

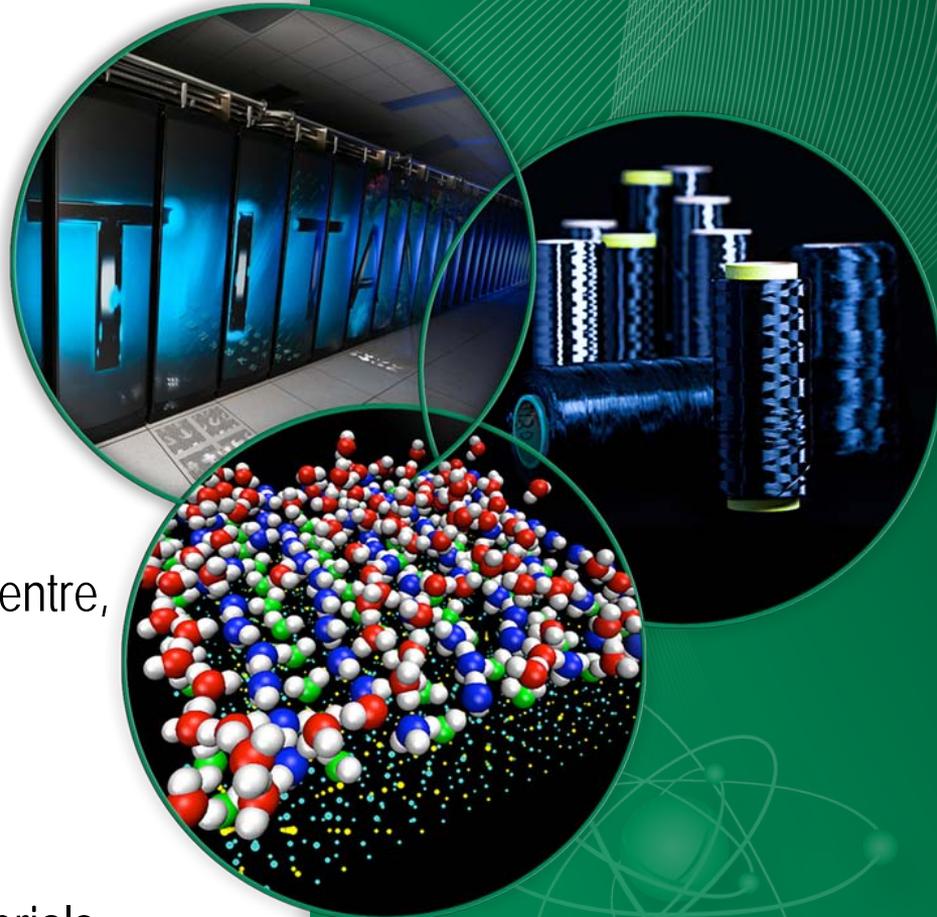
Improving the Performance of Creep-Strength-Enhanced Ferritic (CSEF) Steels

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Bernd Kuhn, Michal Talik (Jülich Research Centre,
Germany)

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Pittsburgh, PA

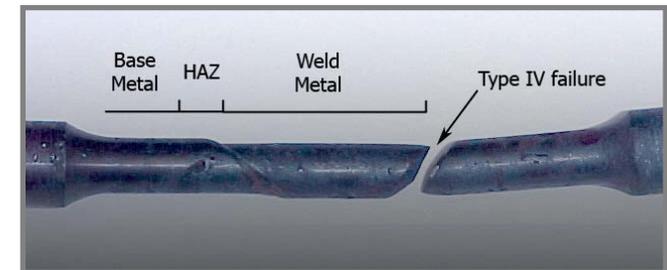


Why “Improvement” of CSEF required?

- Majority of structural components for High-Efficiency Boilers (F/M or bainitic).
 - Headers & piping, boiler tubing: T23, T/P91, T/P92
 - Lots of weldments applied in factory/field.
- Life of weldments shorter than Base Metal.
 - Type IV failure shortens the material life.
 - Caused by weakened microstructure at HAZ.
- Minimizing/ eliminating Type IV failure is essential for CSEF steel weldments.
 - Technical attempts limitedly succeed to minimize Type IV failure (e.g. narrow HAZ width as possible).
 - Numerous studies proved that Type IV failure of traditional CSEF steel weldment is unavoidable.
 - To minimize: *Optimization of heat treatment*
 - To eliminate: *New alloy development*



CSEF Steel Header Fabrication, Courtesy Prof. Masuyama



Weld Joint Strength Reduction Factors ($WSRF = \sigma_{weld} / \sigma_{base\ metal}$) for CSEF steels can be as low as 0.5 at $\sim 600^\circ\text{C}$.

Approaches have been made/to be made

Optimization of HT

- Target **existing CSEF steel** (e.g. *Grade 91*).
- Apply optimized thermo-mechanical heat treatment (*feasible, inexpensive*).
- Based on scientific understanding (*required cumulative efforts*), as well as breakthrough concepts.

New alloy development

- Target **new alloys with optimized properties** (e.g. *higher temperature limits, Type IV failure resistance*).
- Improved mechanical properties demand increased corrosion resistance.
- Maintain competitiveness with existing CSEF steels (*cost, fabricability*).

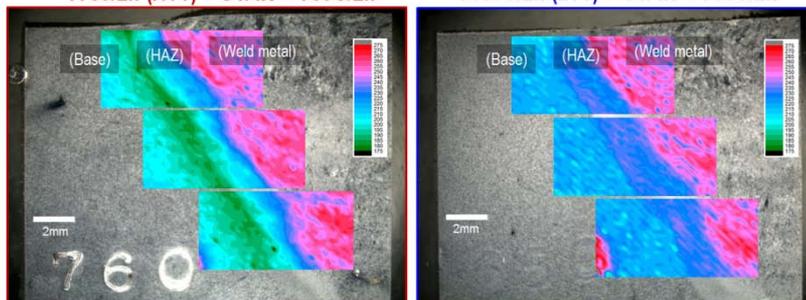
Industry demands

Scientific challenges

Improvement of HAZ strength through Non-standard HT

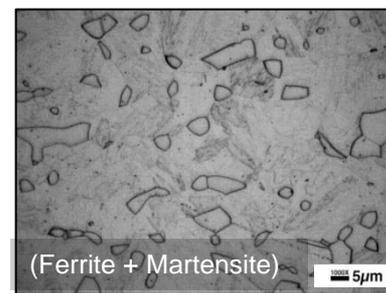
T760/2h (HTT) + GTAW + 760C/2h

T650C/2h (LTT) + GTAW + 760C/2h



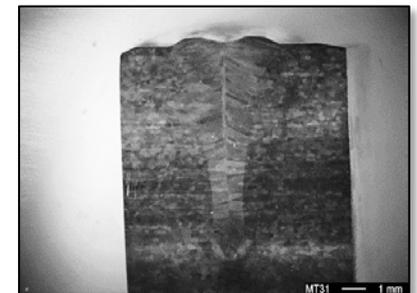
Al-containing 9Cr steel

(for improved oxidation resistance)



New high-Cr ferrite steel

(essentially free from Type IV failure)



Activities in FY13

Optimization of HT:

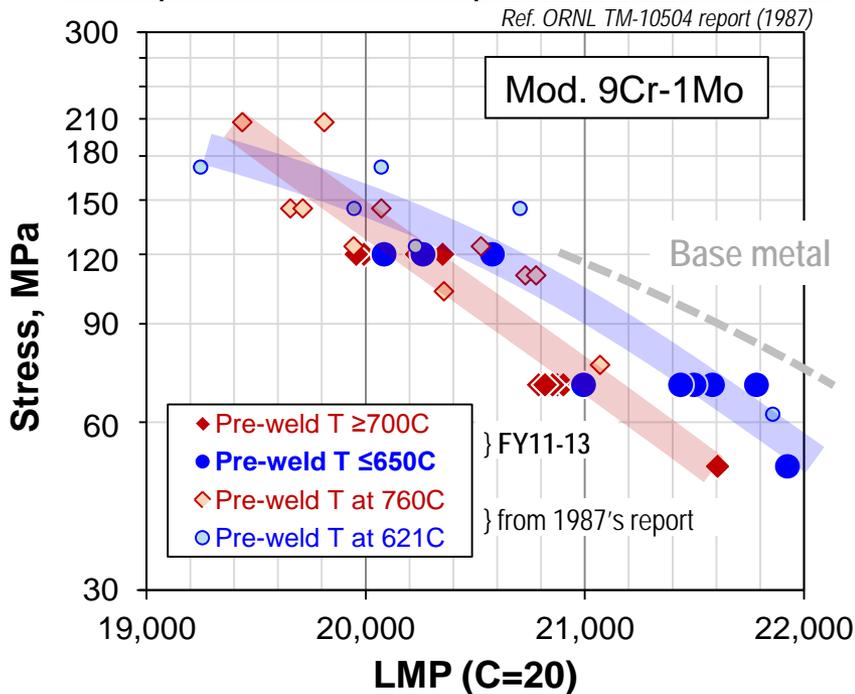
- Update on non-standard pre-weld heat treated 9Cr steel weldments:
 - *Creep-rupture testing / In-situ creep deformation analysis*
 - *Charpy impact testing*
- New TMT approaches:
 - *Two-step tempering to control $M_{23}C_6$ size and distribution*
 - *Aus-forging/-aging to promote MX precipitation*
- Interaction with ASME boiler and pressure vessel code (Section II)

New Alloy Development :

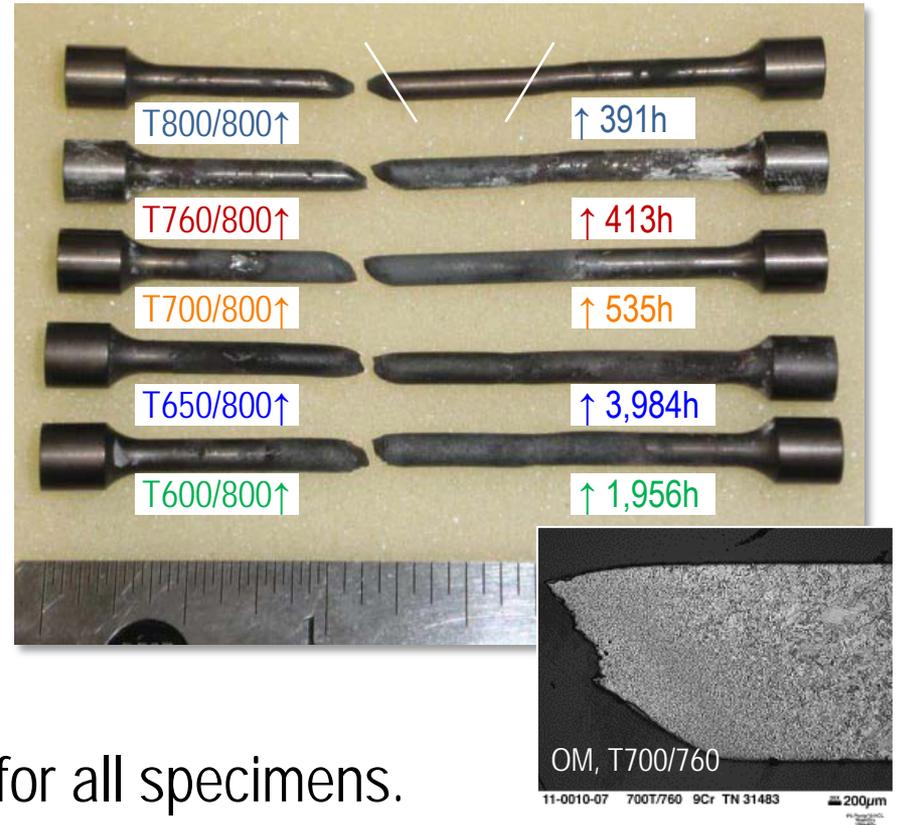
- Al-containing 9Cr steels for improved oxidation resistance
- Collaboration with Forschungszentrum Jülich GmbH, Germany for weldment development of a new high-Cr ferritic steel, Crofer 22H[®]

Optimization of Pre-weld Tempering Temperature for Improved Properties

Creep test results (rep.1987 + FY11-13)



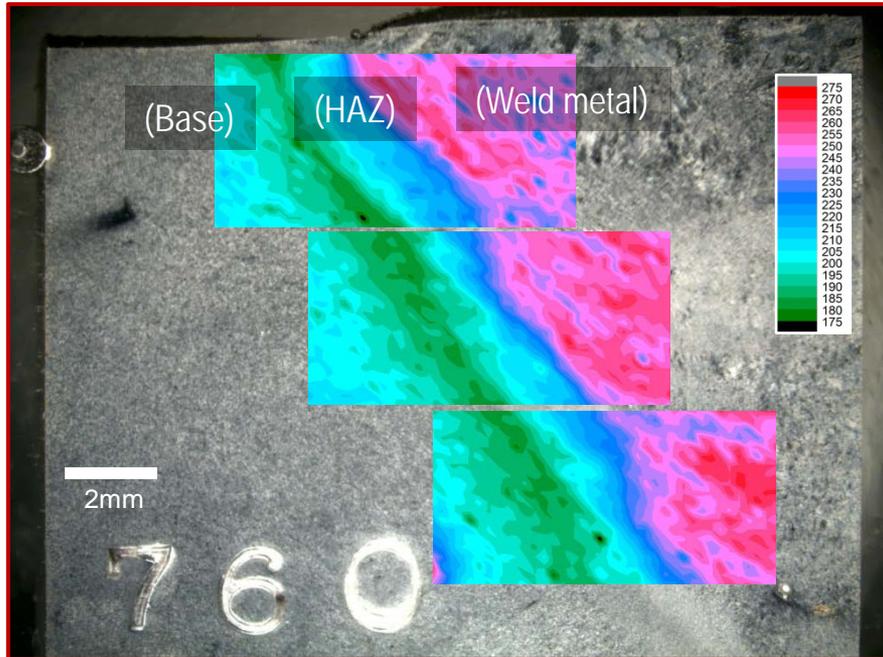
Creep-ruptured specimens (650C/70MPa)



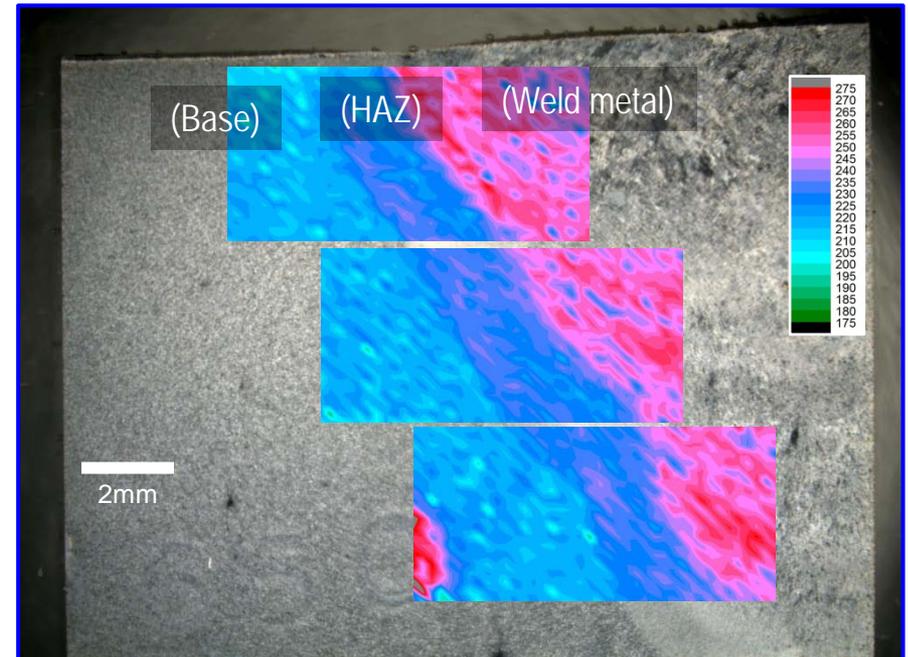
- Creep-rupture took place at FGHAZ for all specimens.
- "Improvement" of HAZ microstructure is the key.

Different Hardness Distribution in HAZ after PWHT

T760/2h (HTT) + GTAW + 760C/2h



T650C/2h (LTT) + GTAW + 760C/2h



- Distinct soft zone formed on FGHAZ (typically observed in CSEF steel weldments).

- No soft zone formed in HAZ.

LTT Resulted in Complete Dissolution/ Re-Precipitation of $M_{23}C_6$ after PWHT

□ In-situ Diffraction Study at SPring-8, Japan (by X. Yu and S.S. Babu, OSU)



http://www.spring8.or.jp/ja/about_us/whats_sr/

- Simulated HAZ heat profile during high energy X-ray diffraction scan.
- Higher time resolution than conventional XRD.

Table: Microstructure evolution at fine grain heat affected zone

	Pre-weld temper	Weld (at FGHAZ)		PWHT
HTT (e.g. 760T/760)				
LTT (e.g. 650T/760)				

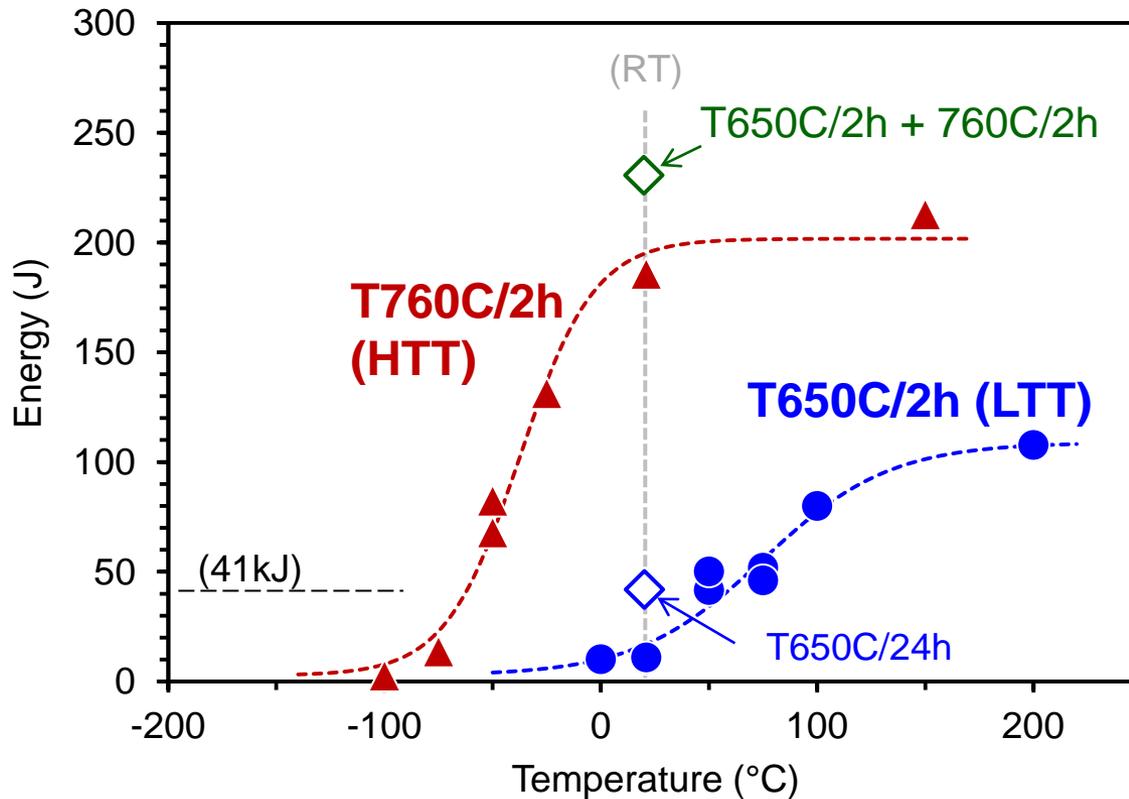
● : $M_{23}C_6$ ● : MX

X. Yu et al., *Acta Materialia*, Published in April 2013.

- Scientific understanding:
 - Refinement of $M_{23}C_6$ before welding is the key to improve creep properties after PWHT.
- Breakthrough concepts:
 1. to control $M_{23}C_6$ size and distribution = Two-step tempering
 2. to promote stable MX precipitation = Aus-forging/ aus-aging

LTT Resulted in Higher DBTT of BM

(Charpy impact test results)



DBTT (at 41kJ*)

➤ T760C/2h: -62°C

➤ T650C/2h: 58°C

*41kJ = engineering threshold absorbed energy between “brittle” and “ductile”

- Further tempering at 650C would not effectively increase the absorbed energy.
- PWHT (760C/2h) increases RT ductility of LTT sample drastically.
→ LTT + HTT (two-step tempering) can be used for pre-weld HT?

Two-step Tempering Needs Optimization

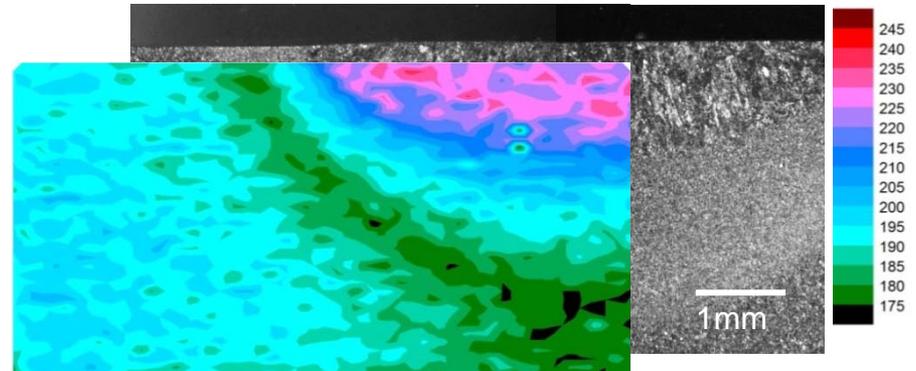
Trial GTAW of Two Step Tempered Gr91 (1050C/1h/AC + 650C/2h/AC + 760C/2h/AC)



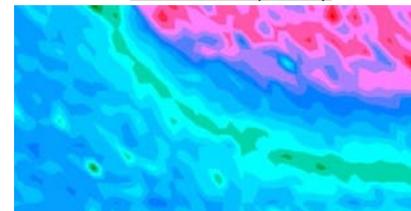
Pre-heated at 250F, and then welded at the condition of 125A, ~15V, ~8inch/min

- Try to achieve both “no weaken-band” and “better RT ductility”, through “ $M_{23}C_6$ refinement” and “full tempering”, respectively.

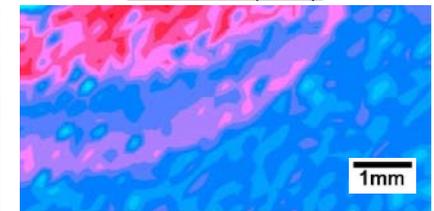
TST Hv map after PWHT (760C/2h)



N&T760 (HTT)



N&T650 (LTT)



- First trial resulted in similar Hv distribution to HTT sample, due to coarsened $M_{23}C_6$ in Two-step Tempered sample.

Applying Aus-forging + Aus-aging for 9Cr Steel Weldments

- ❑ Originally reported by Ron Klueh et al., ORNL (2005), for further improvement of creep properties of 9-12Cr steels (only the base metal studies).
- ❑ Increased amount of **stable, nano-scale MX by TMT** may also improve the creep resistance at FGHAZ, by minimizing the effect of $M_{23}C_6$ coarsening on the creep.
- ❑ Need to understand **the detailed transformation/precipitation kinetics**.

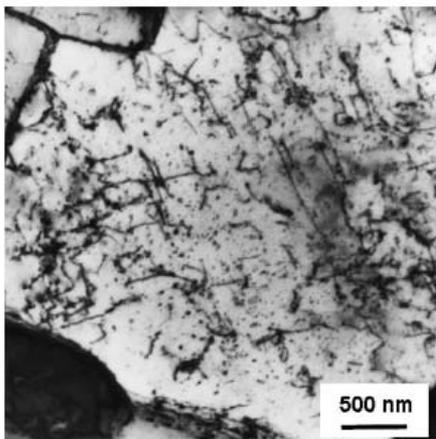


Fig. 2. Transmission electron microscopy photomicrograph of modified 9Cr-1Mo steel after the new thermo-mechanical treatment.

Ron Klueh et al., Scripta Materialia (2005)

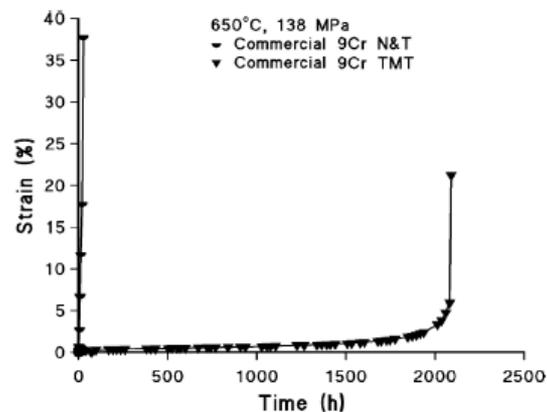
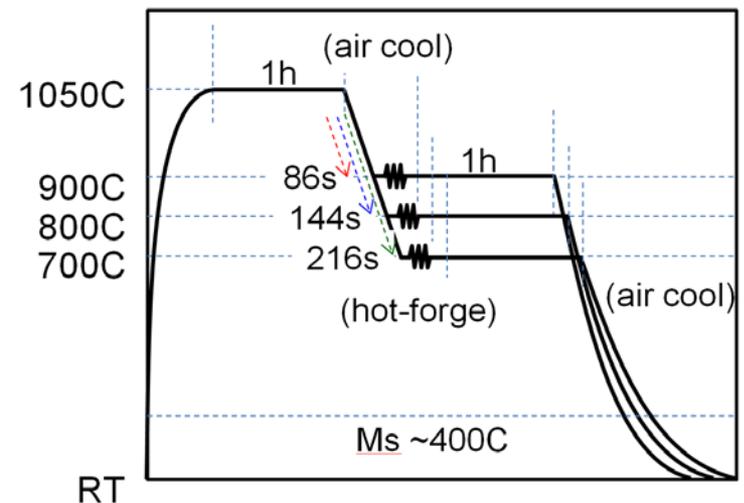
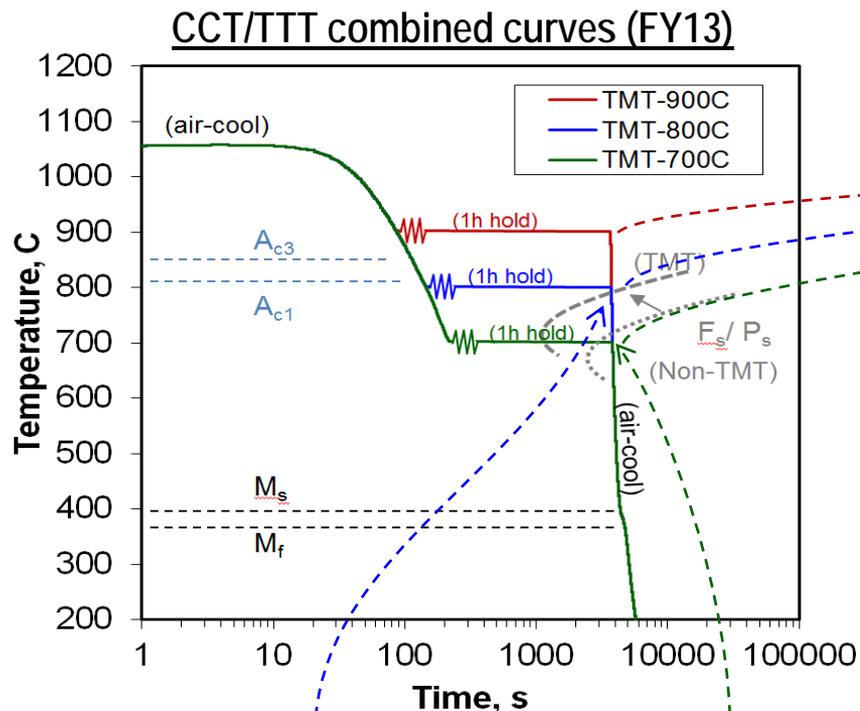


Fig. 5. Creep curves for the commercial 9Cr steel after the conventional normalizing-and-tempering (N&T) heat treatment and the TMT. Creep tests were at 138 MPa at 650 °C until the specimens ruptured.

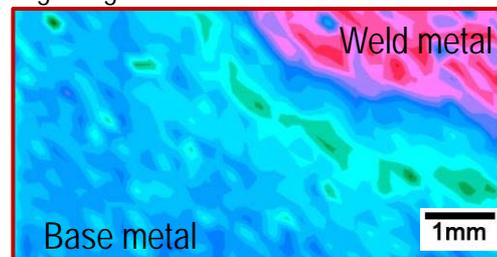
Test plan of aus-forging/-aging study (FY13)



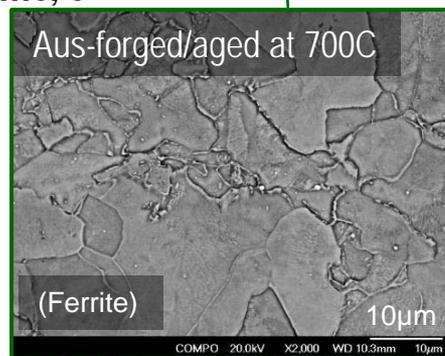
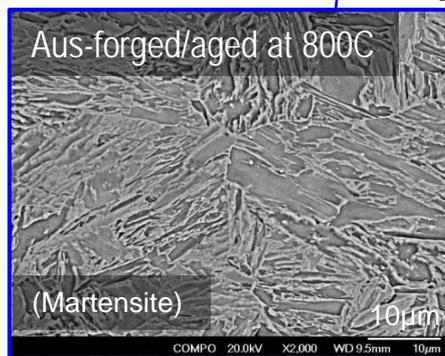
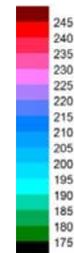
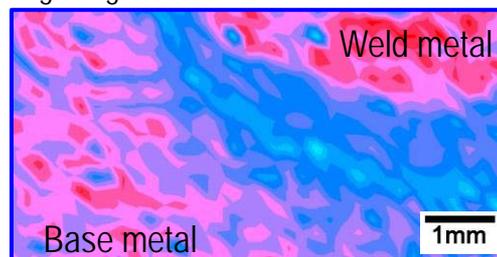
Potential Improvement of Creep Properties



Aus-forged/aged at 900C+760C/2h+GTAW+PWHT



Aus-forged/aged at 800C+760C/2h+GTAW+PWHT

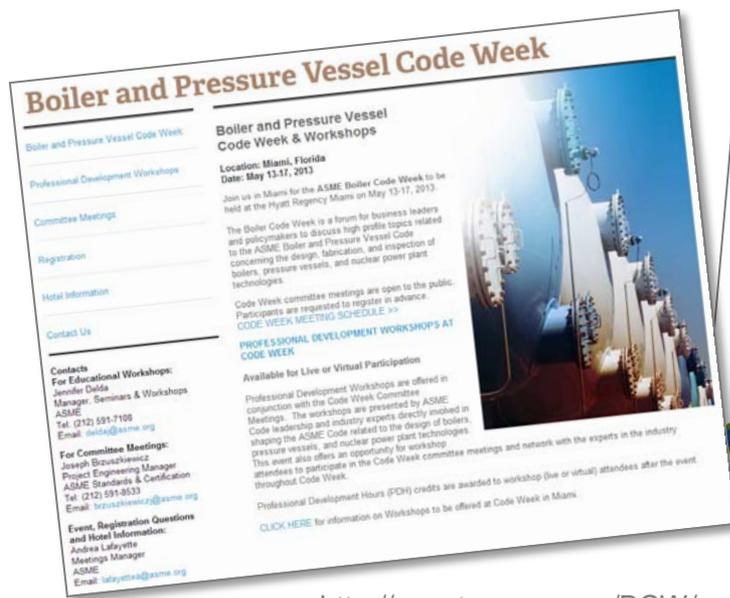


• Transition of ferrite formation between 800 and 700°C.

• Aus-forged/aged at 800°C exhibited “different” Hv distribution.

Interaction with ASME BPV Section II

- An invited presentation was made to the CSEF steels Committee associated with the ASME boiler and pressure vessel code (Section II, Chair: Jeff Henry) in Los Angeles, CA, in February.
 - Presented the recent progress on “improvement of 9Cr steel weldment creep properties via non-standard heat treatment”.
 - The results were positively received, and interactions with the Committee will be ongoing.

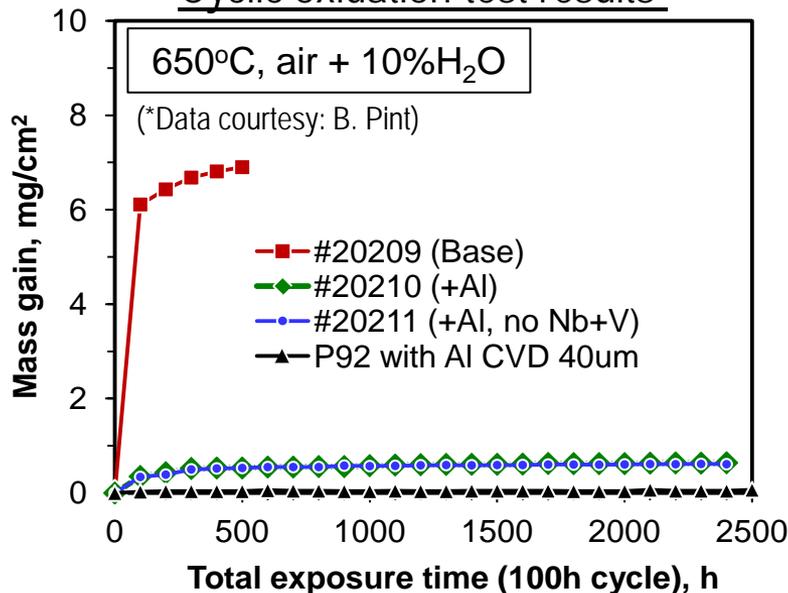


<http://events.asme.org/BCW/>

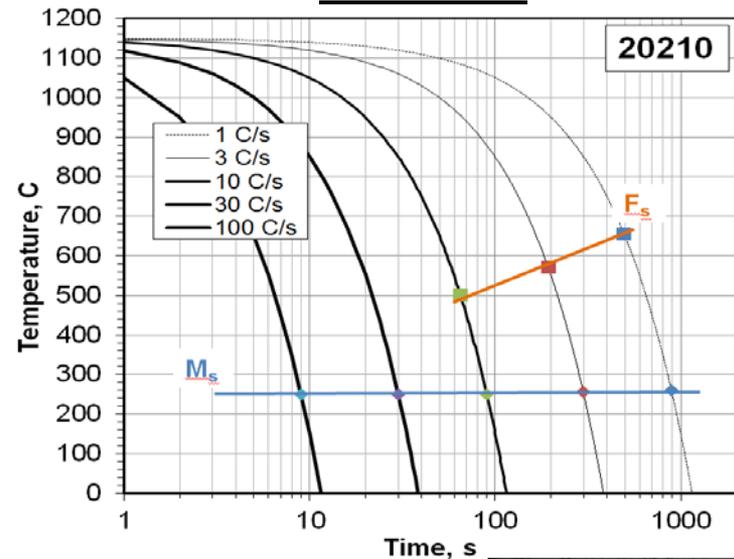
New Alloy Development: Al-containing 9Cr steels

- ❑ Focused on oxidation resistance + potential B2-NiAl (non-carbide) strengthening.
- ❑ 2.5% Al and 5.7% Ni were added (#20210 and #20211).

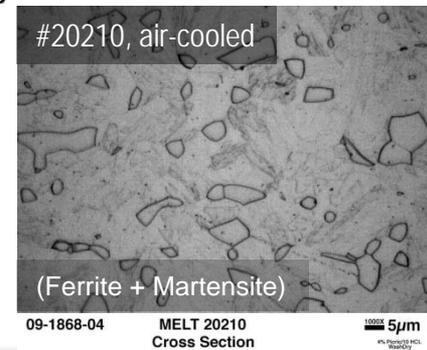
Cyclic oxidation test results



CCT curves



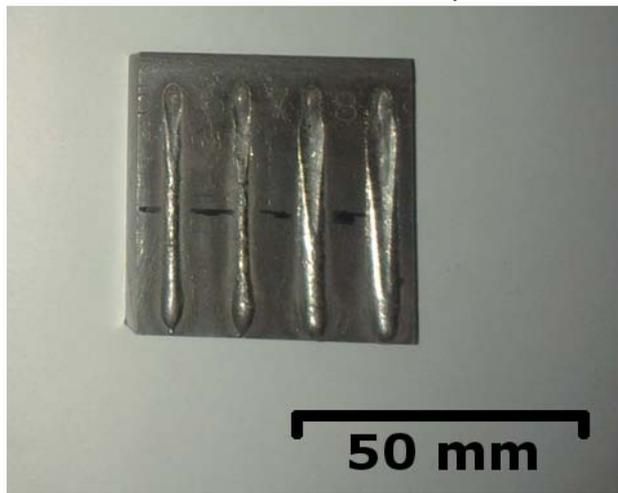
- Excellent oxidation resistance due to alumina scale formation.
- Rapid cooling is required to obtain fully martensitic microstructure.
→ Development of weld technique is also required.



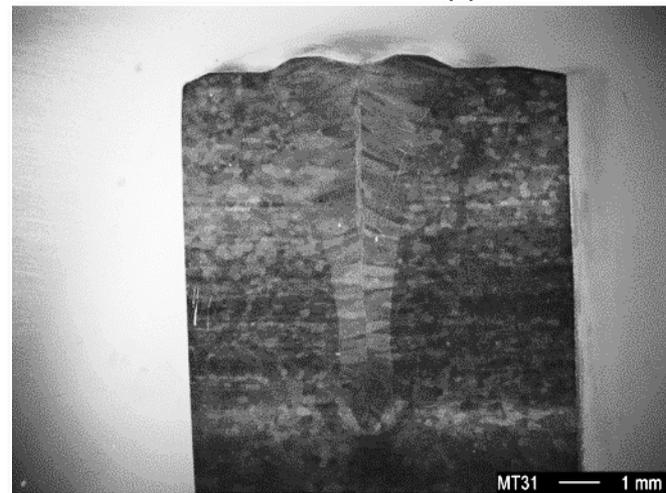
New Alloy Development: Collaboration with Jülich Research Centre, Germany

- Objective: Develop alloy compositions (parent and weld filler metals) optimized for the weldments of a new high-Cr ferritic steel, Crofer 22H®.
 - Led by Dr. Bernd Kuhn, Forschungszentrum Jülich (FZJ), Germany. ORNL supports as co-PI.
 - Identify and qualify suitable welding processes, and evaluate the properties of both the base / cross-weld specimens.
- FZJ student worked at ORNL in a collaborative effort.
 - Initiated microstructure characterization of trial e-beam weld Crofer 22H®.

E-beam welded sample



Cross-sectional view (SA, applied PWHT)



FY12 Milestones & Status

- Characterize cross-weld specimens of 9Cr steel weldments subjected to non-standard heat treatments
 - Status: Met (reported previously)
- Evaluate creep-test results of synchrotron diffraction specimens
 - Status: Partially met (three creep-rupture tests are still in progress)
- Produce a publication on initial results of microstructure characterization of creep specimens from modified heat treatment study (in collaboration with OSU)
 - Status: Met (published one paper in Acta Materialia, 2013)
- Initiate production of experimental heats of new, advanced creep strength enhanced ferritic steels with resistance to type IV cracking
 - Status: Met (Al-containing 9Cr)

FY13 Milestones & Status

- Characterize multi-pass welded Grade 91 steel with the optimized heat-treatment (December 2012).
 - Status: Met
- Rank initial oxidation results from newly proposed, creep-resistant stable ferrite steel with closest commercial alloy (March 2013).
 - Status: Met (Al-containing 9Cr)
- Complete short-time stress-rupture tests of full-size weldment specimens (June 2013).
 - Status: Partially met (two creep tests at 600°C/100MPa are in progress.)
- Complete correlation of microstructure with creep strength for synchrotron diffraction specimens (September 2013).
 - Planned September 30, 2013 (it would be delayed if the samples were not broken.)

Summary / Future Works

Optimization of HT:

- Non-standard pre-weld heat treated 9Cr steel weldments:
 - *Advantage of creep properties needs to be balanced with DBTT*
- New TMT approaches:
 - *Initiated Two-step tempering/ Aus-forging & aus-aging evaluation*
 - *TST Optimization is required for further improvement of properties.*
 - *Creep test of Aus-forged/aged Gr91 weldments is planned.*
- Interaction with ASME BPV code (Section II):
 - *Obtained various opinions from industrial participants.*
 - *Continuous interaction with the committee will be maintained.*

New Alloy Development :

- Potential development of Al-containing 9Cr steels
 - *Seek further alloy composition ranges to optimize the properties.*
- Weldment development of a new high-Cr ferritic steel, under a collaboration with Jülich Research Centre, Germany.
 - *Maintain the partnership to extend the research area.*

Acknowledgements

Sponsor:

- Department of Energy, Office of Fossil Energy

Major contributors and supporters:

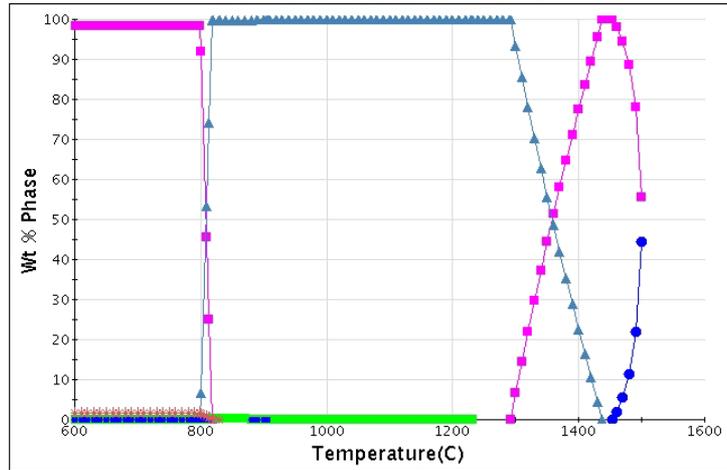
- ORNL: **Xinghua Yu, Pete Tortorelli, Mike Santella (retired), Bruce Pint, Zhili Feng, Stan David, Ying Yang, Allan Frederick, Jeff McNabb, Jeremy Moser**
- OSU: **Suresh Babu, Ben Shassere**
- Forschungszentrum Jülich GmbH (Jülich Research Centre), Germany: **Bernd Kuhn, Michal Talik**
- EPRI: **John Shingledecker**
- NIMS, Japan: **Fujio Abe**
- ASME BVP, Section II Chair: **Jeff Henry**
- NETL: **Vito Cedro III, Pat Rawls**

Phase equilibrium of Al-containing 9Cr steels

(Calculated by JMatPro with Stainless steel database)

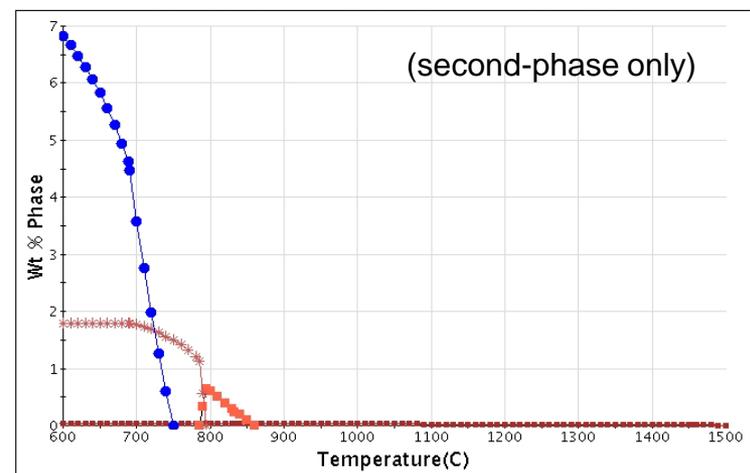
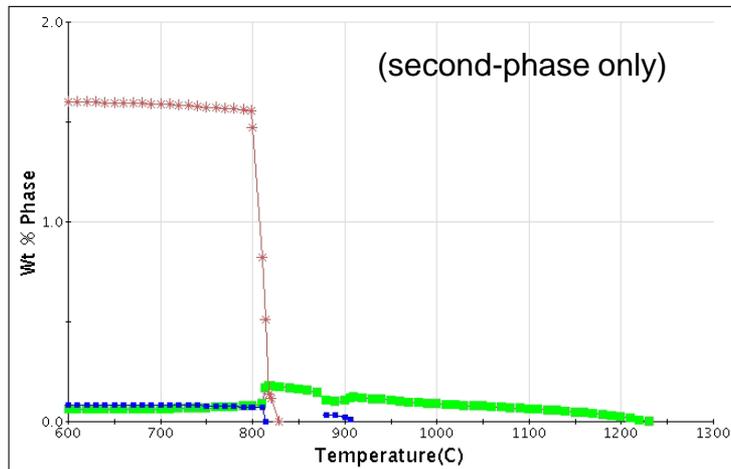
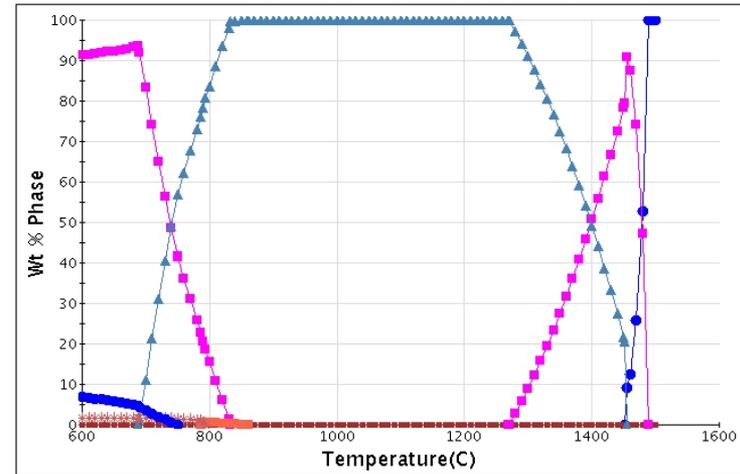
Base (#20209)

Fe-0.1C-9.0Cr-0.3Mn-0.01N-0.07Nb-0.2Ni-0.2Si-0.2V wt(%)



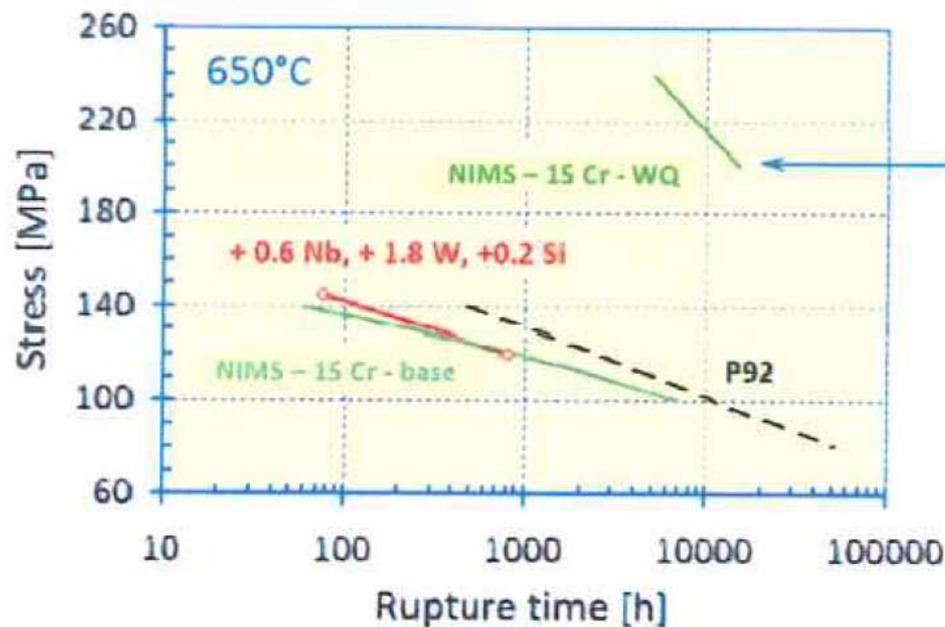
Al, no V and Nb (#20211)

Fe-2.5Al-0.1C-9.0Cr-0.3Mn-0.01N-5.7Ni-0.2Si wt(%)



Creep Properties of Crofer 22H[®] at 650C

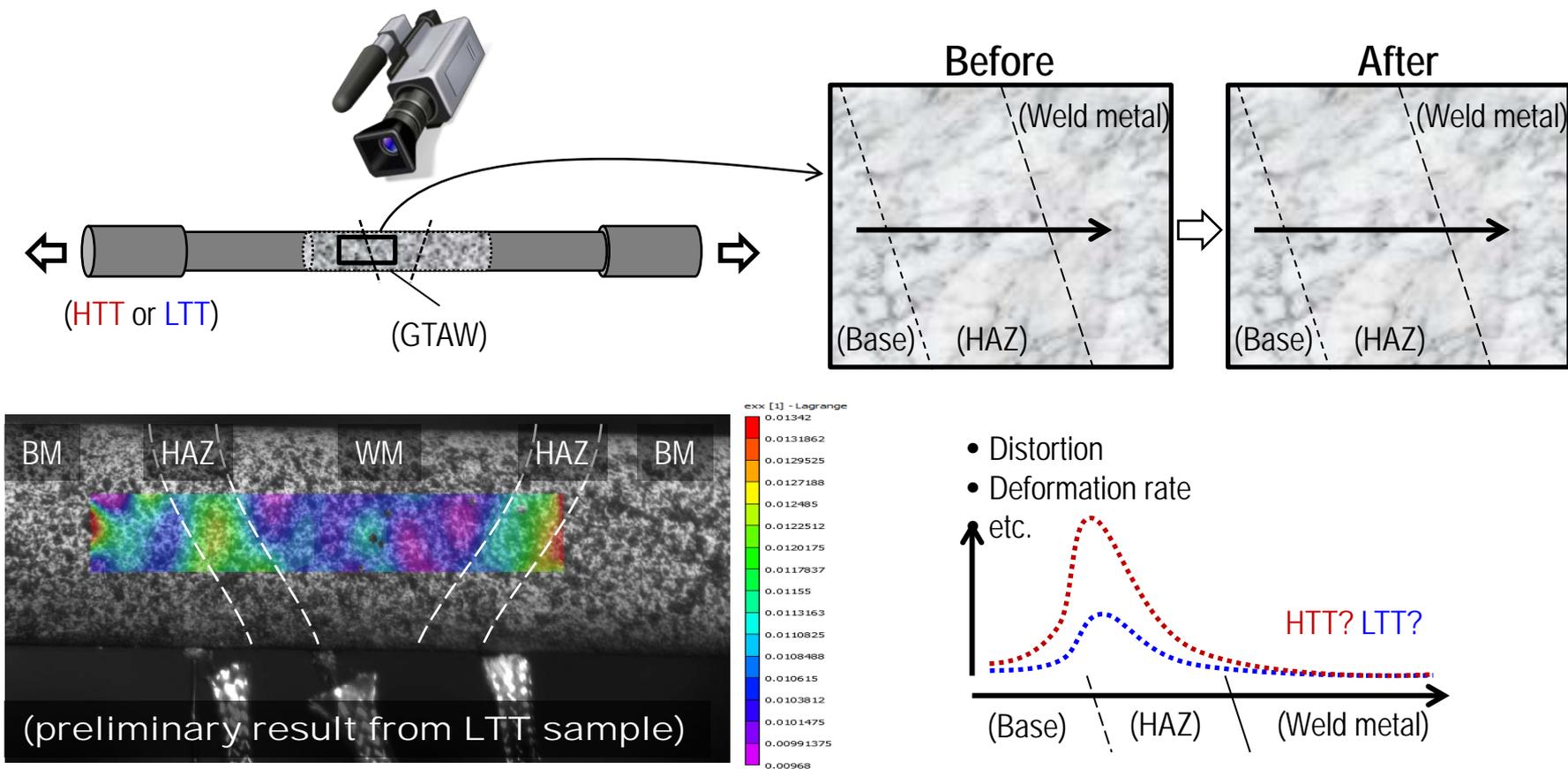
Benchmarking: Comparison to P92 and NIMS 15 Cr – steel (*)



*Future perspectives
and further work !*

In-situ Creep Deformation Analysis

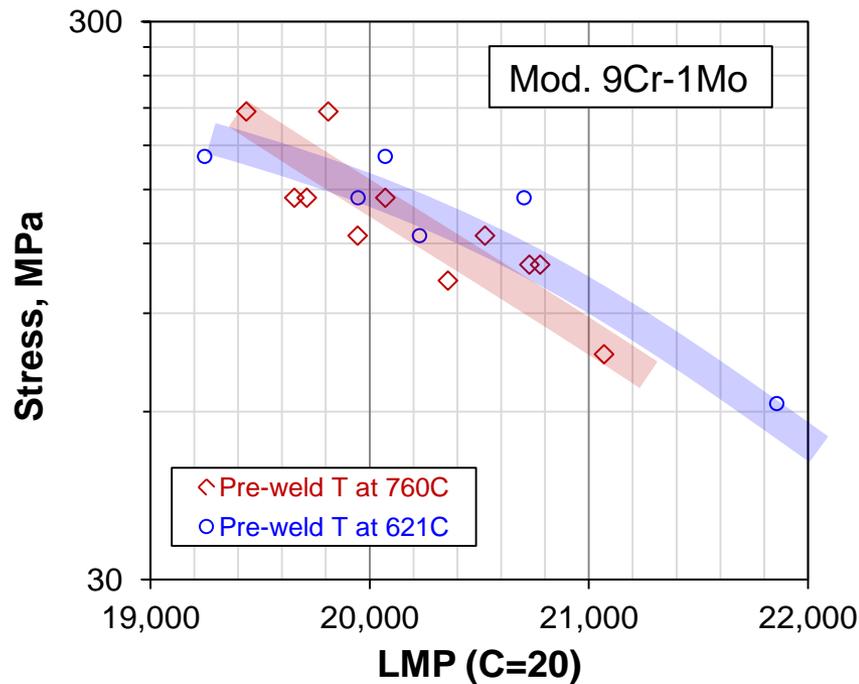
- Observation of creep deformation on mosaic painted specimens by using high-resolution video capturing (currently in progress, X. Yu at ORNL).



- Challenging work, but worth doing to have a direct evidence of partial deformation.

Initial Stress-rupture Results Suggested Improved Properties with Low Tempeing Temperature (Gr. 91)

Creep-rupture test results (1987)



- Ref: ASME SA213, SA335, SA387

	Old	New
Normalize	1040°C min.	1040-1080°C
Temper	730°C min.	730-800°C
PWHT	704°C min.	730-775°C -790°C, if $1 \leq (\text{Mn} + \text{Ni}) < 1.5$ -800°C, if $(\text{Mn} + \text{Ni}) < 1$

Figure: Gr. 91 welds, creep-rupture tested at 593°C,
ORNL TM-10504 report (1987)