9Cr ODS Material Development

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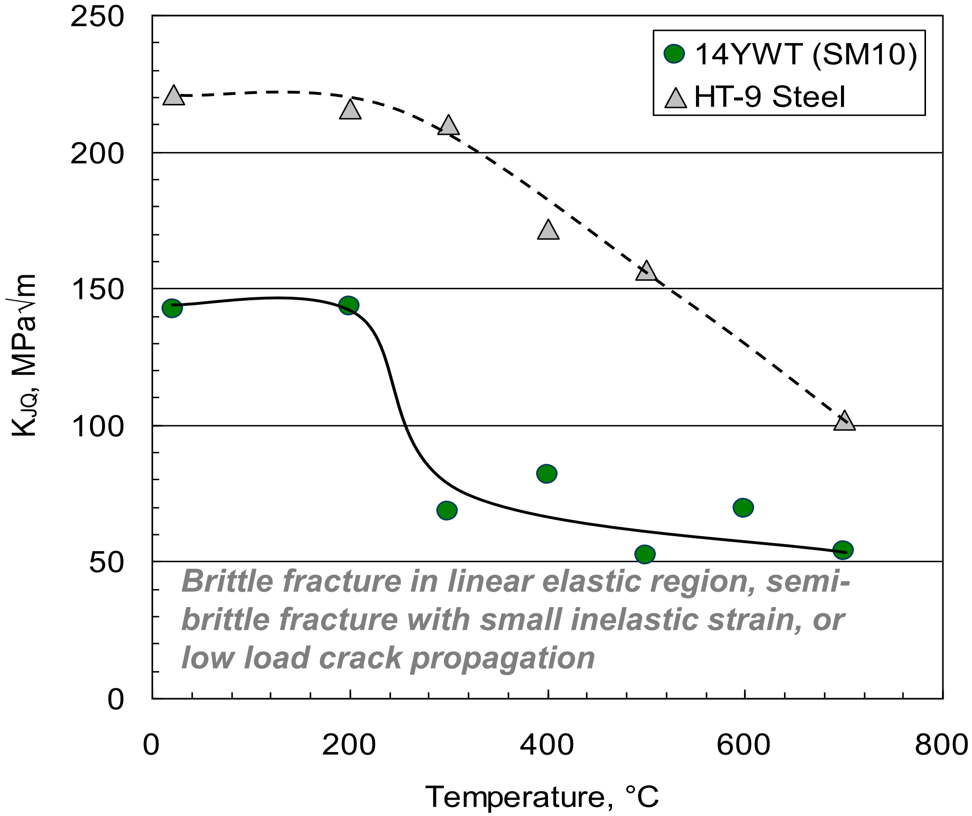
Acknowledgement & Contents

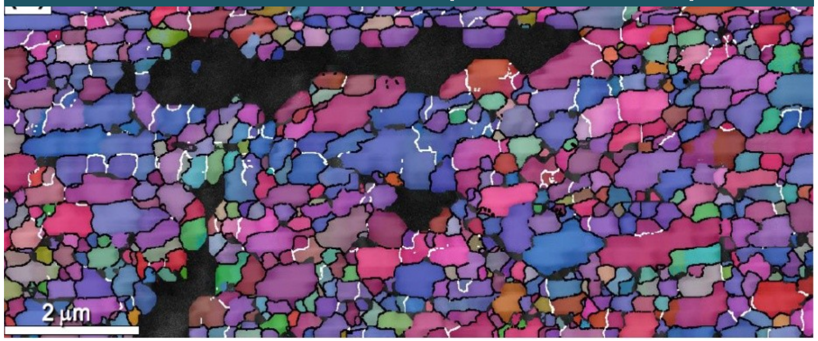
- *DOE/FC R&D Core Materials (led by S.A. Maloy of LANL)*
- *I-NERI Collaboration (US-Korea) for Developing 9Cr NFA with High Toughness/Third (last) Yr*
- *T.S. Byun & D.T. Hoelzer (ORNL)/J.H. Yoon (KAERI)*

- **Low toughness issue in high strength NFAs (Advanced ODS Steels)**
- **Proposed approach for toughening Fe-9Cr NFAs**
- **Integration of process technologies for high toughness NFAs**
- **Microstructural characteristics & stability of developed materials**
- **High temperature mechanical properties**
- **Summary & further studies**

High Temp. Fracture Process & Toughness



Tensile fracture at 900°C (14YWT-SM10).

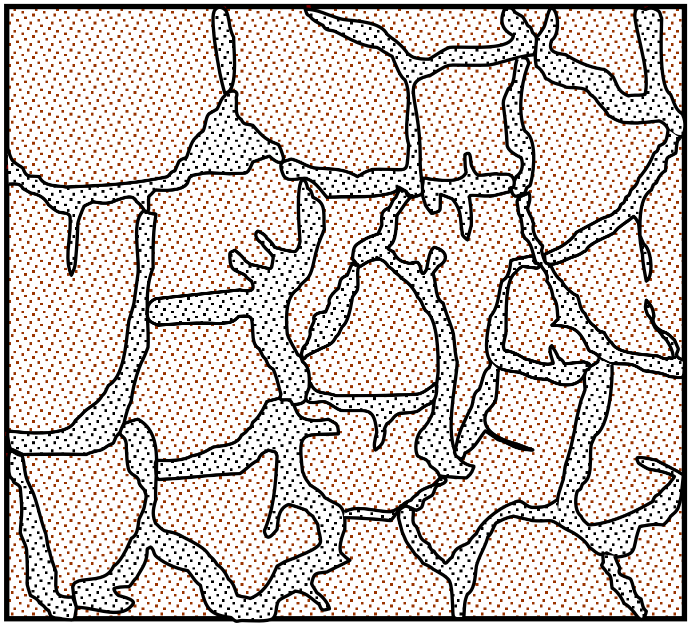


- **Microcracks propagate along grain boundaries without significant deformation: Low energy GB decohesion results in low fracture toughness.**

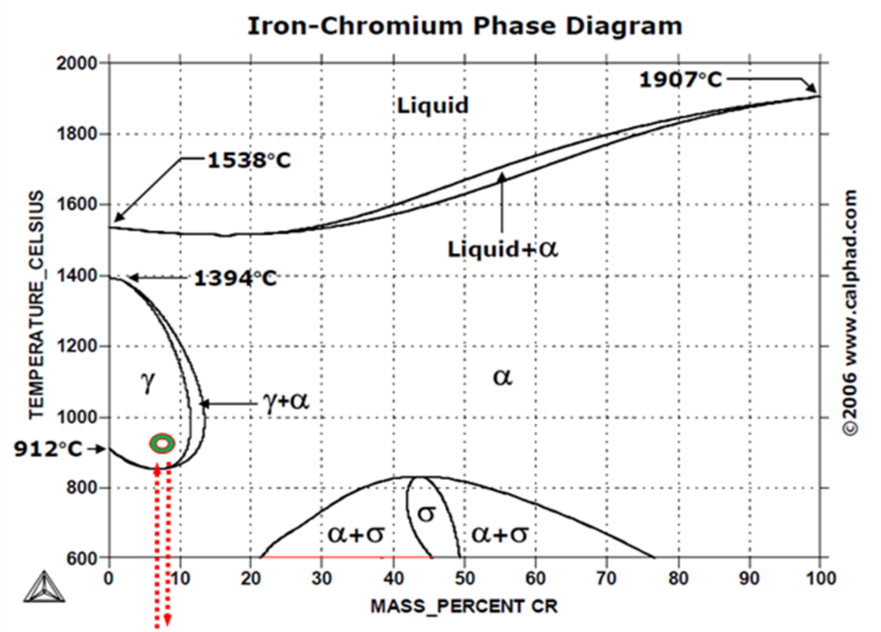
Application to reactor core components requires high toughness for both irradiation performance & manufacturing process:

- ***Low DBTT shift; resistance to radiation-induced embrittlement***
- ***High irradiation & thermal creep strength; high swelling resistance***
- ***Deformability & cracking resistance for tube drawing and forming processes***

Approach for Toughening NFAs: Modification of Grain Boundary



- Erase/modify weak grain & aggregate boundaries for higher bonding.
- Diffusion is greatly enhanced with partial phase transformation, and further enhanced by plastic deformation.



Heating to an austenite (γ) temperature and cooling after partial α→γ transformation.

Designed to enhance diffusion bonding at grain boundaries:

I. Partial phase transformation (intercritical annealing)

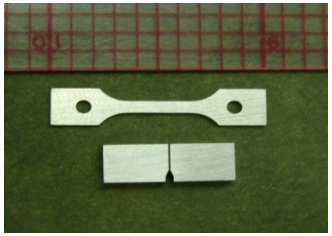
II. Partial phase transformation along with plastic deformation (controlled rolling)

Process Technology: Production of Base Materials (NFA 9YWTVs)

Two alloy power heats (8 kg each) have been produced by gas atomization process at ATI Powder Metals:
Fe-9Cr-2W-0.4Ti-0.2V-0.12C+0.3Y_2O_3 &
Fe-9Cr-2W-0.4Ti-0.2V-0.05C+0.3Y_2O_3

Ball milling for 40 hours in Zoz CM08 machine (6 loads)/ Canned & degassed (6 cans, 920g each)

Extrusion below 850°C & Cut into 4 inch long coupons



Characterization

- **Post-Extrusion TMT Optimization**
- **Micro & High T. Mechanical Characterization**
- **Feedbacks for new processing**

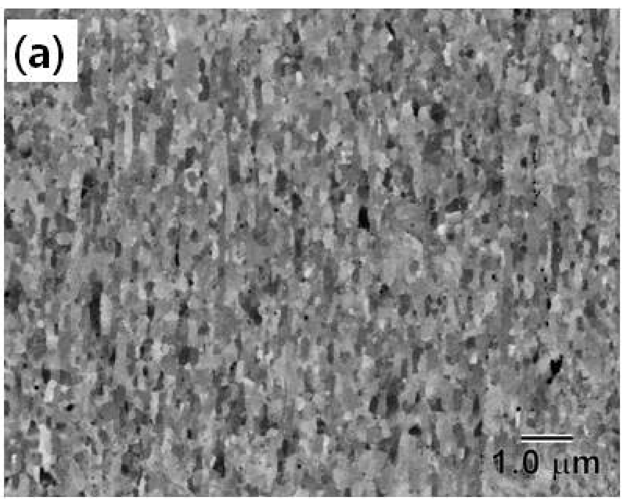
Base Materials:
NFA 9YWTV-PM1 & PM2

Chromium (ferrite stabilizer) equivalent of NFAs:

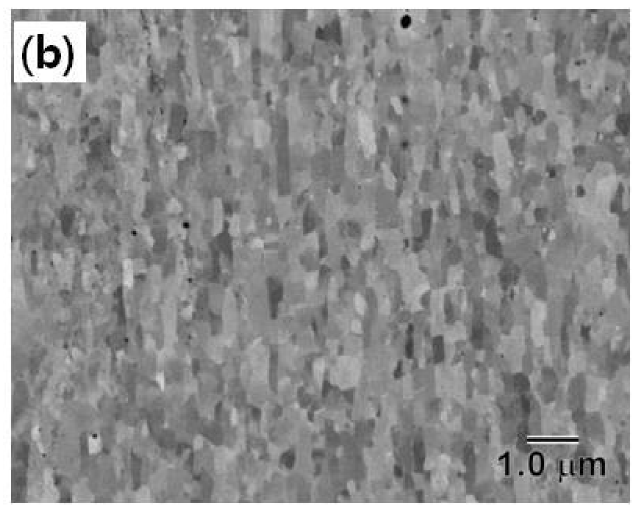
- ***14YWT (17%)***
- ***9YWTV-PM1 (10%)***
- ***9YWTV-PM2 (11.7%)***

****α to γ transformation needs Cr-eq < ~12%***

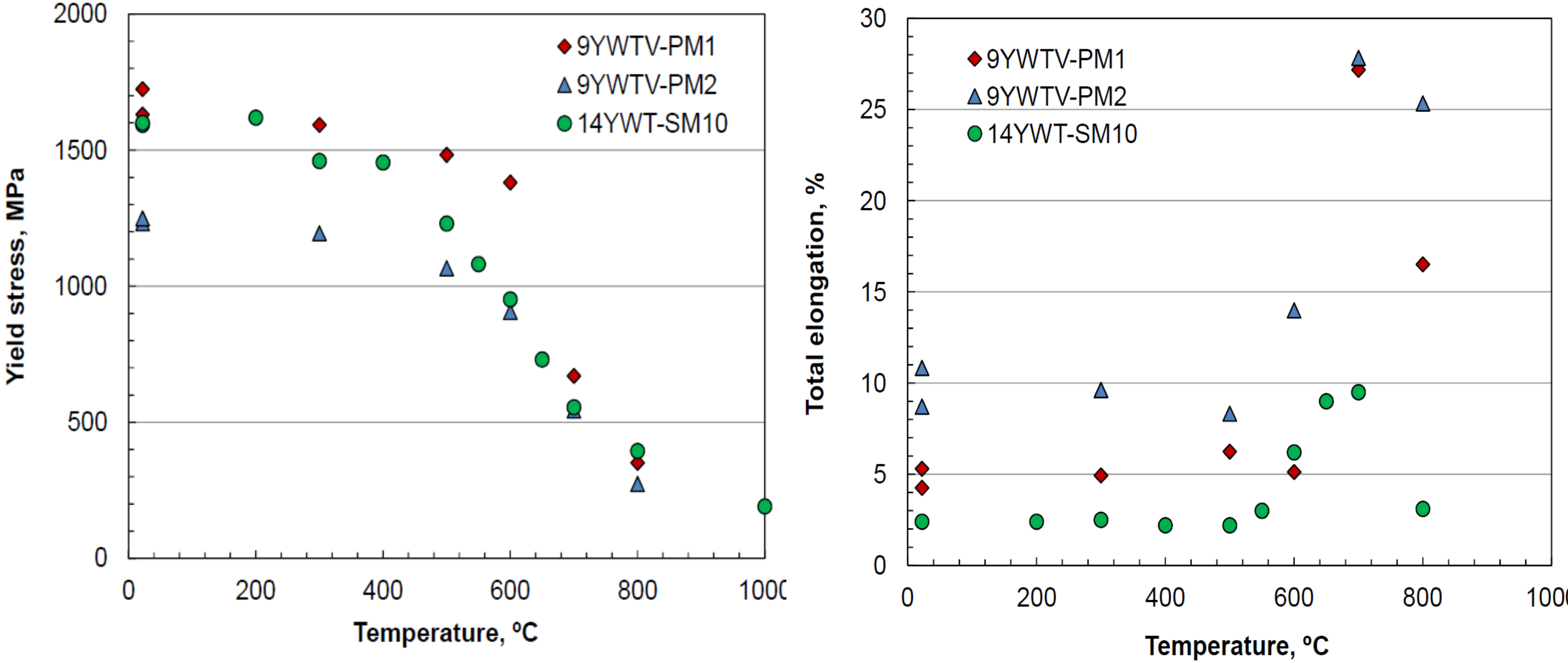
9YWTV-PM1
Fe-9Cr-2W-0.4Ti-0.2V-0.12C-0.3Y_2O_3



9YWTV-PM2
Fe-9Cr-2W-0.4Ti-0.2V-0.05C+0.3Y_2O_3

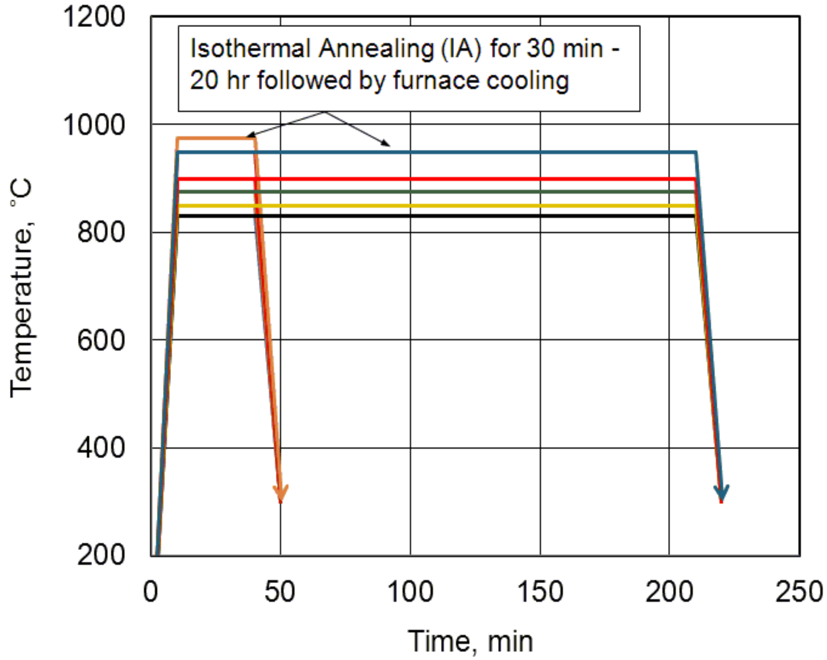


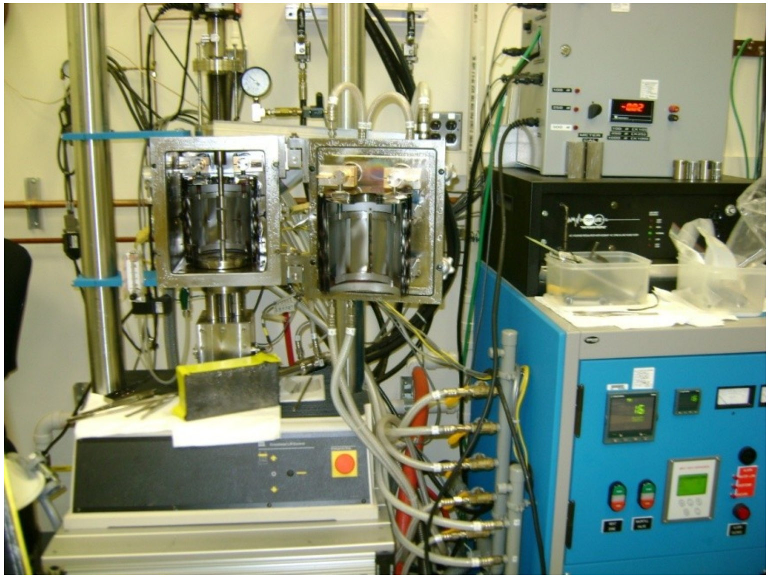
Base Materials: Strength & Ductility of 9YWTVs & 14YWT



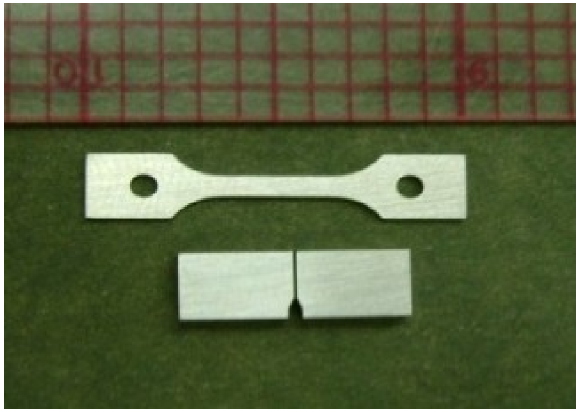
- New base NFAs retain YS higher than 500 MPa at 700°C.
- Improved ductility is measured for both new NFAs.

Process Development & Optimization: Isothermal Annealing (IA)



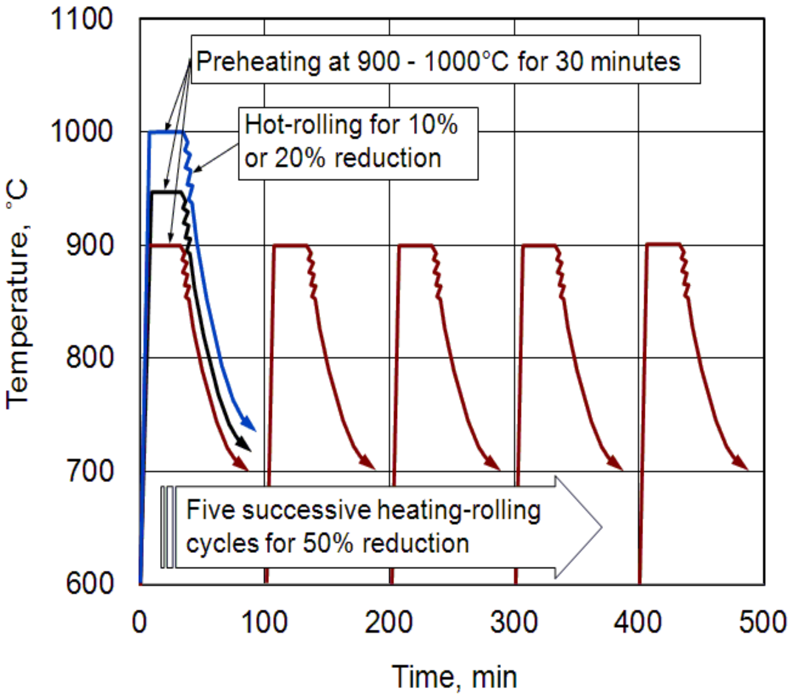


Servohydraulic testing system (MTS 858 table top model) equipped with high vacuum (10^{-7} torr), high temperature (1000°C) furnace (Oxy-Gon, custom model, Ta heating elements)



Mini tensile (SS3) and fracture (TPB & DCT) specimens

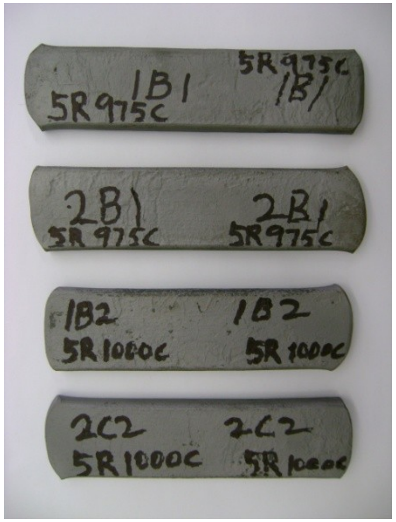
Process Development & Optimization: Controlled Rolling (CR)



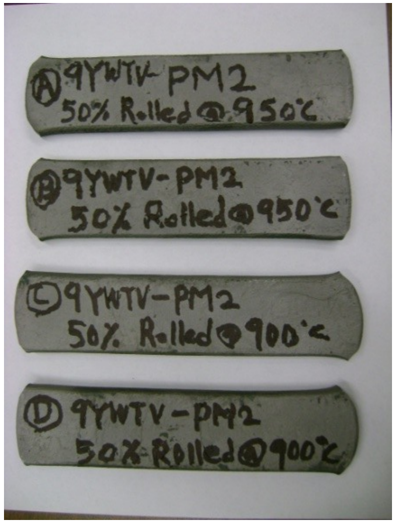
- **The as-extruded coupons were hot-rolled in controlled conditions: at 900 – 1000ºC for 20 or 50% total thickness reduction.**

Production of 9Cr NFAs

- 1st Production: 9YWTV-PM1 & PM2
- About 6 kg total
- Consumed for basic property examination and major mechanical properties



- 2nd Production: 9YWTV-PM2 only
- About 3 kg total
- Consumed for major mechanical property tests
- Further studies



Process Development & Optimization: TMT Matrix

- ❑ 9YWTV PM1: Fe(bal.)-9Cr-2W-0.4Ti-0.2V-0.12C
- ❑ 9YWTV PM2: Fe(bal.)-9Cr-2W-0.4Ti-0.2V-0.05C

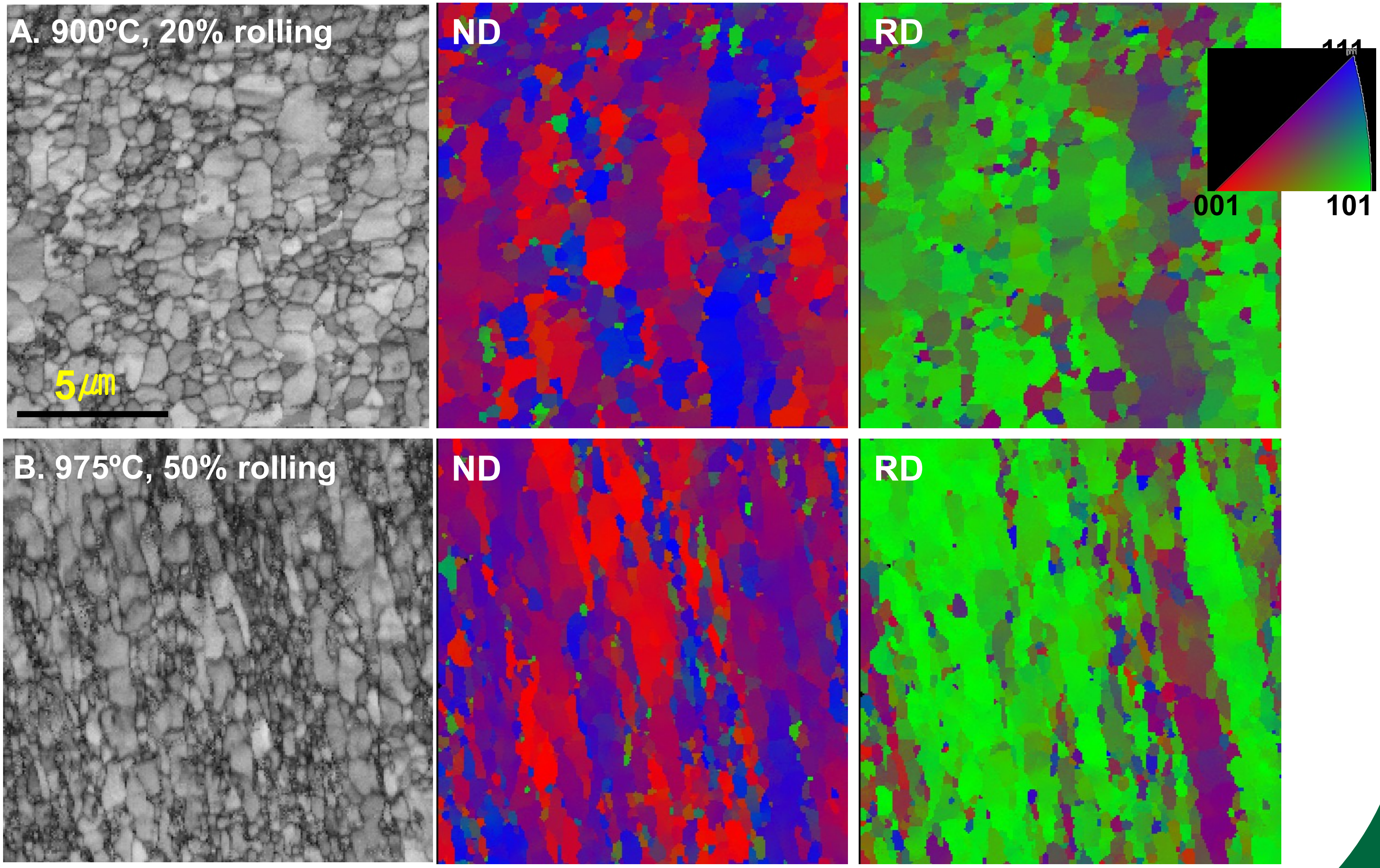
Isothermal (Intercritical) Annealing (IA)

Annealing Time/Temp.	30 or 60 min	200 min	20 hr
830 °C	O	O	
850 °C	O	O	O
875 °C	O	O	
900 °C	O	O	
950 °C	O	O	
975 °C	O		

Controlled Rolling (CR) with IA

Rolling Strain/Temp.	20%	50%	Remark (Bend Bar)
900 °C	O	O	L-T/T-L
925 °C		O	L-T
950 °C		O	L-T
975 °C		O	L-T
1000 °C		O	L-T

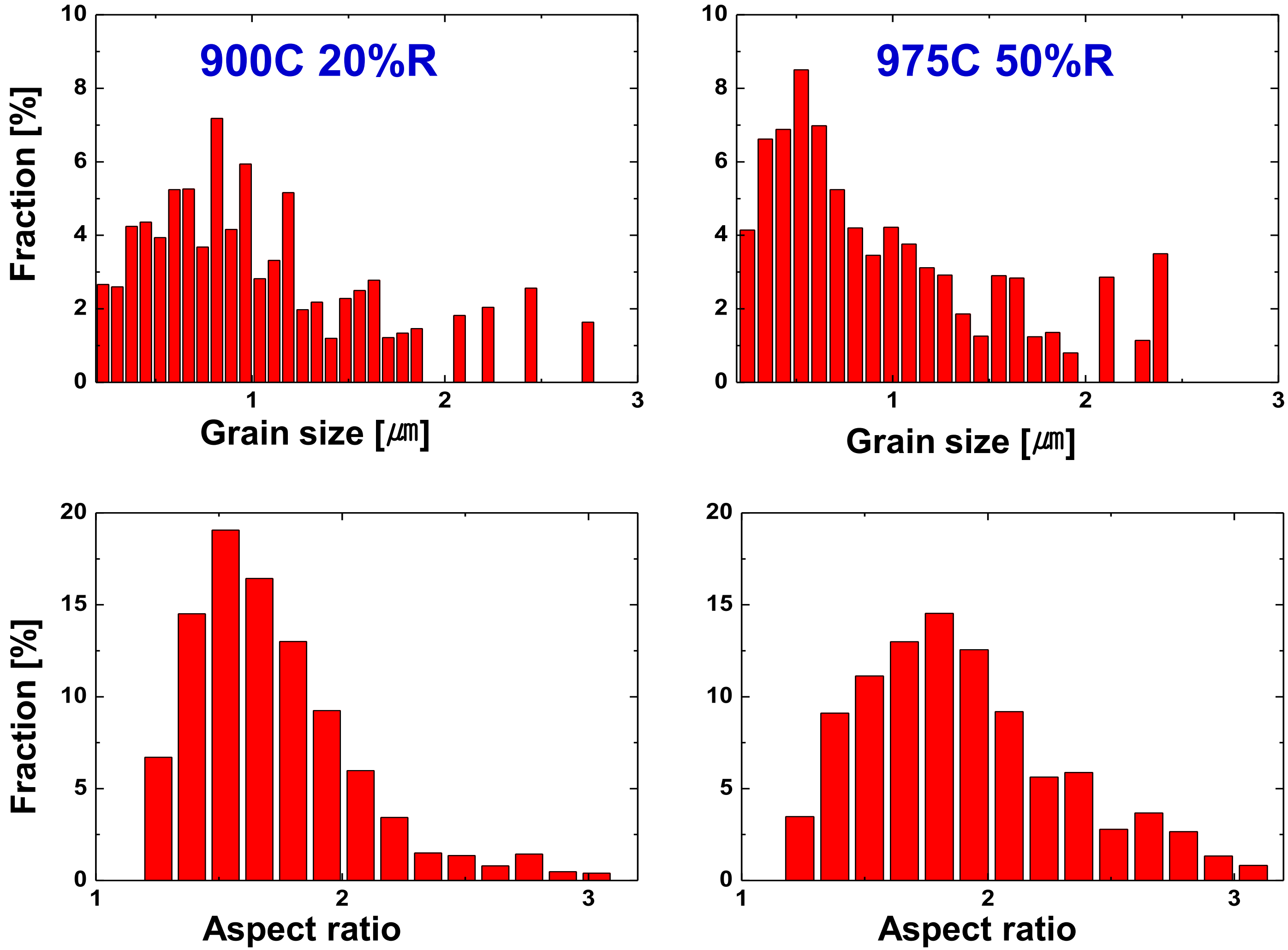
EBSD Images of 9Cr NFAs (PM2) after CR



- Relatively equiaxial in A; higher aspect ratio in B, RD//110 grains dominant

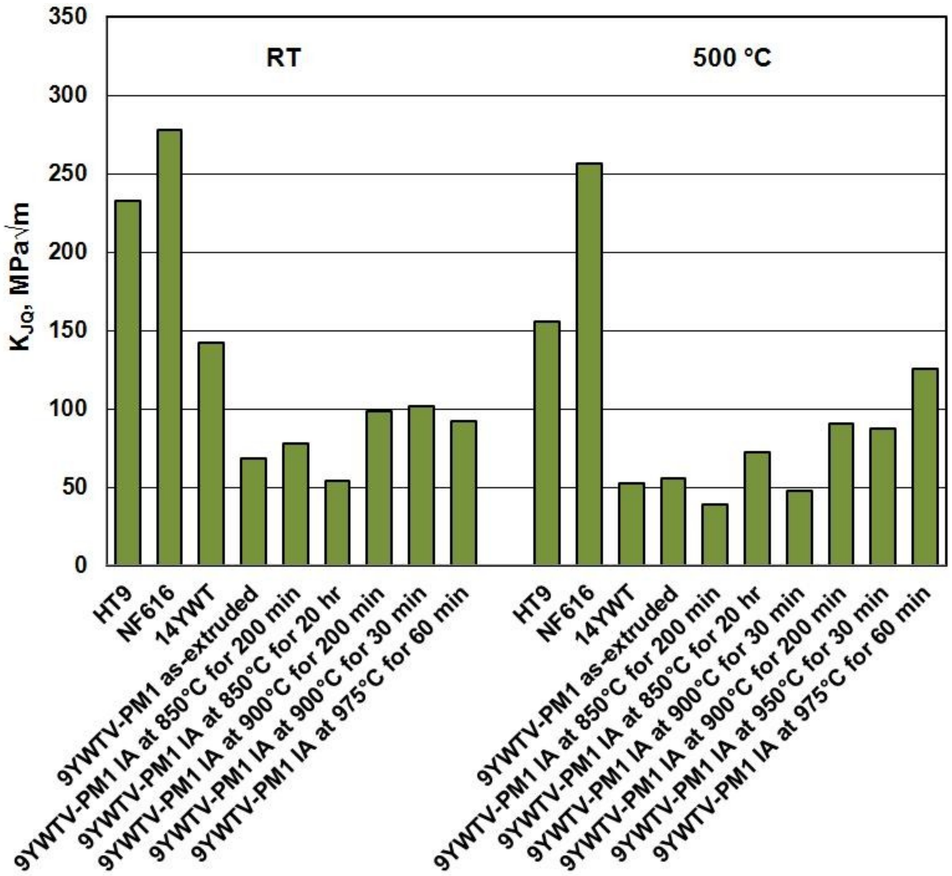
Grain Size of 9Cr NFAs (PM2) after CR

(Equivalent Circular Diameter)



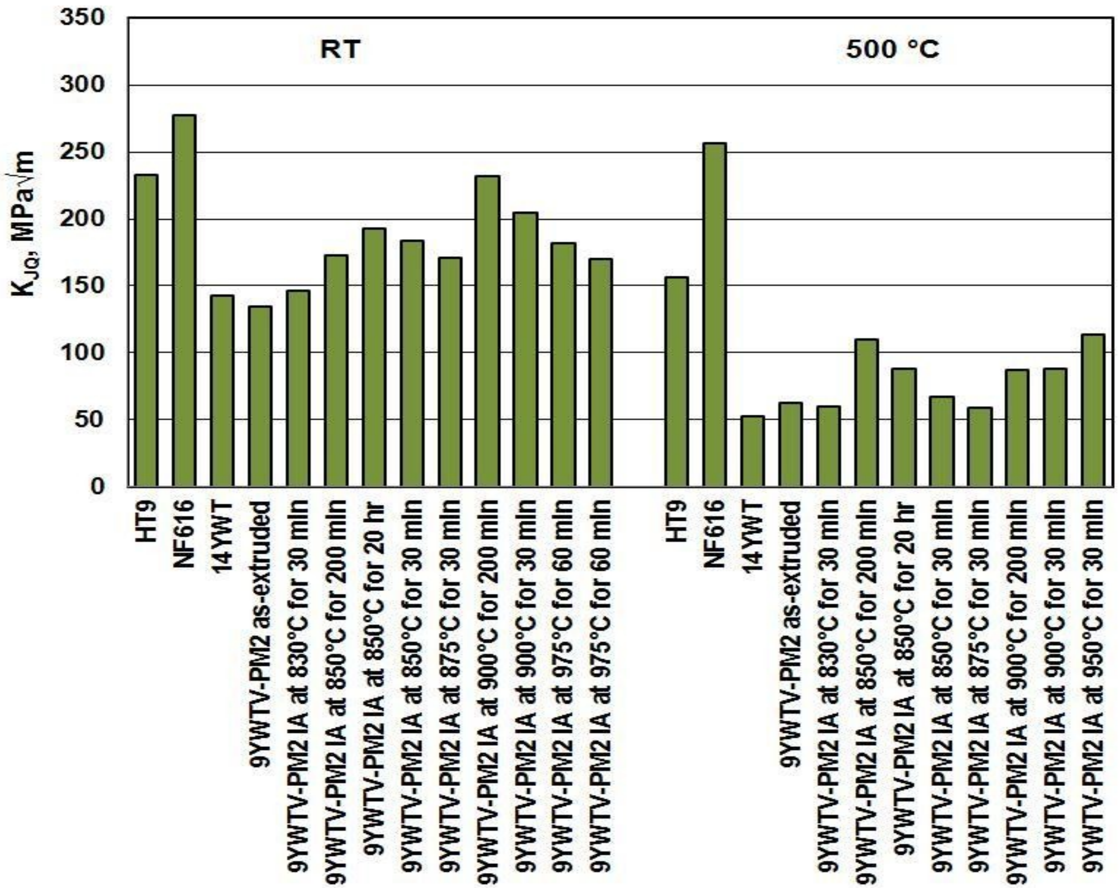
- Grains after 900C rolling are slightly larger and have are more equiaxial.
- Grains grow from 100 – 300 nm in as-extruded condition to 300 – 900 nm after CR.

High Temp. Fracture Test Data
Effect of IA in 9YWTV-PM1



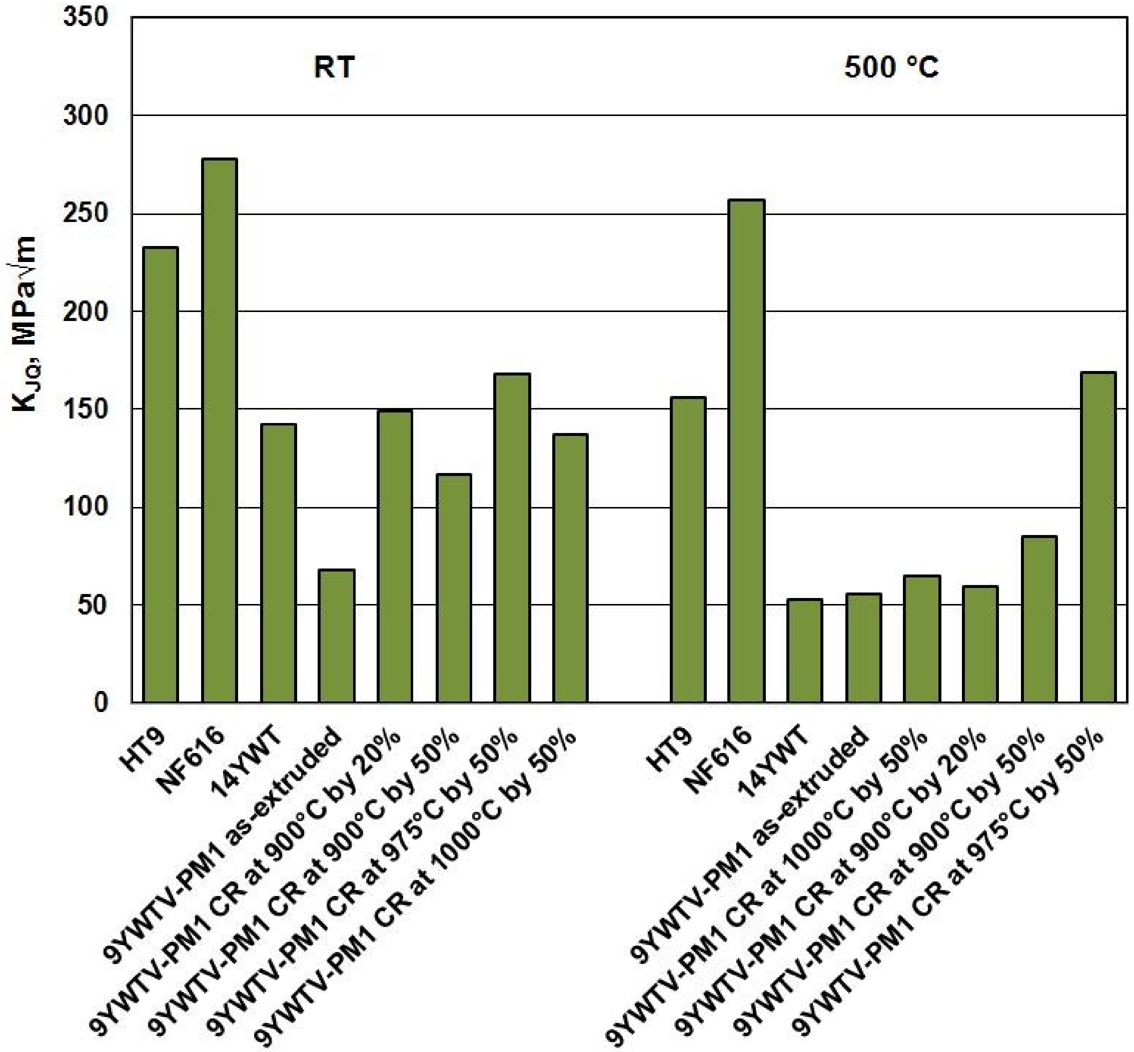
- Small change of strength is observed after annealing at 975°C.
- No evidence of fracture toughness improvement is found in annealed 9YWTV-PM1 except for the 975°C annealed specimen tested at 500°C.

High Temp. Fracture Test Data
Effect of IA in 9YWTV-PM2

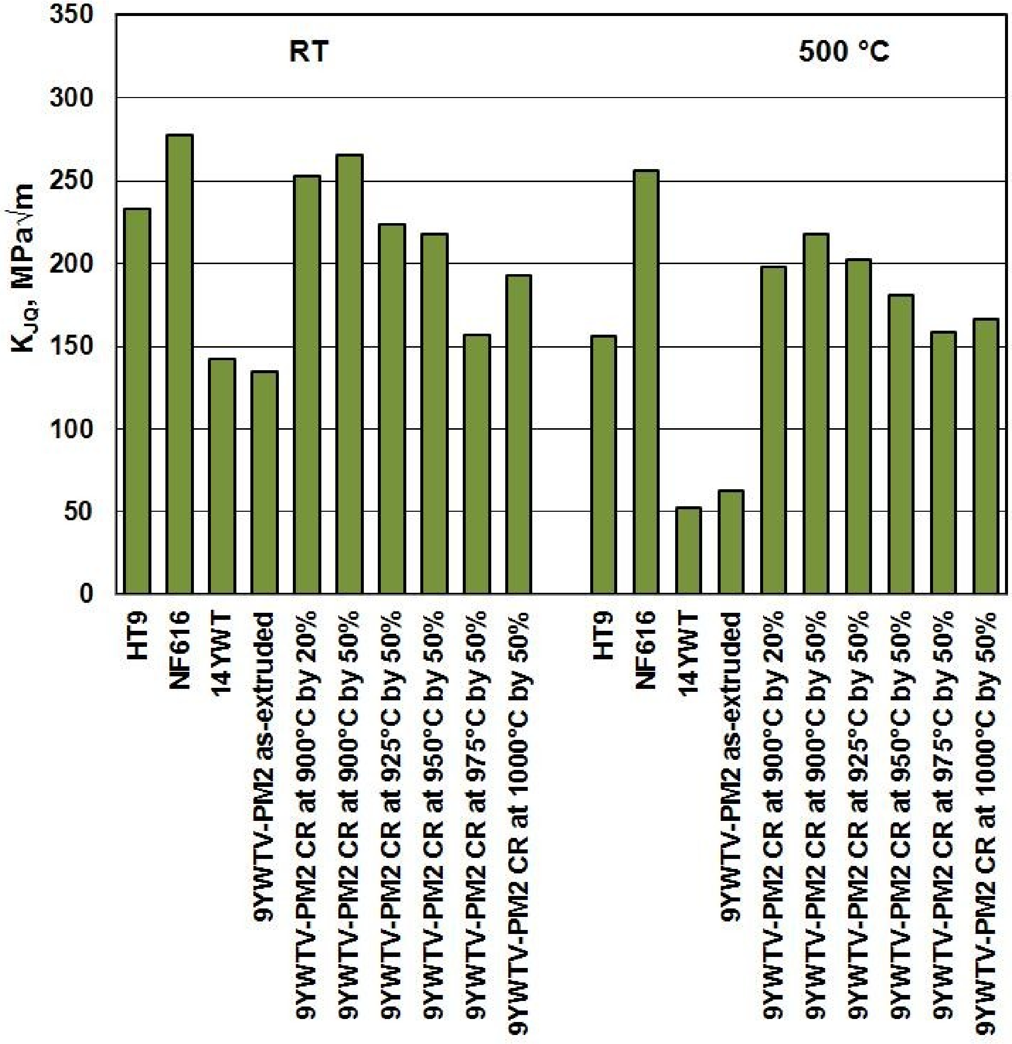


- **Fracture toughness is improved in 9YWTV-PM2 by intercritical annealing, but further improvement is desirable especially at high temperatures.**

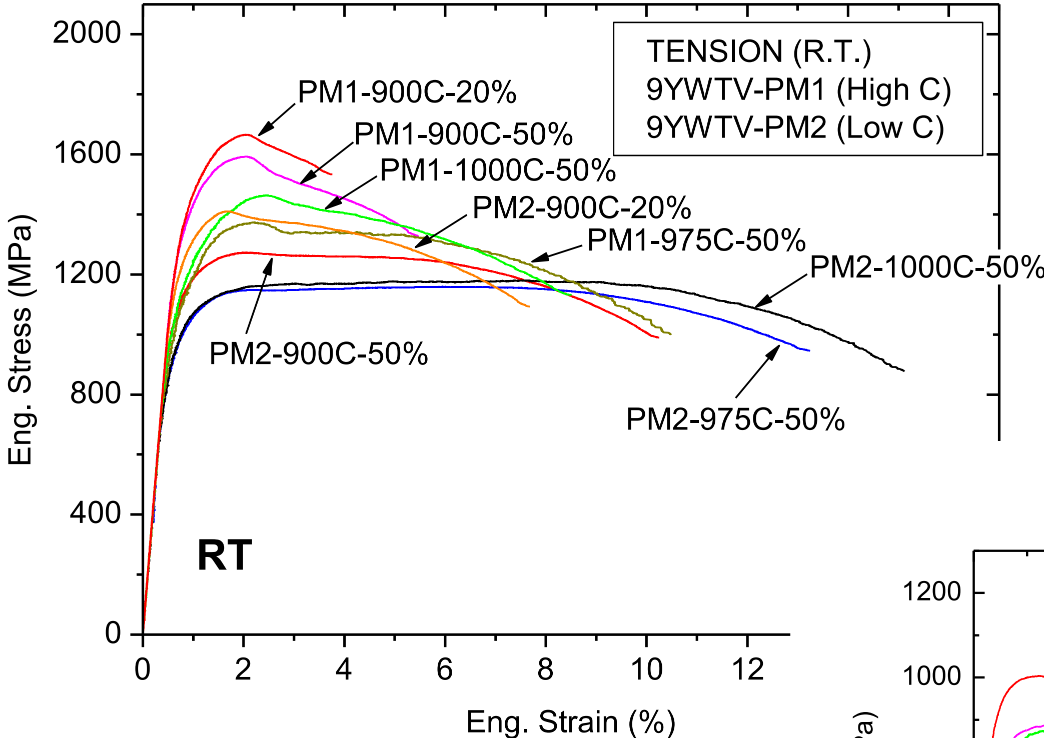
High Temp. Fracture Test Data
Effect of CR in 9YWTV-PM1



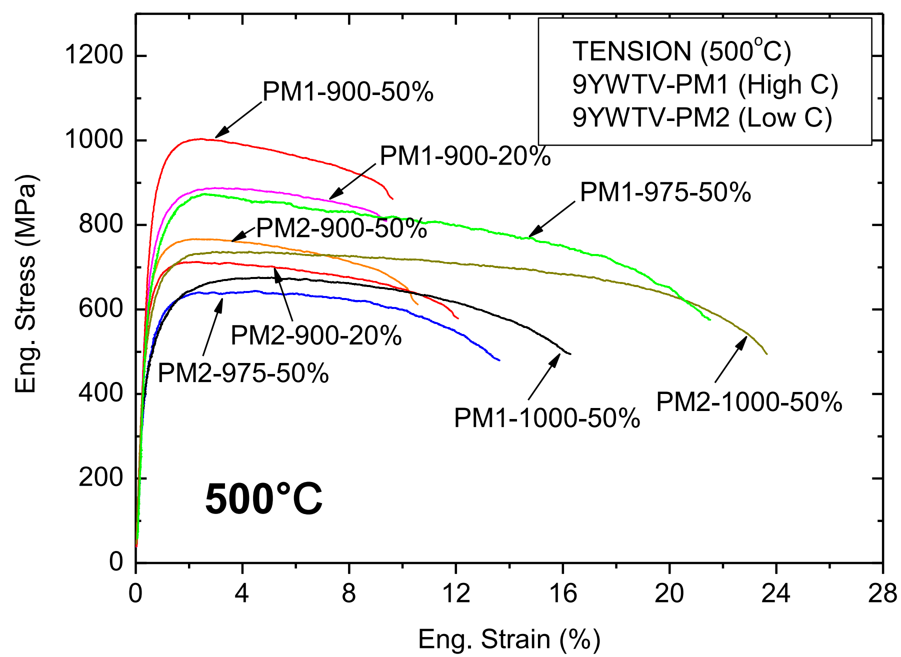
High Temp. Fracture Test Data
Effect of CR in 9YWTV-PM2



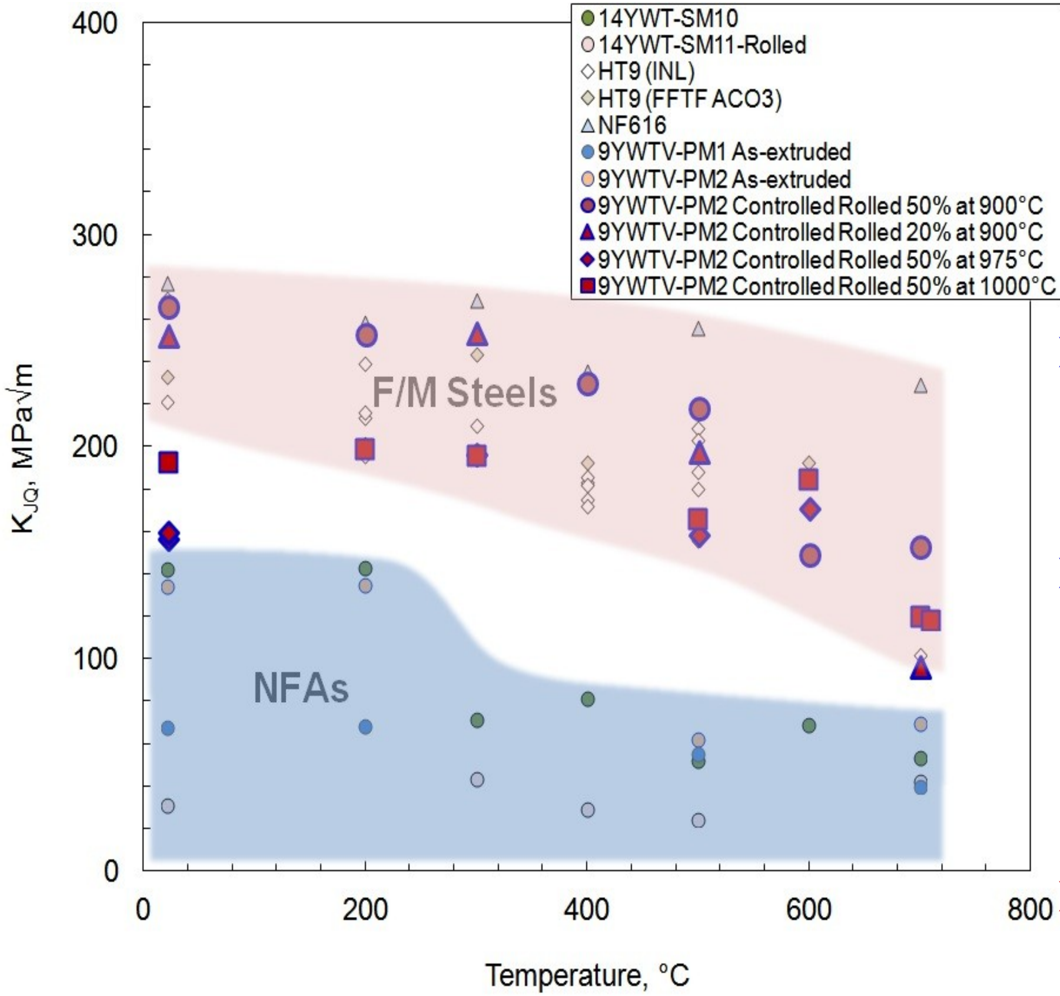
Uniaxial Tensile Curves
Effect of CR in 9YWTV-PM1 & PM2



- Strength decreased with the rolling temperature and reduction of thickness.
- High temperature strength was reduced by CR (by ~200 MPa).

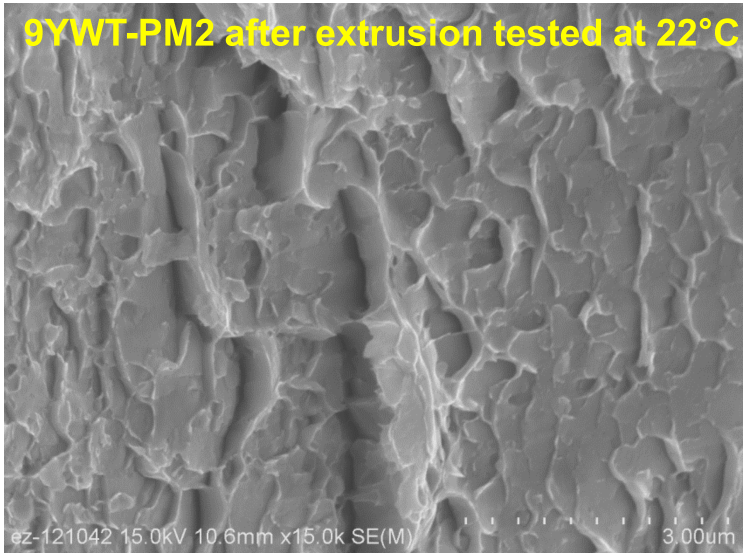


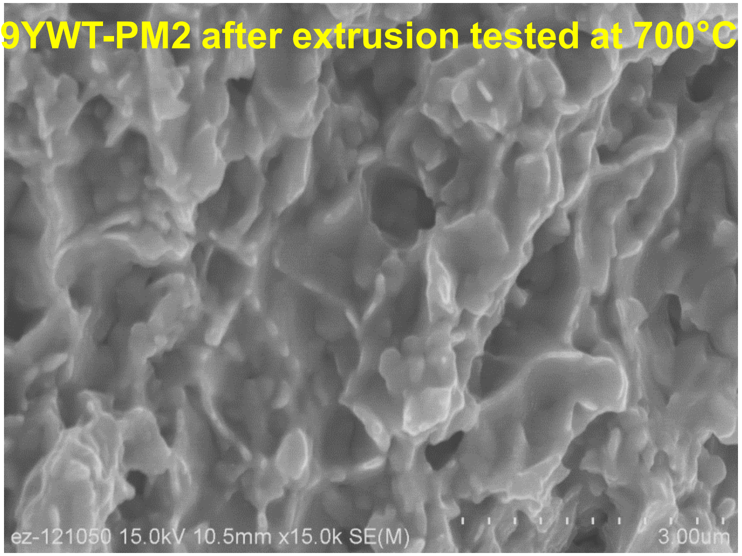
High Temp. Crack Resistance Test
Effect of CR in 9YWTV-PM2

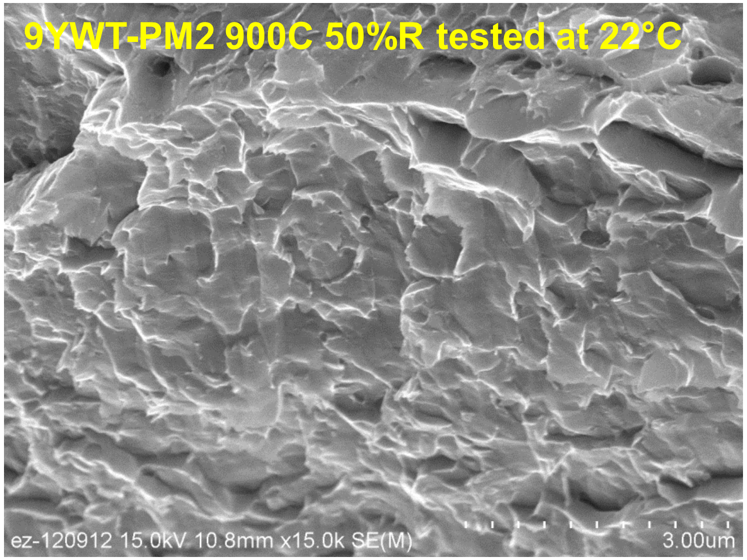


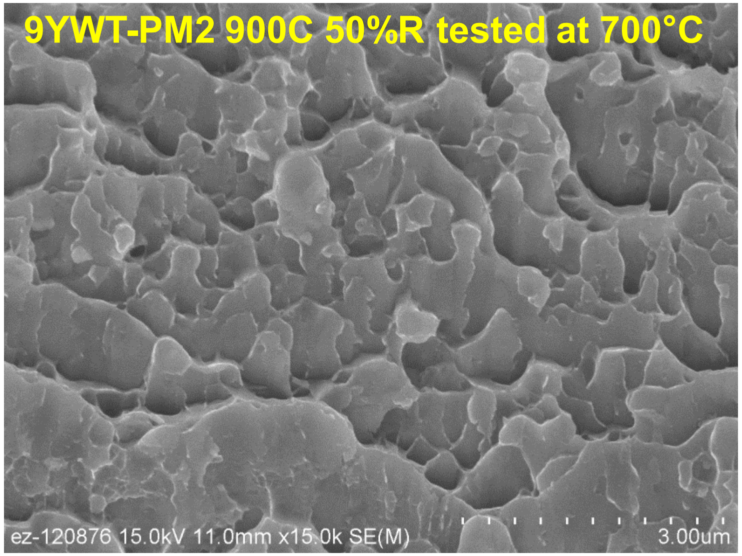
- **Improvement of fracture toughness in controlled rolled 9YWTV-PM2 is significant.**
- **The 9YWTV-PM2 controlled rolled at 900°C resulted in the best fracture toughness among NFAs, which is as high as those of non-ODS F/M steels.**
- **TMT condition is selected for detailed characterization and further studies: 900°C 50%R.**

Effect of CR on Fracture Mechanism in 9YWTV-PM2





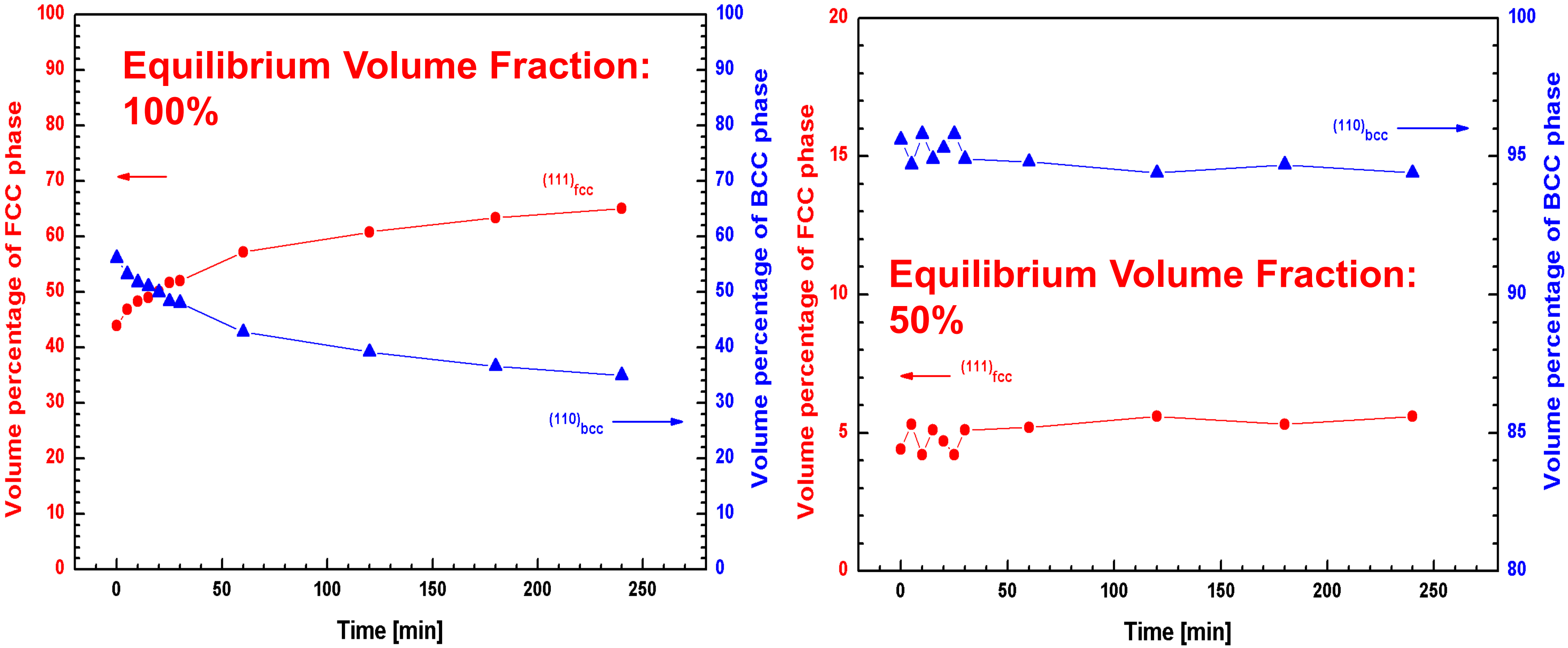




- **A change in high T fracture mechanism from boundary decohesion to formation of flake-like shear tongues.**

Issue in Process Development: Microstructural Characterization by In-Situ X-Ray

Volume fractions of FCC phase (red) and BCC phase (blue) in 9YWTV-PM1 (left) and in 9YWTV-PM2 (right) after heat-treatment at 1000°C



- The volume fraction of FCC in 9YWTV-PM1 gradually increases with time up to about 65%, while that in 9YWTV-PM2 appears to saturate at about 5%.
- This sluggish phase transformation in PM2 might be due to the early depletion of carbon content in ferrite, which is a strong austenite former.
- Detection accuracy is low with these nanostructured materials.

Summary & Further Studies

1) Very high strength can be achieved in NFAs at the expense of fracture toughness and ductility. Low energy decohesion at boundaries causes poor fracture resistance at high temperatures.

2) Isothermal annealing & controlled hot-rolling were used to strengthen the powder-metallurgy produced weak boundaries by enhanced diffusion bonding.

3) Improvement of fracture toughness in controlled rolled 9YWTV-PM2 was significant: fracture toughness was as high as those of non-ODS F/M steels.

4) In particular, the 9YWTV-PM2 controlled-rolled at 900°C resulted in the best fracture toughness among NFAs (> 150 MPa√m over RT - 700°C). High toughness (engineering grade) NFA has been developed.

5) Detailed characterization & further studies on selected materials need to be done: (a) delayed phase transformation, (b) stability of nanoclusters, (c) grain growth, (d) irradiation experiments of the base 9Cr NFAs and high toughness NFAs, and (e) testing in thin-walled tube form.