Modeling Microstructural Development During Hot Working of Carpenter Technology Ni-Based Alloy 680

PURDUE UNIVERSITY
School of Materials Engineering

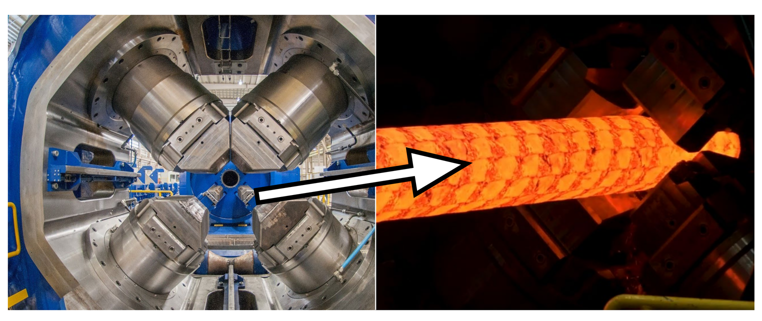
Student Names: Will Heffern, Spencer Hagaman, Matias Filloy
Faculty Advisors: Prof. Michael Titus, Prof. Matthew Krane
Industrial Sponsors: Stephane Forsik, Mario Epler, Austin Dicus, Jose Gonzalez-Mendez

Carpenter® Technology is seeking to develop models that describe the microstructural behavior of the alloy 680 (UNS #N06002) Ni-based superalloy during hot-working. Hot compression tests conducted on a Gleeble® 3800 under varied thermomechanical processing conditions were utilized to generate JMAK recrystallization curves and grain size models. Optical microscopy and EBSD were carried out to characterize the microstructural progression of recrystallization during forging. High fractions of dynamic recrystallization were observed at temperatures of 1093°C and above when the compressive strain was at or above 0.8. Hot working below 982°C produces minimal recrystallization and is not recommended. Grain sizes of 10 – 15 µm were found in dynamically recrystallized samples and the target grain size of ASTM 6 was produced at a strain of 1.2, a temperature of 1093°C, and holding time of 60 s.

This work is sponsored by Carpenter® Technology Corporation, Reading, PA.

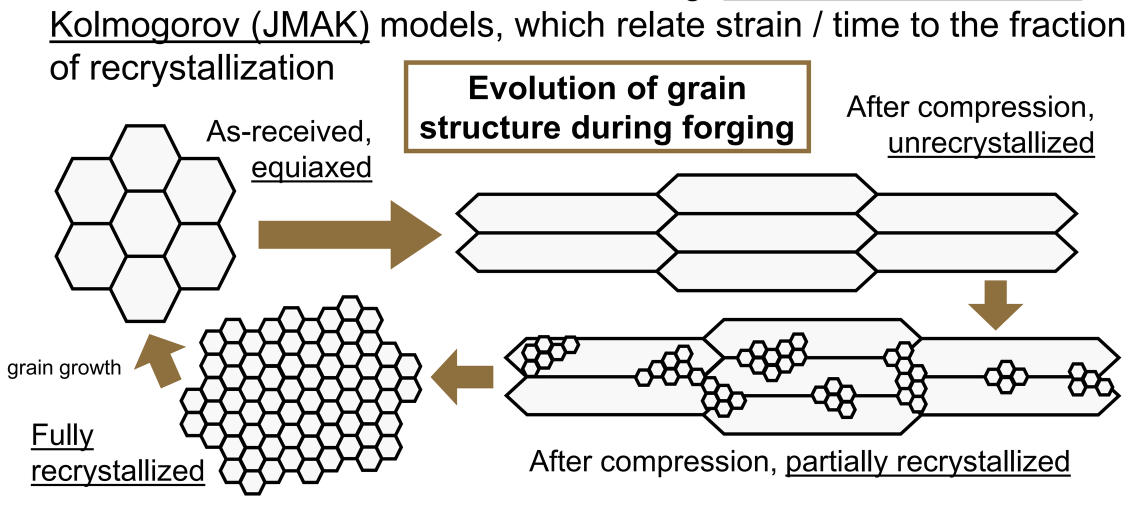
CARPENTER® TECHNOLOGY

Project Background



Carpenter Technology SMX-1100 radial press used for hot-working of specialty alloys at the company's Athens, AL forge [1].

- Producing a high temperature, critical application material, such as an Ni-based superalloy like **alloy 680 (UNS #N06002)**, requires multiple stages to convert material from as-cast ingot to usable billet
- Recrystallization of the material is a key parameter for meeting both product quality and mechanical properties requirements
- Hot deformation microstructural modeling: Johnson-Mehl-Avrami-Kolmogorov (JMAK) models, which relate strain / time to the fraction of recrystallization



Carbon	0.05 to 0.15 %
Phosphorous (Maximum)	0.04%
Silicon (Maximum)	1.00%
Nickel	Balance
Cobalt	0.50 to 2.50 %
Iron	17.00 to 20.00 %
Manganese (Maximum)	1.00%
Sulfur (Maximum)	0.03%
Chromium	20.50 to 23.00 %
Molybdenum	8.00 to 10.00 %
Tungsten	0.20 to 1.00 %

Alloy 680 is a nickel-based, solid-solution strengthened γ alloy used in applications such as turbine rotors & shafts, afterburner components, furnace hardware, as well as in the chemical processing industry [2].

Alloy 680 composition (wt %) [2]

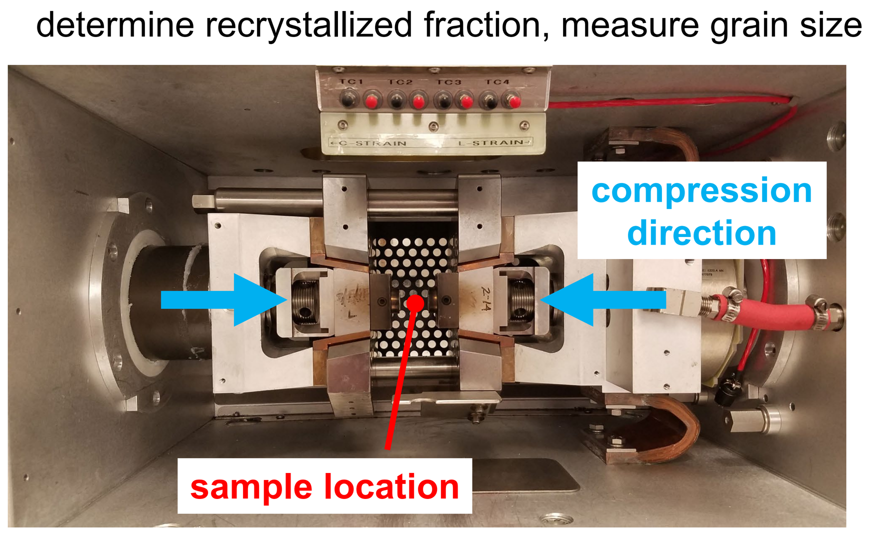
Experimental Methods

Gleeble 3800 thermomechanical simulator:

- Replicated industrial hot-working conditions on a lab testing scale
- Conducted 114 hot compression tests on small cylindrical samples
- Conditions of thermomechanical processing tested: strain, strain rate, temperature, holding time, plus initial grain size

Characterization:

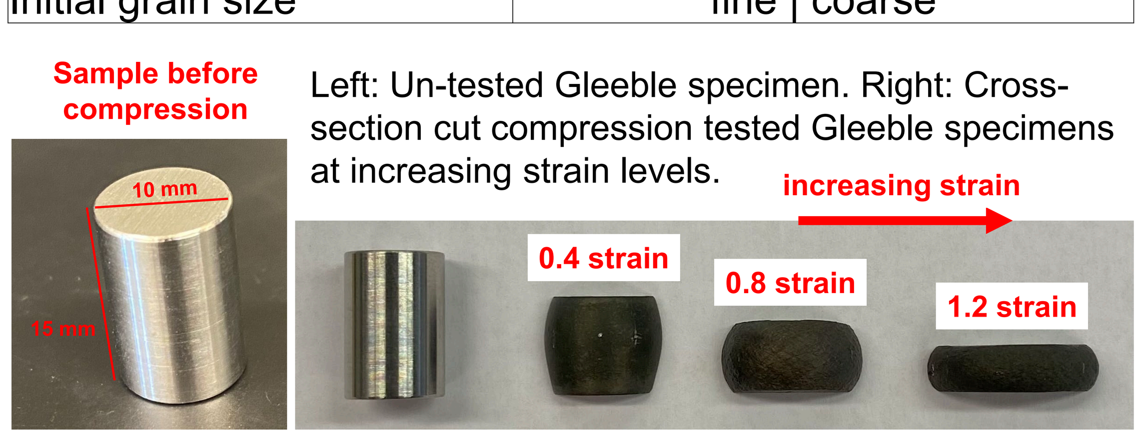
- Samples were cut, mounted and polished for optical microscopy
- Material etched using Waterless Kalling's etchant (ASTM E 407)
- Examined using optical microscopy and ASTM E 562 procedure to determine recrystallized fraction, measure grain size



Working action of the Gleeble 3800 thermo-mechanical simulator, as set up for hot compression testing [1].

Below: Array of sample conditions tested via the Gleeble 3800.

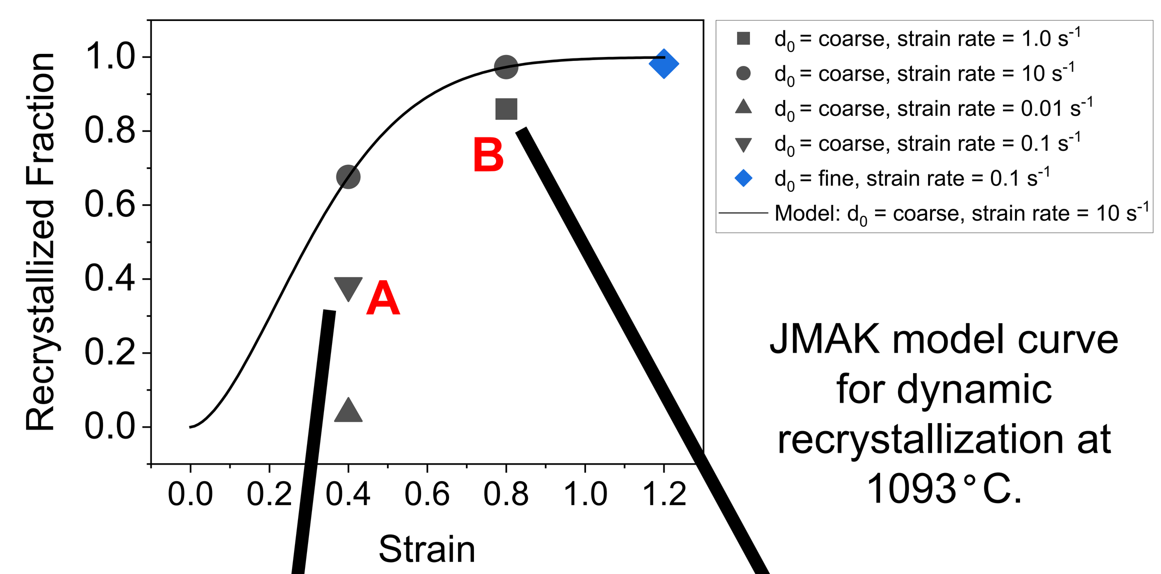
Strain	0.4 | 0.8 | 1.2
Strain rate (s^{-1})	0.01 | 0.1 | 1.0 | 10
Temperature (Celsius)	927 | 982 | 1038 | 1093 | 1149
Post-forging hold time (s)	0.1 | 10 | 20 | 60
Initial grain size	fine | coarse



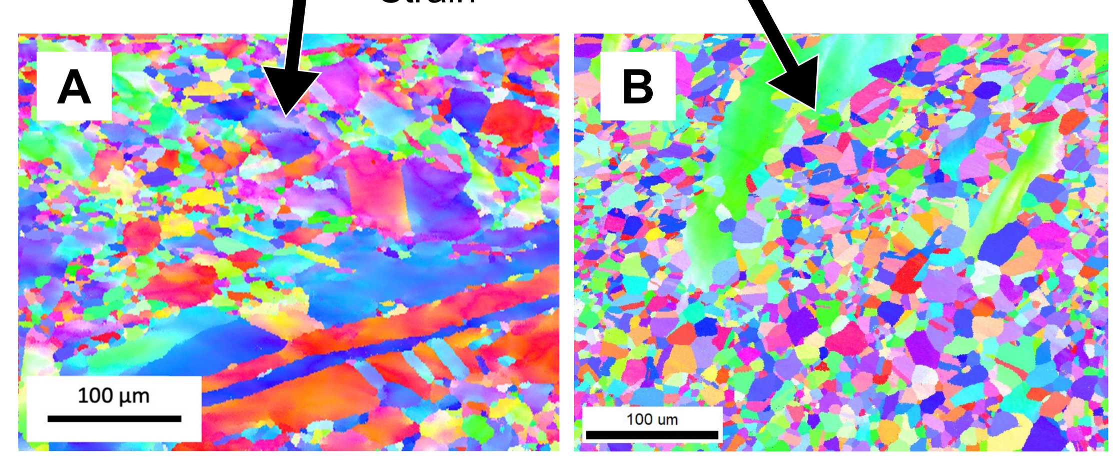
Left: Un-tested Gleeble specimen. Right: Cross-section cut compression tested Gleeble specimens at increasing strain levels.

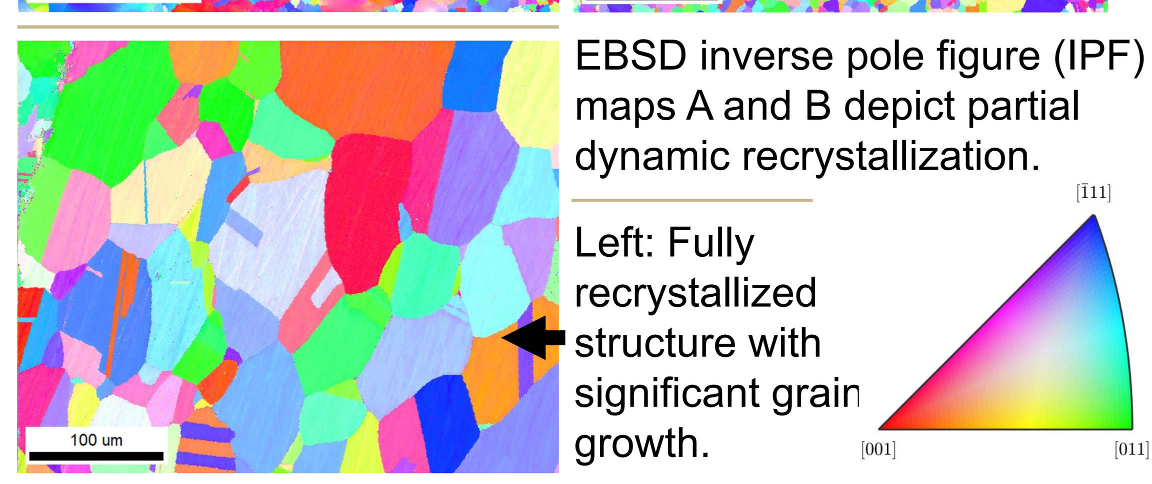
Further characterization of select partially recrystallized microstructures was carried out via electron backscatter diffraction (EBSD) on a Quanta™ 650 field emission scanning electron microscope (FESEM).

Recrystallization Results



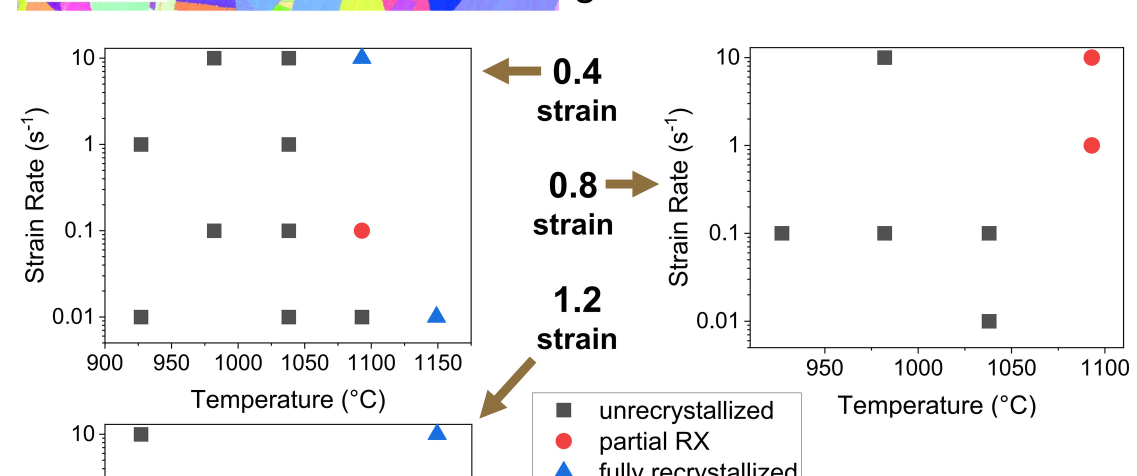
JMAK model curve for dynamic recrystallization at 1093°C.

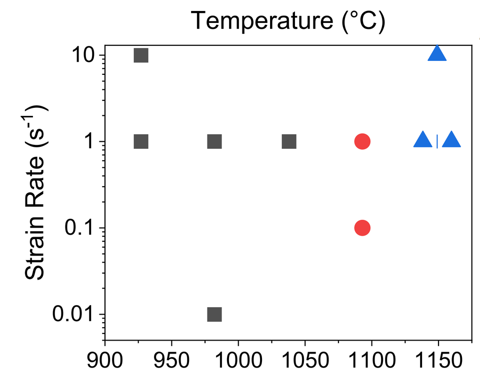




EBSD inverse pole figure (IPF) maps A and B depict partial dynamic recrystallization.

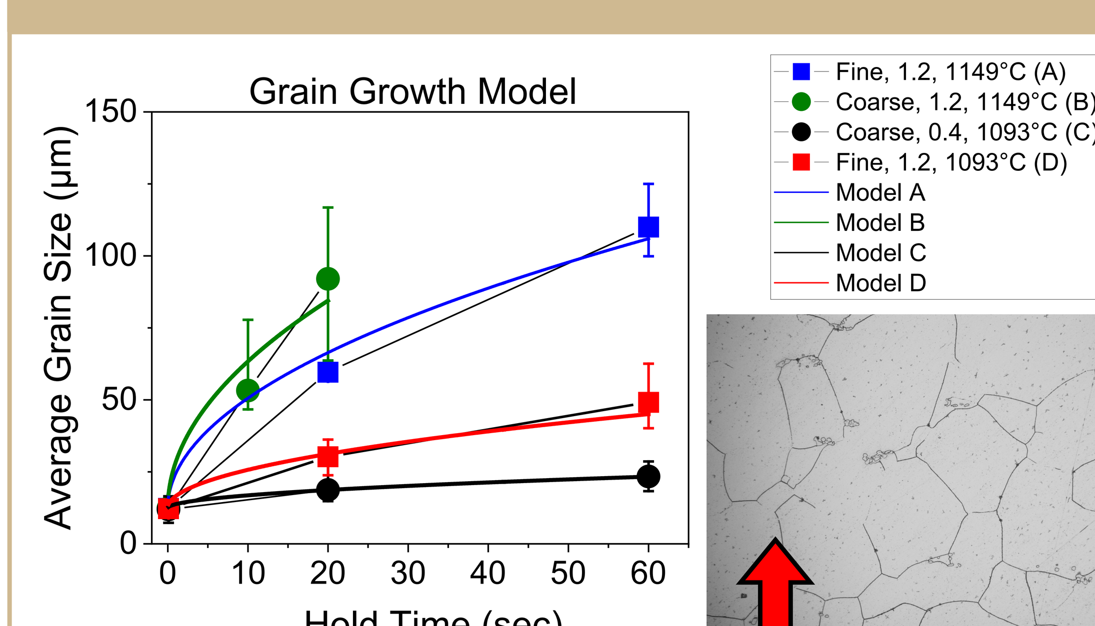
Left: Fully recrystallized structure with significant grain growth.

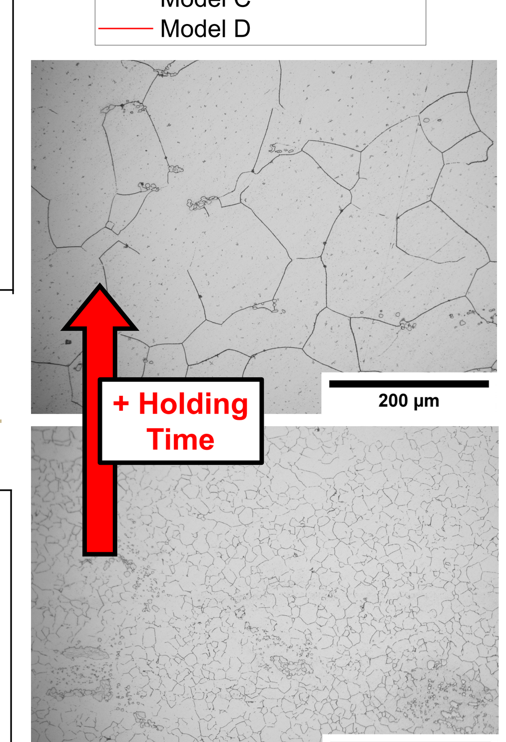


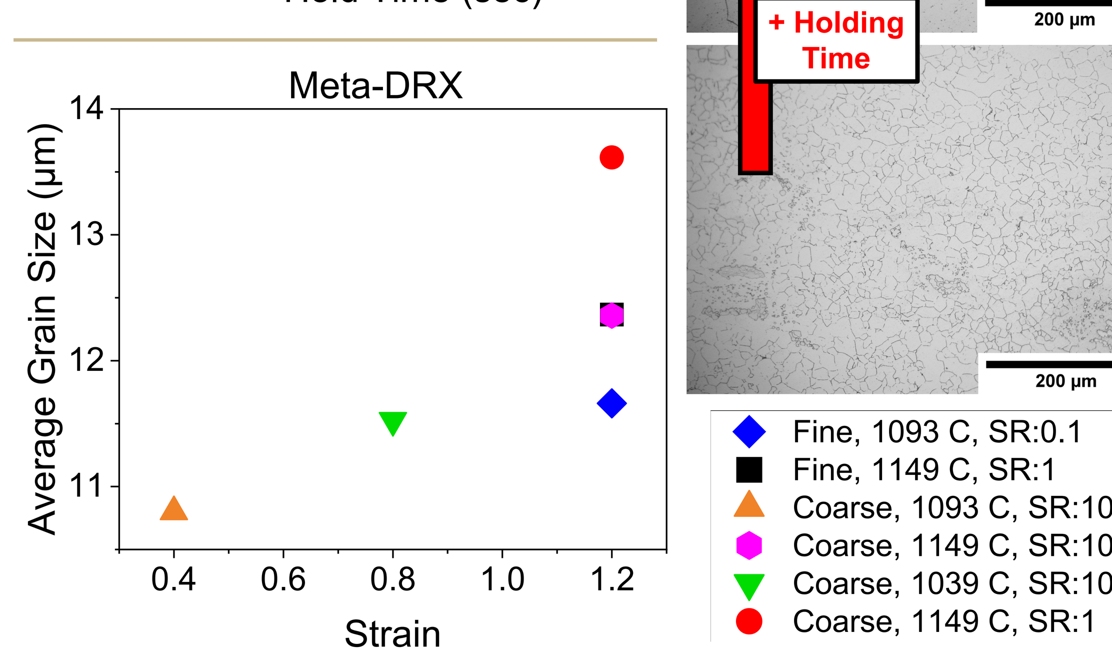


Recrystallization maps depicting the conditions producing dynamic recrystallization at varied strains. Dynamic recrystallization occurred above 1093°C.

Grain Size Results







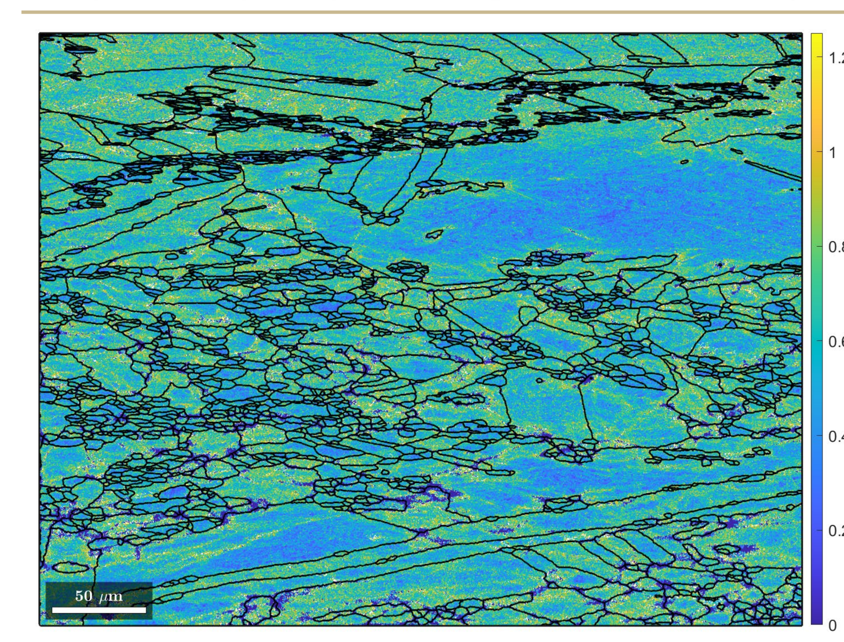
Discussion & Analysis

Recrystallization

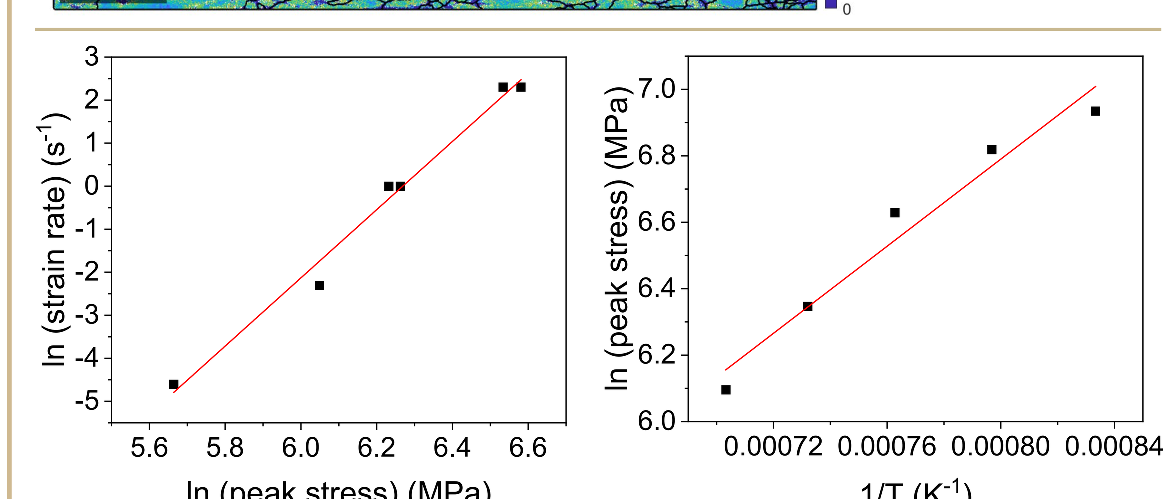
- Alloy 680 possesses a narrow recrystallization range
- Temperatures of 927°C and 982°C failed to produce considerable recrystallization
- Dynamic recrystallization occurs at 1093°C and 1149°C
- 0.8 and 1.2 strain compressions at a minimum temperature of 1093°C provide high recrystallized fractions with controlled grain growth (under tested short hold times)

Grain Growth

- Higher temperatures induce rapid grain growth
- Dynamically recrystallized grain size: 10 – 15 µm



EBSD-based kernel average misorientation (KAM) map of sample A on JMAK curve. Small recrystallized grains with low internal misorientation can be seen in dark blue.



Plots showing the relationships used for calculation of parameters m (left) and r (right) needed for calculation of activation energy, Q, based on flow stress curves for alloy 680.

- The activation energy Q for dynamic recrystallization in alloy 680 was calculated to be 431 kJ/mol, a value within the expected range of 300 – 450 kJ/mol for Ni-based superalloys [3].

Conclusion & Recommendations

- Hot working below 982°C does not produce reliable recrystallization
- Hot working between 1093°C - 1149°C consistently yields high fractions of dynamically recrystallized material (85% +)
- Dynamic recrystallization activation energy: 431 kJ/mol
- The initial grain size has a minimal impact after grain growth, with the controlling process parameters being strain and temperature
- Higher strain rates increase the recrystallized fraction when other conditions are equal
- A grain size of ASTM 6 is achievable via the following conditions: fine initial grain size, strain of 1.2, and either a temperature of 1149°C with a hold time of 20 seconds or a temperature of 1093°C with a hold time of 60 seconds for less variability

References

1. Images courtesy of Carpenter Technology
2. CarTech® 680 Alloy Technical Datasheet: https://www.carpentertechnology.com/
3. Gujrati, R., Gupta, C., Jha, J. S., Mishra, S., & Alankar, A. (2019). Understanding activation energy of dynamic recrystallization in Inconel 718. *Materials Science and Engineering: A*, *744*, 638–651. https://doi.org/10.1016/j.msea.2018.12.008