Study on temperature dependent EBSD measurement

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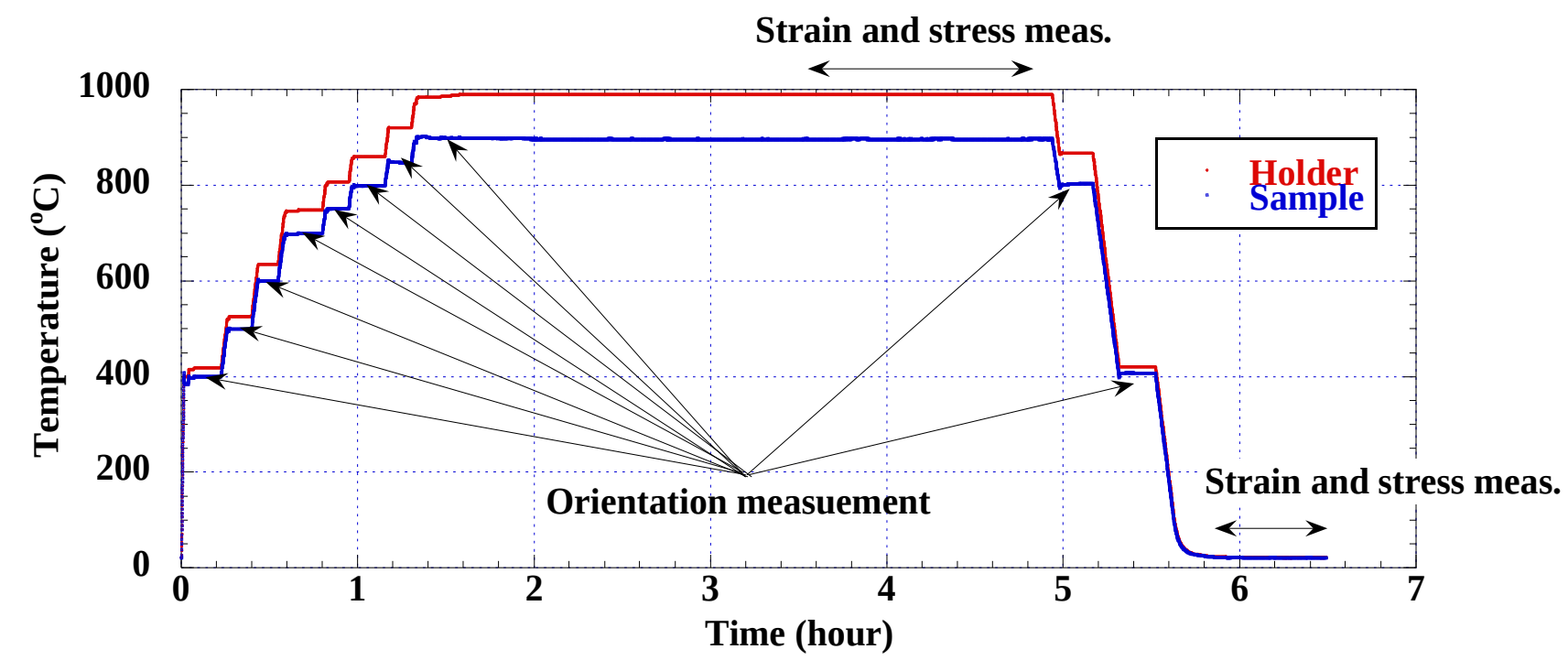
EBSD at NIMS (National Institute for Material Science)

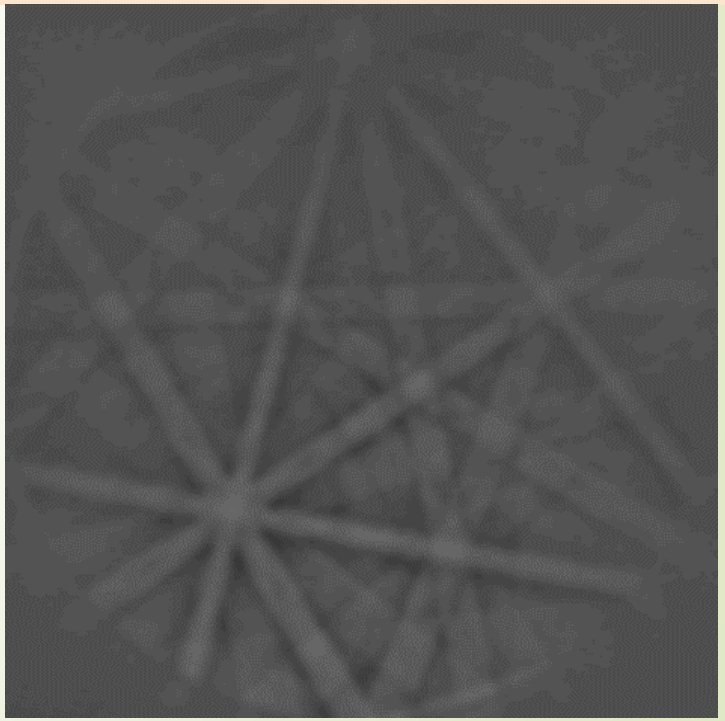
- EBSD (Electron Beam Scatter Diffraction) can be measured crystallographic characterization from micro-area (<50nm).
- Information such as grain orientation, stress, etc. of the sample can be obtained by scanning the electron beam.
- EBSD was measured by SEM specialist in NIMS.

	SPEC
Type	Schottky Field Emission Scanning Electron Microscope
Model Number	JEOL JSM-7001F
Resolution	1.2 nm (30kV), 3.0 nm (1kV)
Accelerating Voltage	0.5 kV ~30kV
Magnification	x10~ 1,000,000

Temperature dependent EBSD measurement

- We measured the recrystallization of Tokyo-Denkai Fine grain sample.
- Cristal orientation was measured at each temperature step during heating up and cool down.
  - It takes about 6 minutes for each.
- Strain and stress were measured at 900 °C and room temperature after cooling.
  - It takes about 1.5 hours because all Kikuchi patterns are saved.



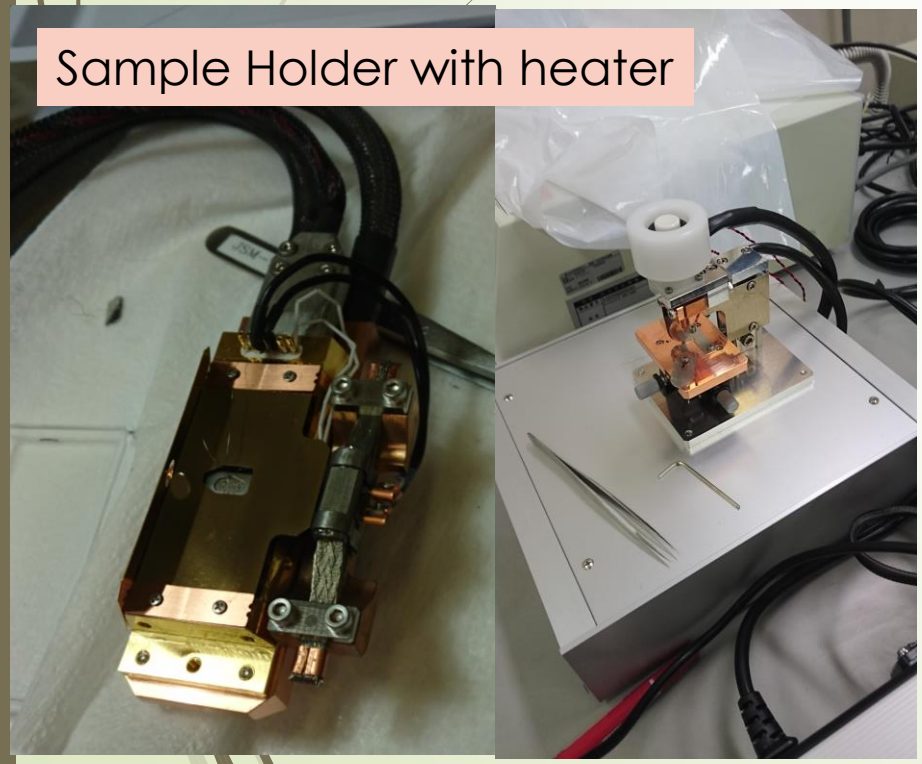


Nb Kikuchi Pattern

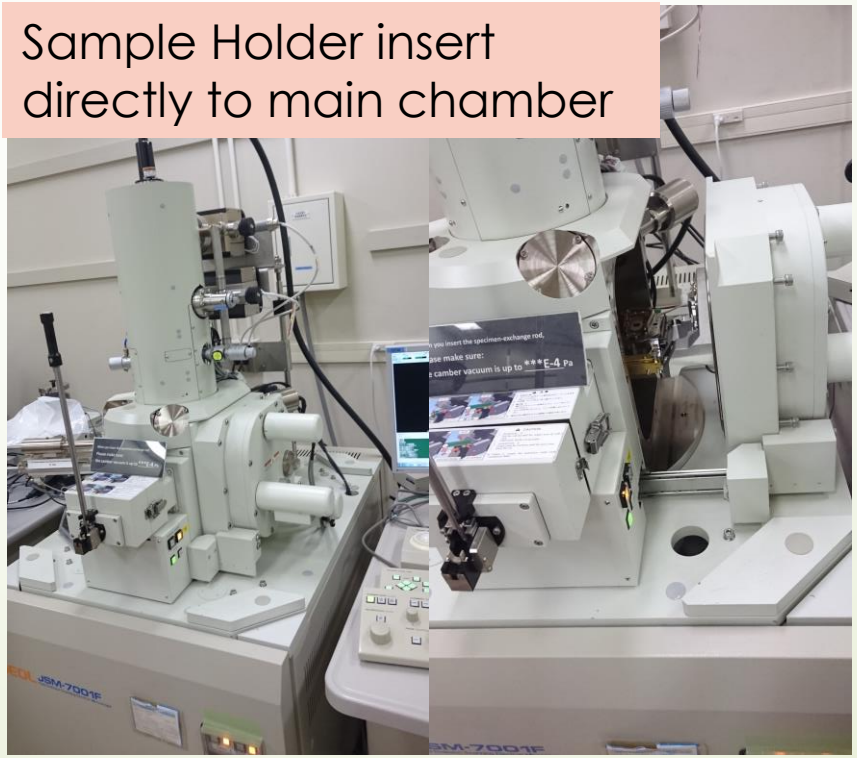
Sample Setting

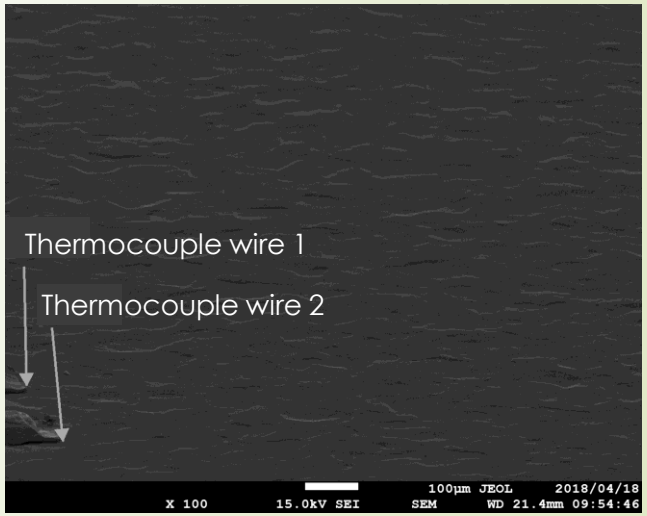
- Sample temperature can be controlled by using the special sample holder with heater.
  - Sample size should be 5mm x 7mm x 0.5mm. Maximum temperature is 1000 °C .
- K type Thermocouple is directly welded by small spot welder.
  - Temperature controlled by holder temperature.
  - The temperature difference between sample and holder is about 100 °C at 1000 °C
- Sample analysis position can move following the position fluctuation during heating by watching the thermocouple wire.

Sample Holder with heater

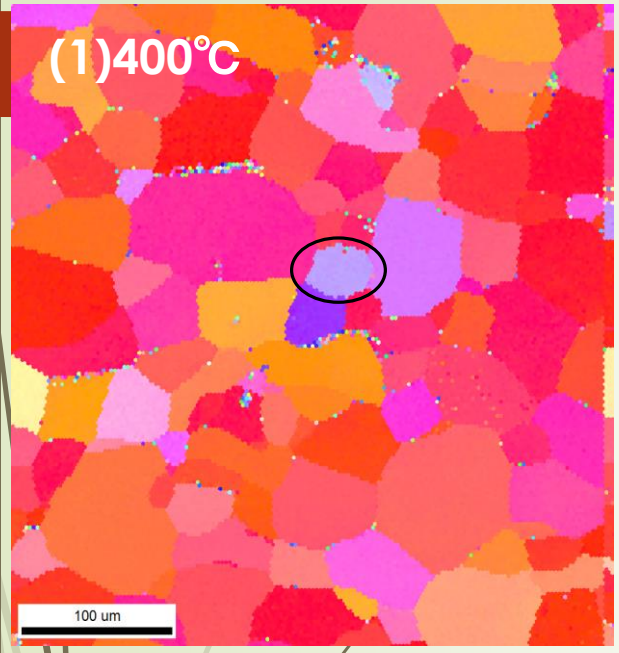


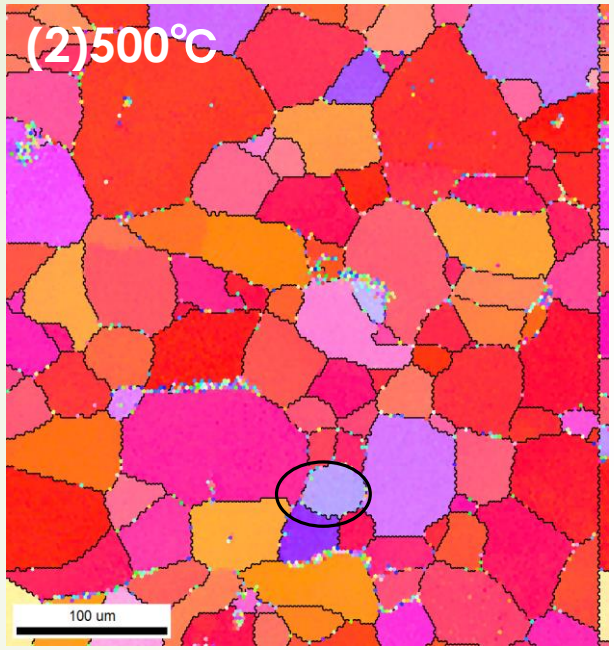
Sample Holder insert directly to main chamber

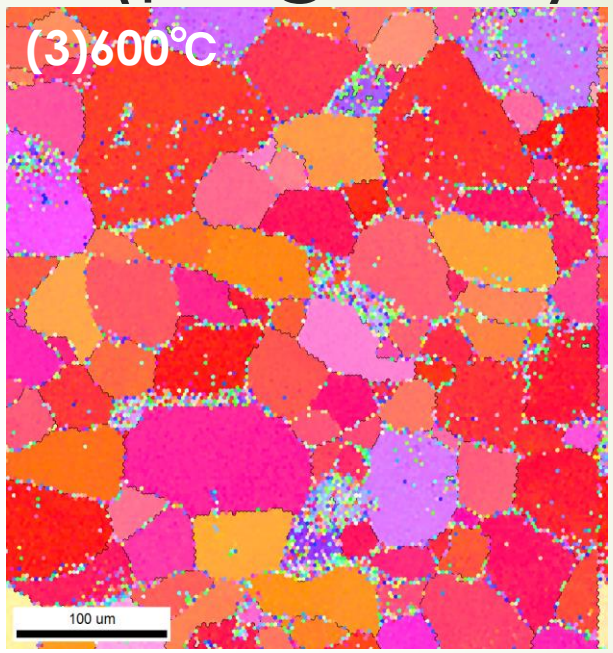


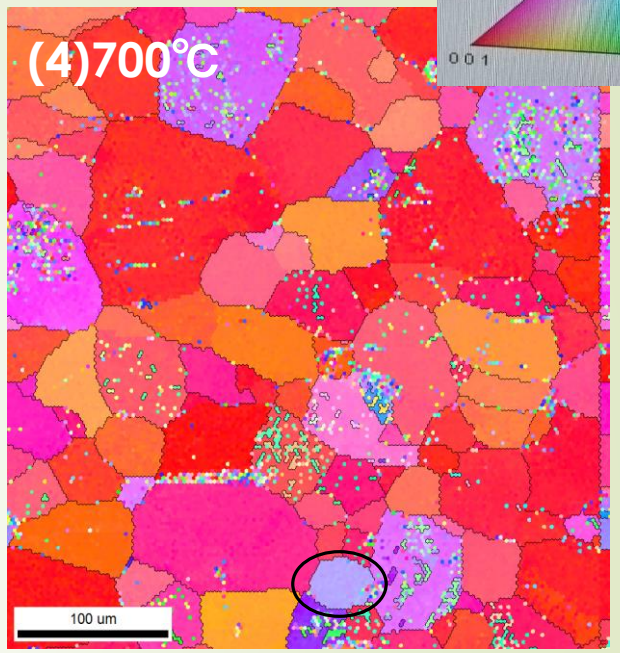


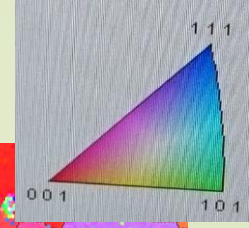
Grain Orientation (page1/3)

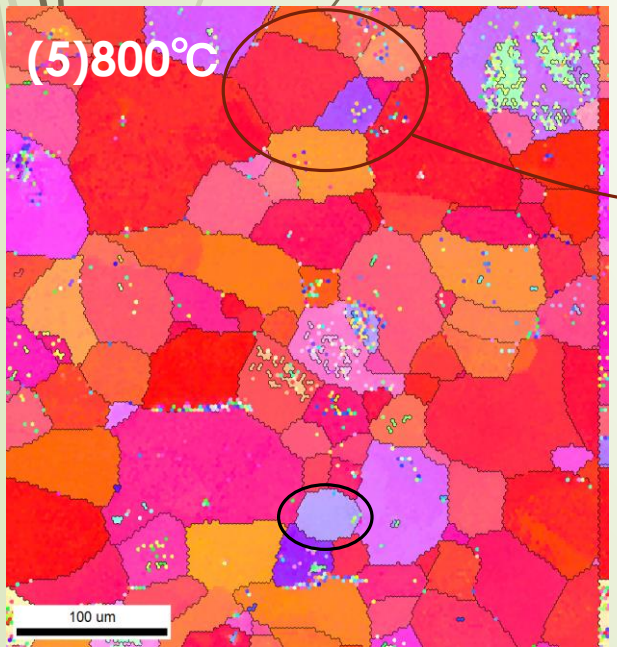


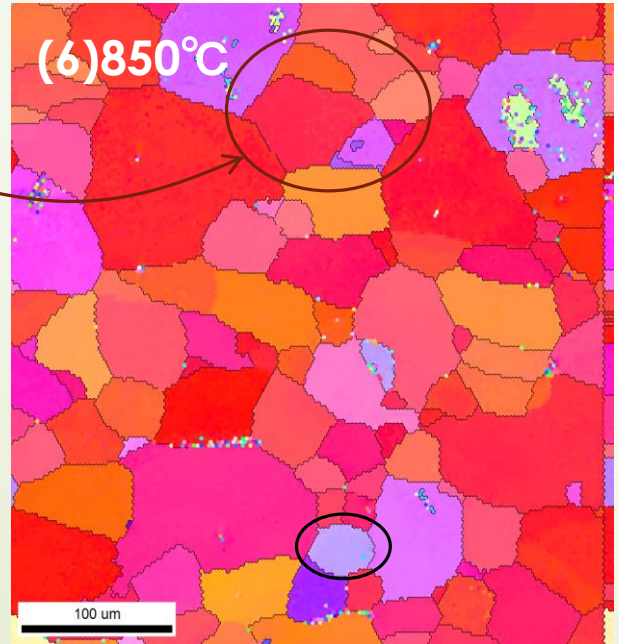


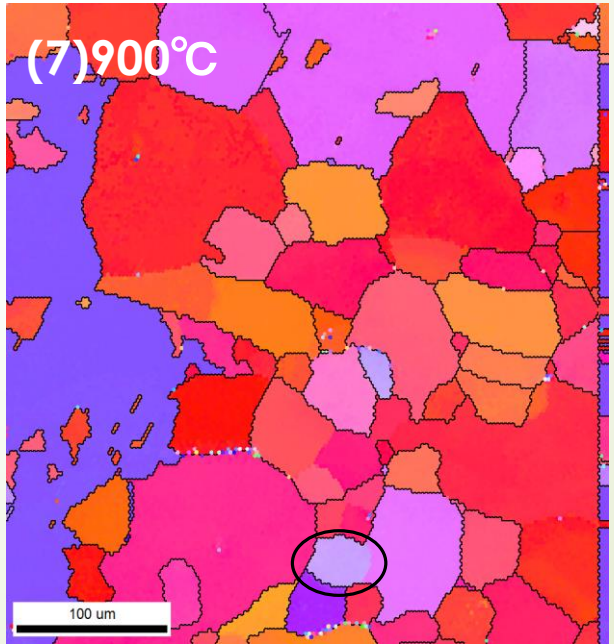


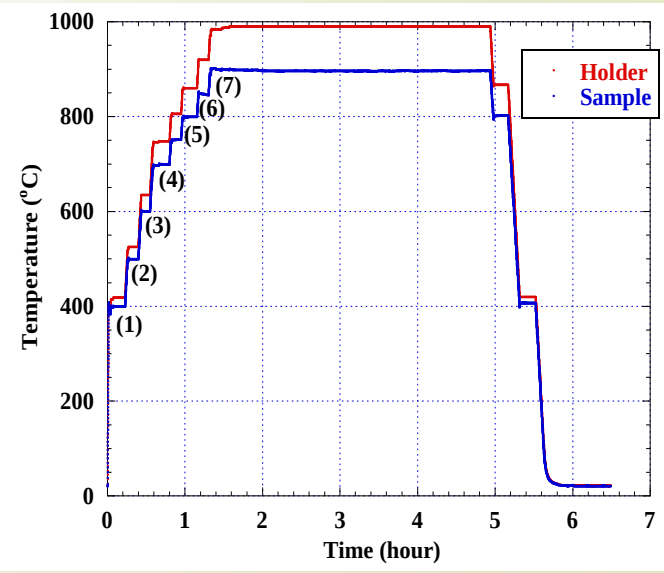






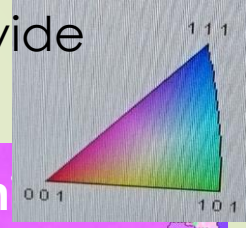


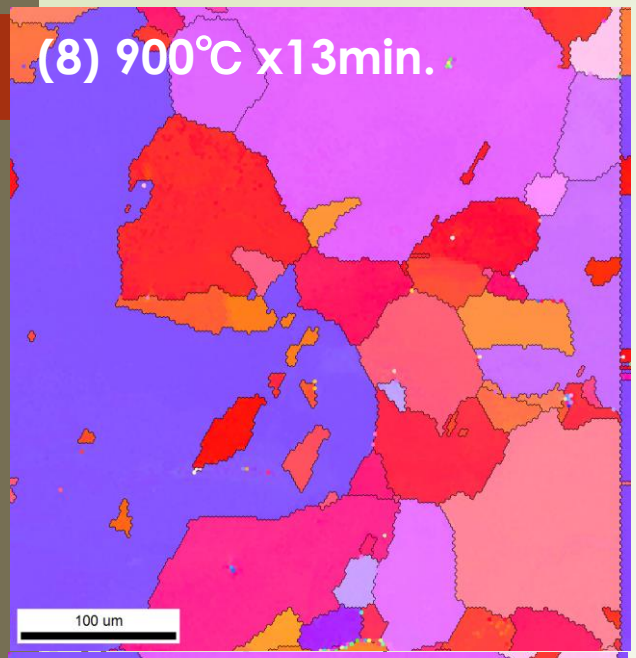


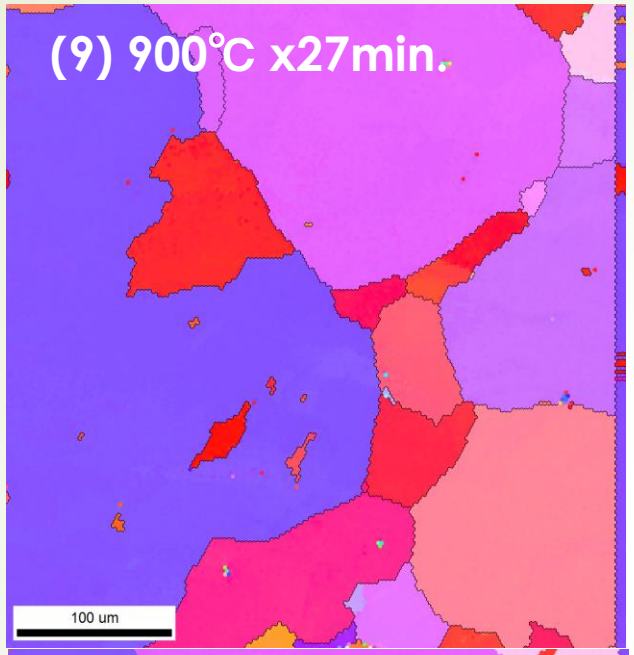


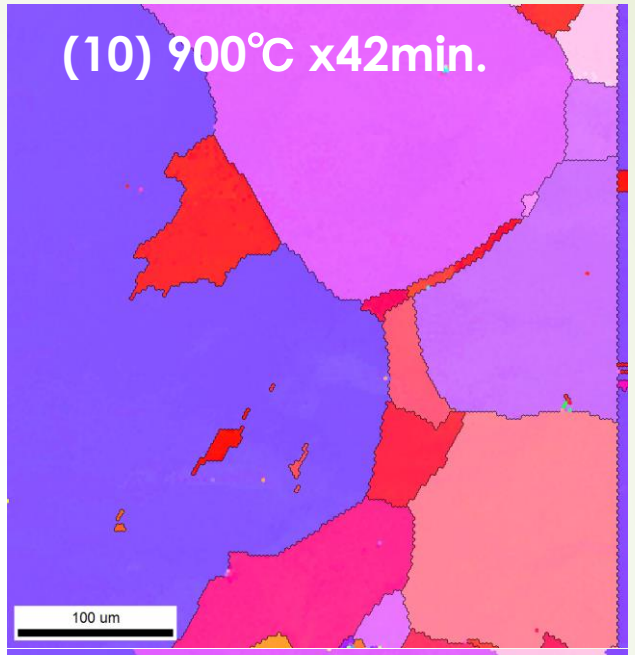
Grain Orientation (page2/3)

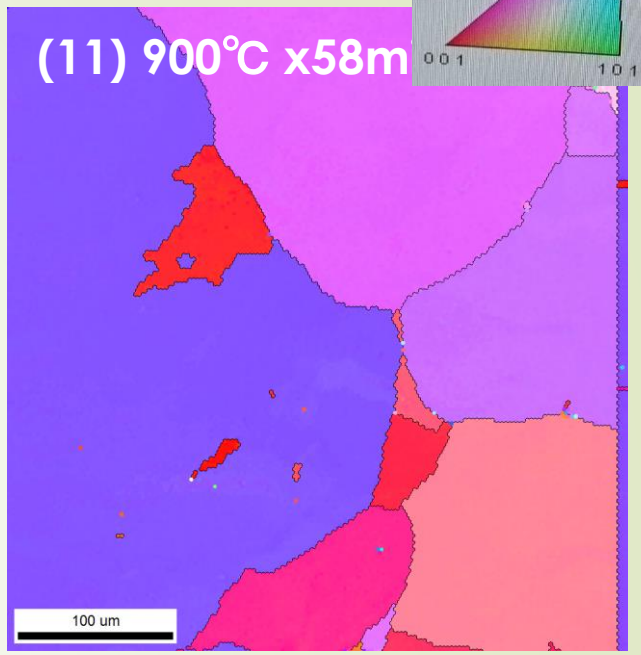
Solid black line divide
5° difference

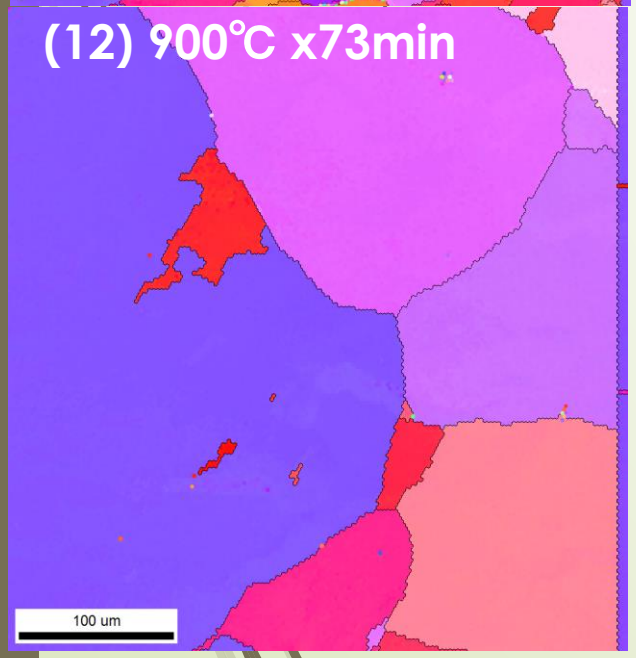


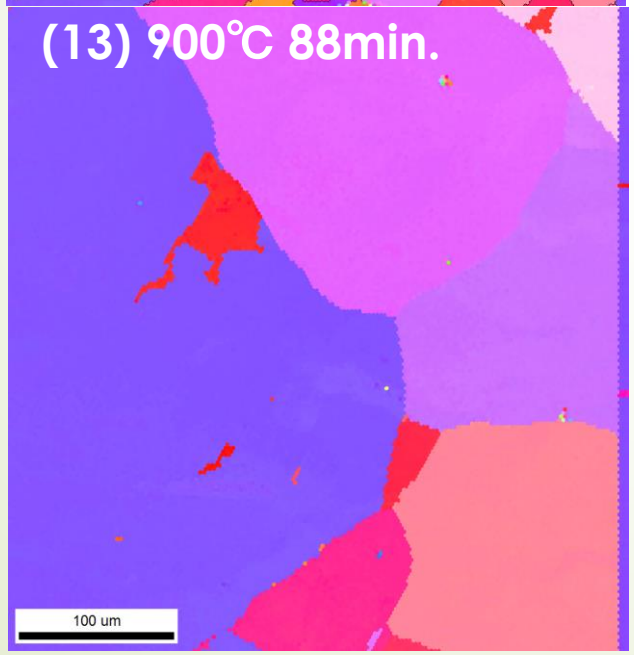


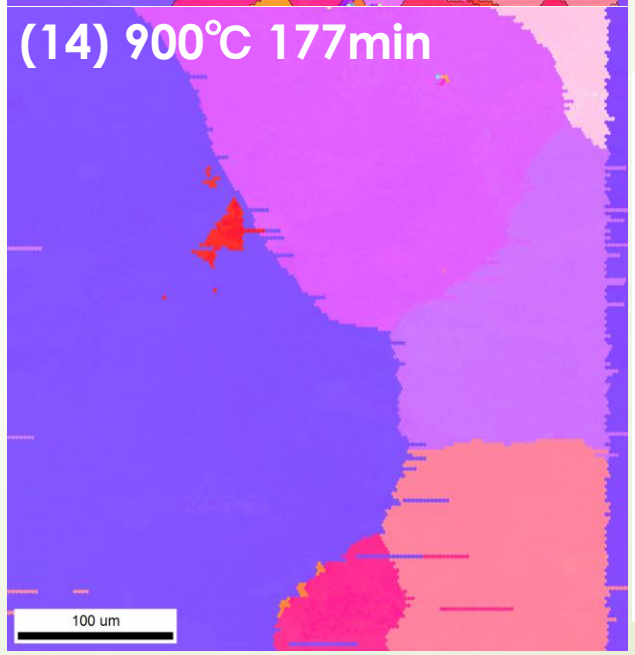


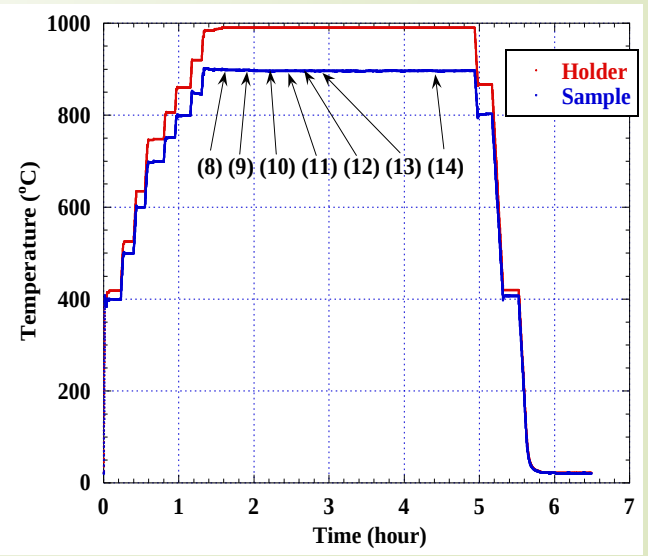






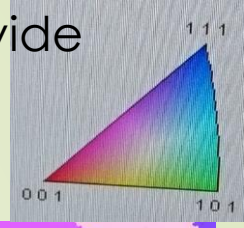


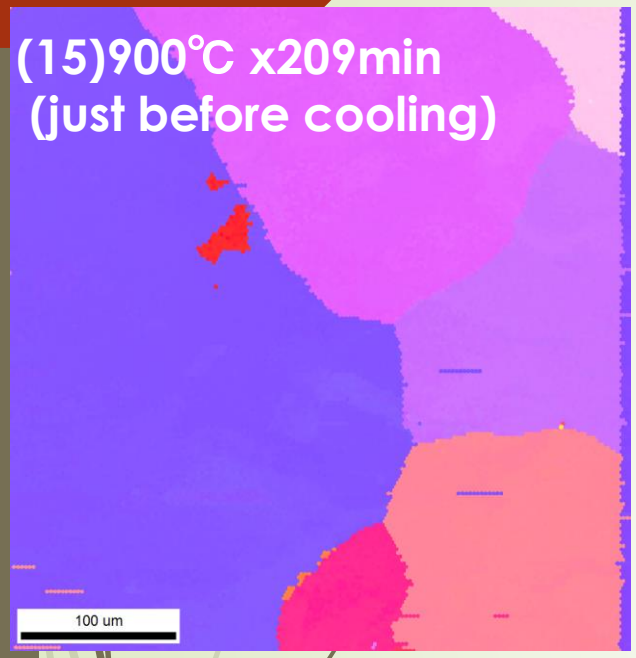


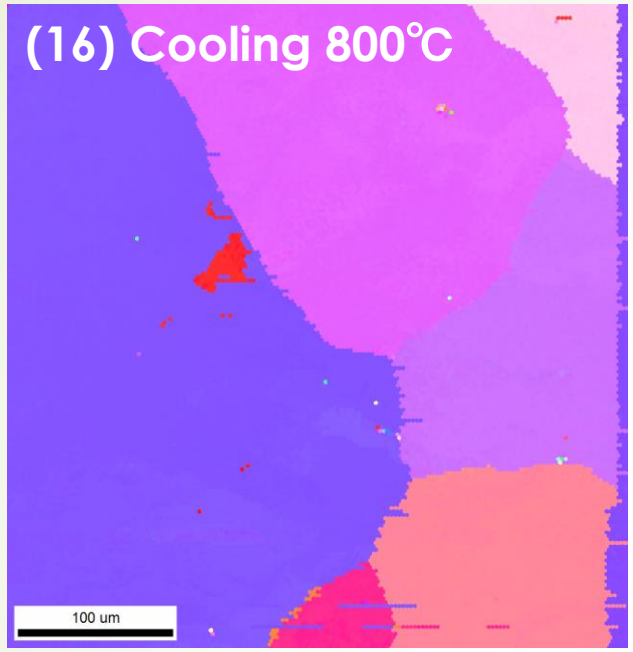


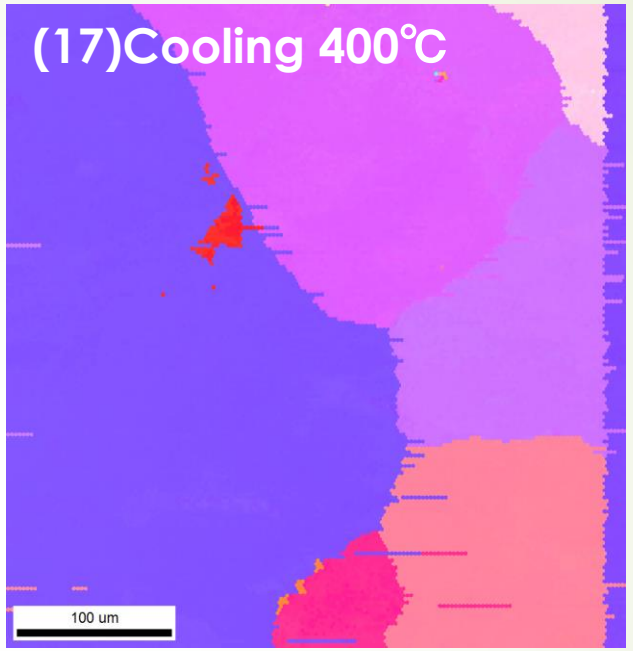
Grain Orientation (page3/3)

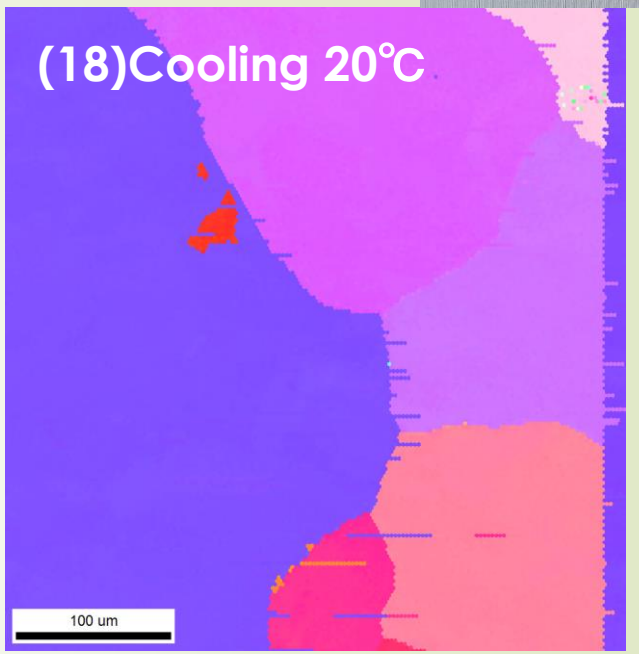
Solid black line divide
2° difference



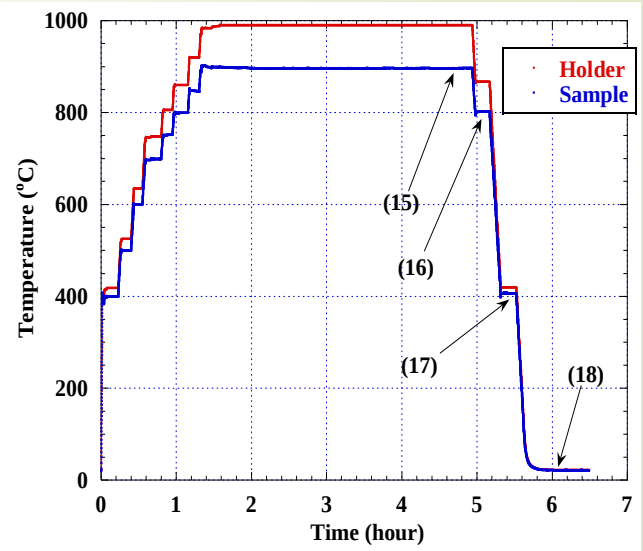






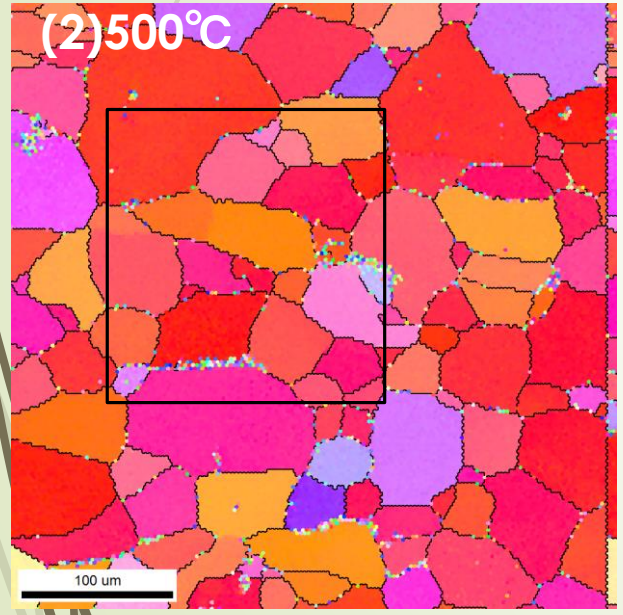


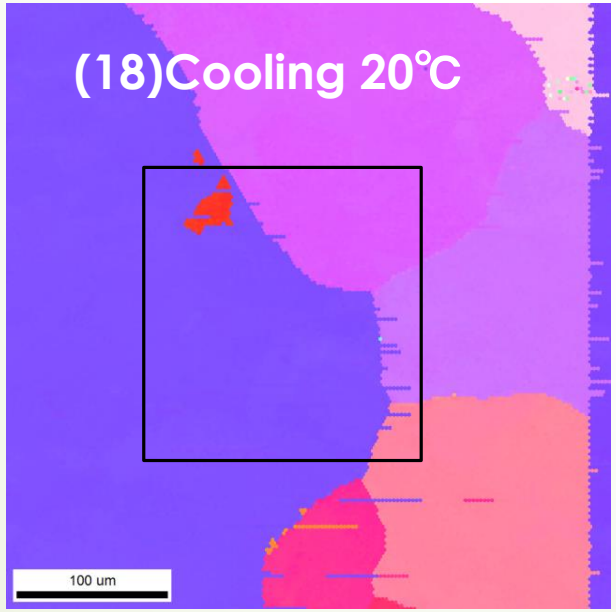
- Recrystallization stars from at least 850 °C.
- Grain size rapidly expanded from 900 °C.
- Grain size is not change during cooling.
- 3 hours are enough for recrystallization at 900 °C.
  - Grain size expanded more than 100 um x100 um.

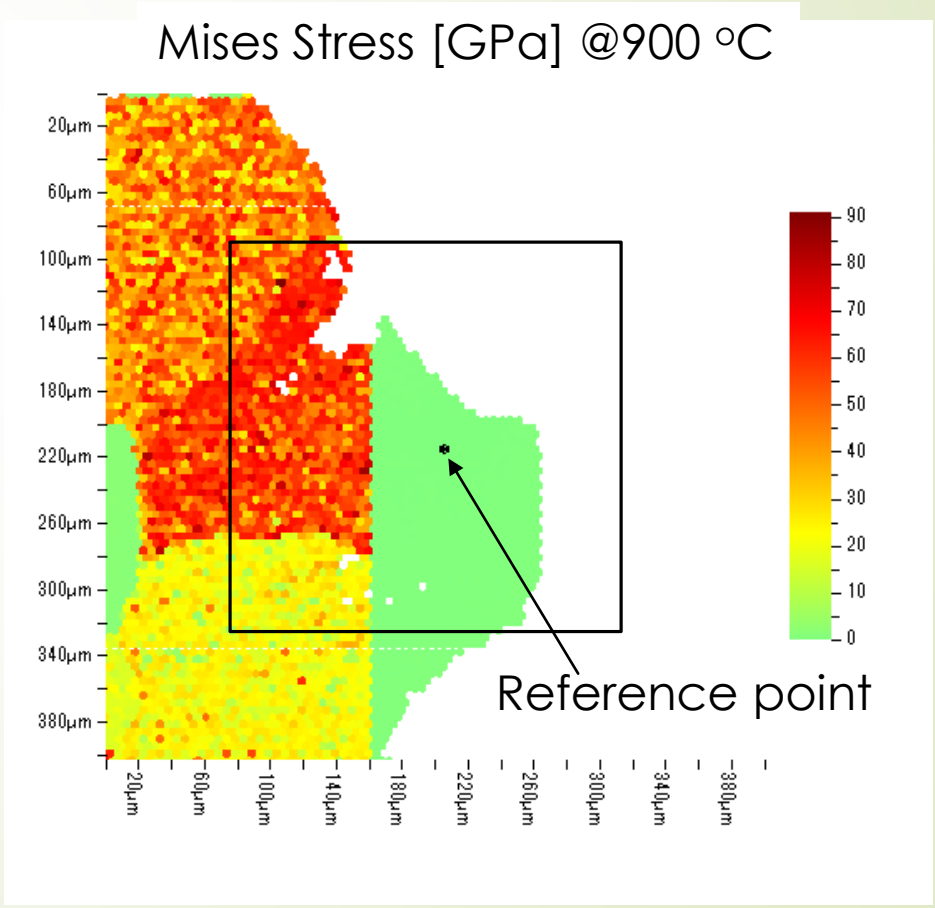


Stress measurement

- The stress seems not depend on the original gain orientation pattern.
- In next chance we will check the mises stress after cooling.
  - This image scan takes more than 1.5 hours.
  - We didn't have enough time to measure after cooling .

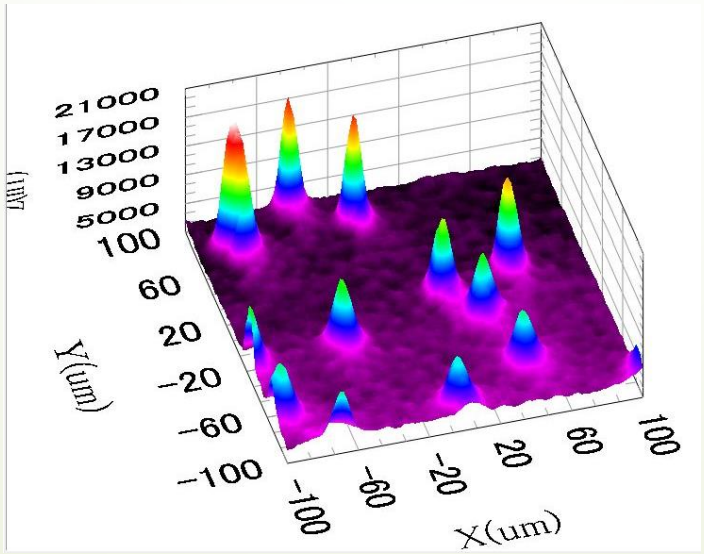






Summary and future

- We carried out temperature dependent EBSD measurement. It is powerful tool to understand how to grow grains.
- Tokyo-Denkai FG Nb sample shows drastic change at 900℃.
- We try to relate information from EBSD measurement, i.e. grain boundary, stress, dislocation etc., to pinning center.
- Trapped magnetic flux can be measured by scanning SQUID at NIMS.



Scanning SQUID (left) at NIMS and observed quantum magnetic flux on EP Nb sample (right)