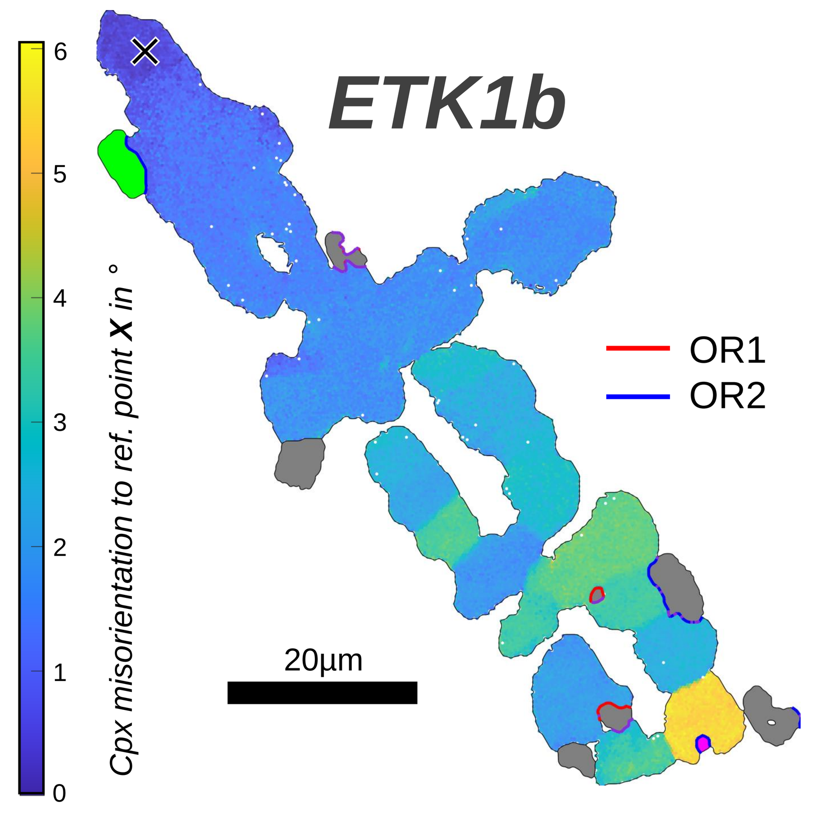
Dendrites in the wet, 8hr annealed sample show bending up to 7° and clear subgrain boundary development



The maps above and below show Cpx grains color-coded by misorientation angle to a reference point (black **x**). In the corresponding upper hemisphere equal angle pole figures (right) the orientation of several Cpx crystallographic directions is indicated for each map, with the same color-coding. Both Cpx crystals rotate around their [010] direction, up to 11° (ETK2a) and 6° (ETK1b). For each Cpx two associated Timt are highlighted, in contact with the areas of **lowest** and **highest** misorientation of Cpx. Both selected Timt show a perfect COR to the adjacent Cpx, despite bending of the dendrites > 5° !

Also shown are all other Timt grains touching the selected Cpx (**grey fields**) and all Cpx - Timt boundary segments within 2.6° misorientation of OR1 (**red lines**) and OR 2 (**blue lines**). Boundary segments outside OR1 and OR2 but still within 6° misorientation of the mean of OR1 and OR 2 are colored **purple**.





Conclusions: Bent Cpx dendrites nucleated on early-formed or unmelted Cr-oxides. Dendrite bending may potentially recover with longer annealing times. Cr-free Timt forms clear CORs with locally adjacent Cpx, despite >10° of total dendrite bending. Thus, most Timt must have nucleated on (or attached to) Cpx simultaneous with or after dendrite growth.