# Emulating Neutron Irradiation Effect on Stainless steel by Non-Irradiation Method

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# Contents



**ACKNOWLEDGEMENT**

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# Structure of Nuclear Power Plant

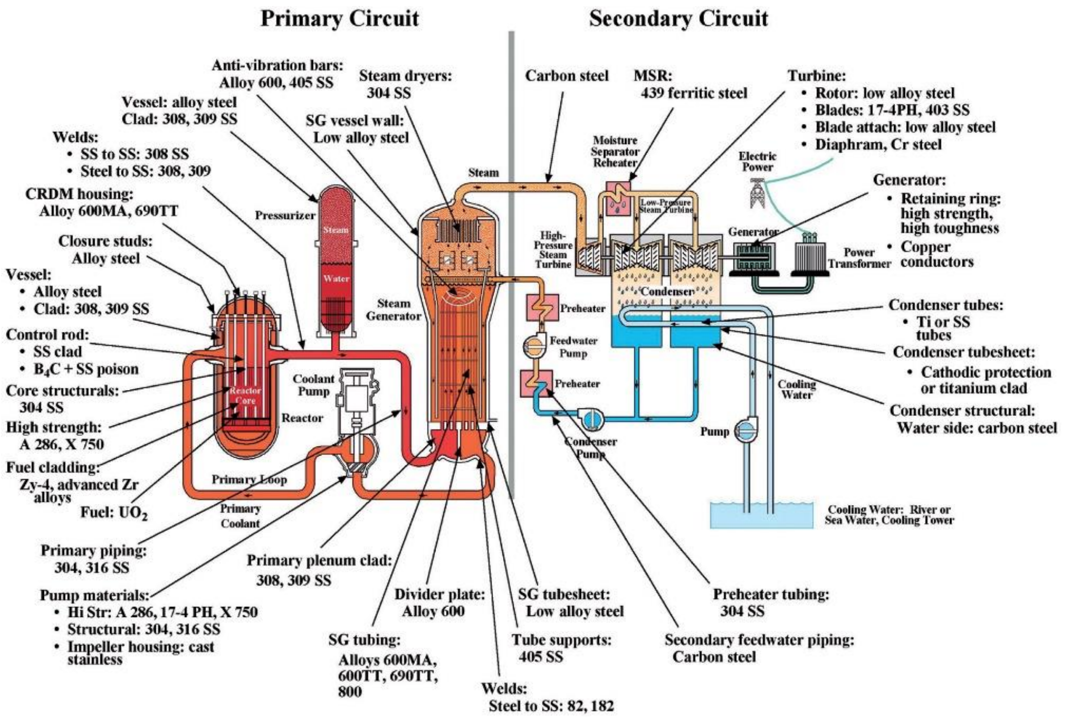



Fig. PWR components and materials [1]

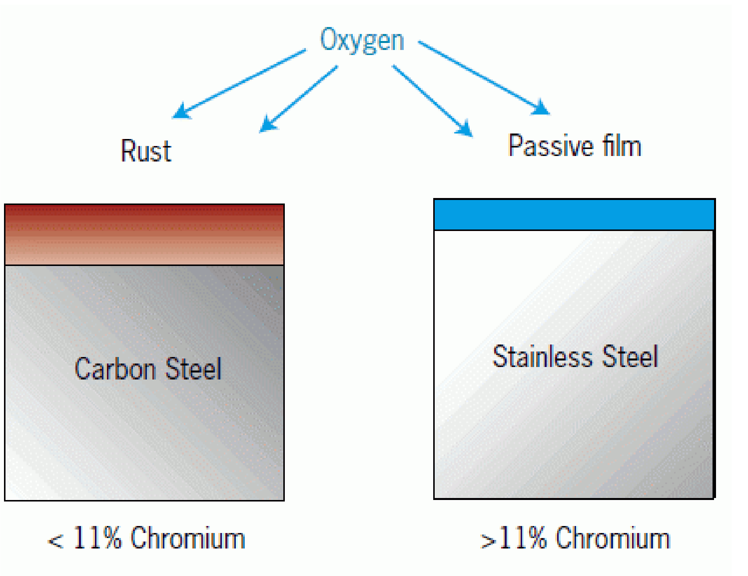



Fig. Difference between ordinary steel and stainless steel

- Austenitic stainless steel, such as 304 and 316, demonstrates superior mechanical properties and corrosion resistance in nuclear reactor environments.

- Compared to conventional steel, stainless steel contains a higher chromium content, which enhances corrosion resistance through the formation of a passive layer.

1. Todd Allen et al., Materials today (2010)

# Effects of Neutron Irradiation on Metals

- Radiation-induced segregation (RIS) modifies the micro composition in regions with high diffusion coefficients, such as grain boundaries.

- Defects arise within the material, resulting in its hardening due to these imperfections.

- This process diverges from conventional thermodynamic material alterations and tendencies, warranting additional research.

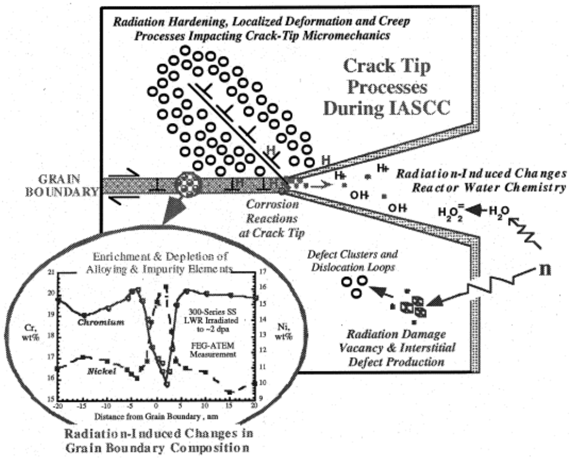



Fig. Radiation induced segregation [1]

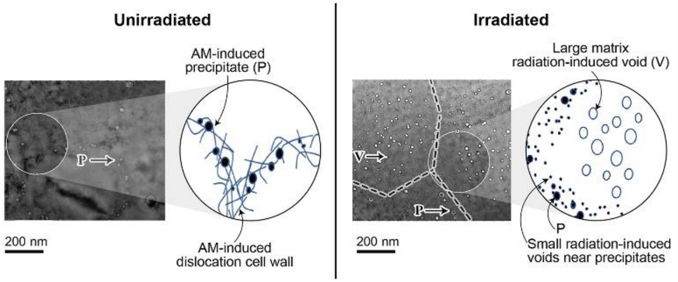



Fig. Defect [2]

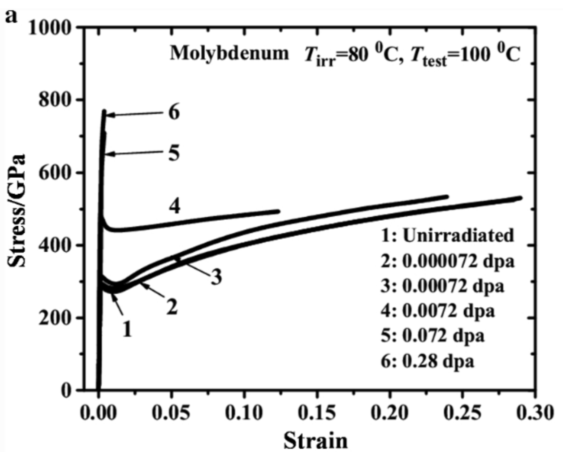



Fig. Irradiation hardening [3]

*1. S.M Bruemmer et al., Journal of Nuclear Materials (1999)*
*2. G. Meric de Bellefon et al., Journal of Nuclear Materials (2019)*
*3. Xiazi Xiao et al., Acta Mechanica Sinica (2020)*

# Importance of IASCC

- **Irradiation assistants stress corrosion cracking (IASCC)** describes a phenomenon where the interplay of environment, stress, and material, akin to conventional SCC, is hastened by radiation. This results in alterations in **corrosion resistance and mechanical properties, worsening SCC**.

- To assess the integrity of materials afflicted by IASCC, comprehending the **ramifications of neutron irradiation is paramount**.

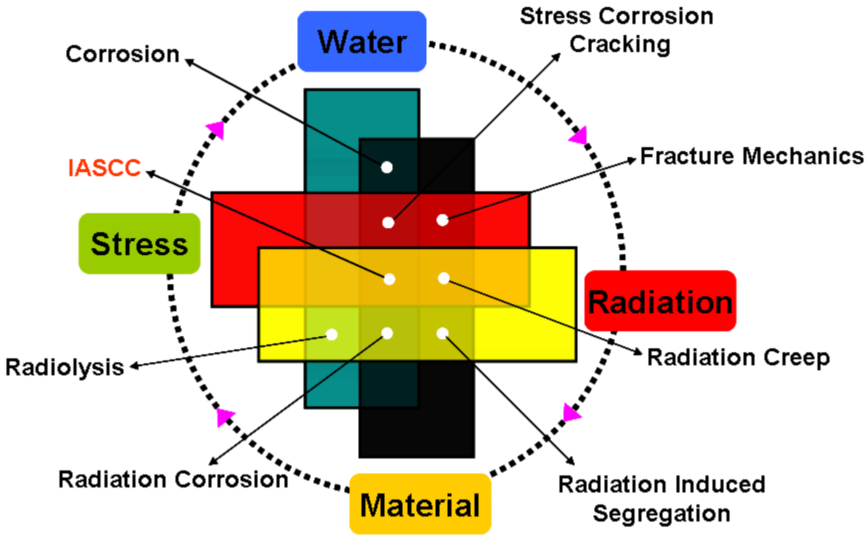



Fig. Irradiation Assisted Stress Corrosion Cracking [1]

Fig. IASCC crack observed in Baffle bolt [2]

1. *S. Shipilov et al., Engineering, Materials Science, Environmental Science (2019)*
2. *Jack McHale et al., U. S. Nuclear Regulatory Commission (2016)*

# The Cr Depletion caused by RIS

- RIS arises from variations in vacancy and interstitial concentrations induced by material diffusion rates.

- In stainless steel, chromium depletion hampers the development of the passive layer, rendering it prone to corrosion and serving as a critical factor to assess as a primary contributor to SCC.

- Chromium depletion initially occurs rapidly, according to a 2 dpa criterion, before decelerating and manifesting within a narrow range.

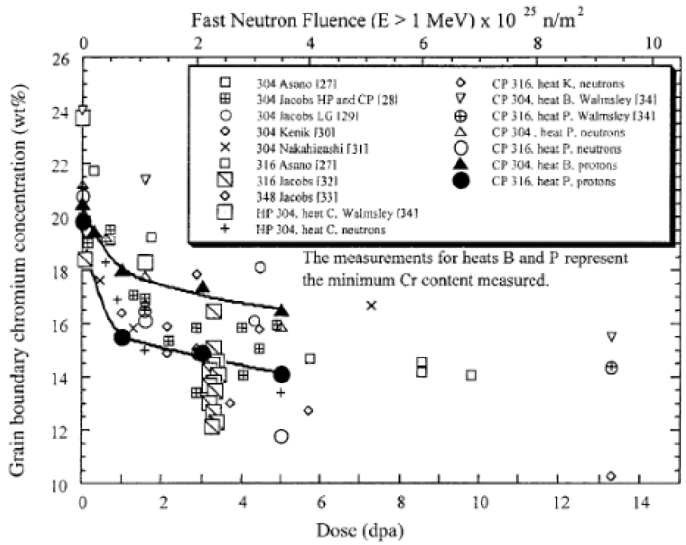



Fig. Cr composition as a function of dose for irradiated [1]

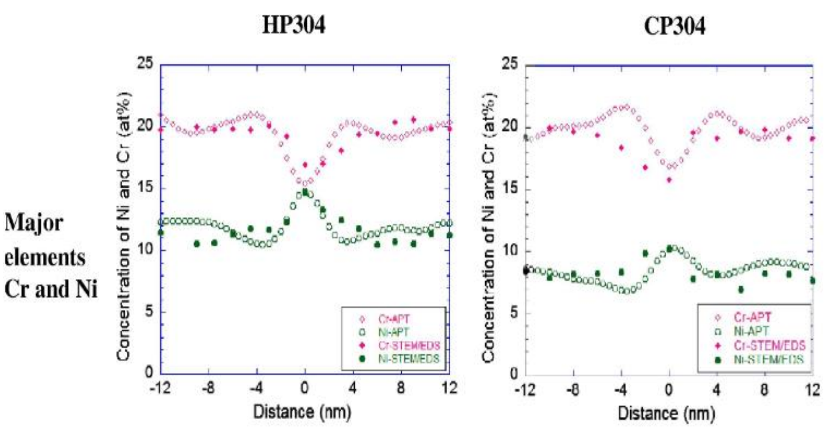



Fig. Cr composition as a function of dose for irradiated [2]

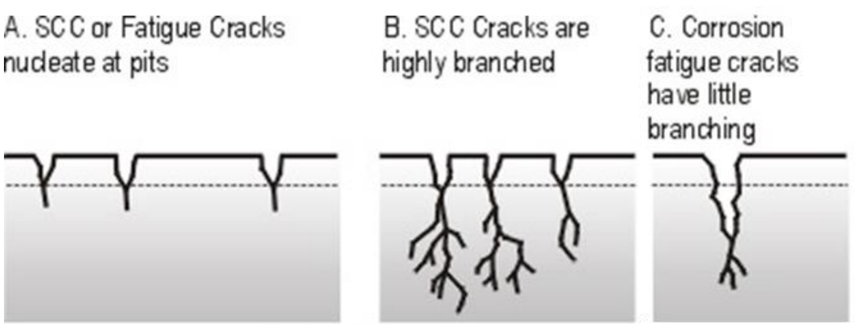



Fig. The mechanism of SCC caused by pitting formation

*1. G.S. Was, J.T. Busby et al., Journal of Nuclear Materials (2002)*
*2. Z.Jiao, G.S. Was et al., Acta Materialia (2011)*

# Mechanical Properties by Irradiation Hardening

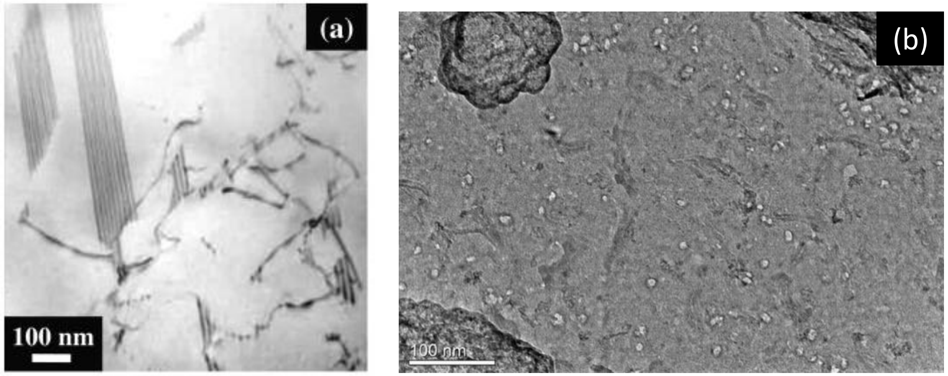



Fig. Investigation hardening due to microstructural changes
(a) dislocation loop, (b) He bubble [1], [2]

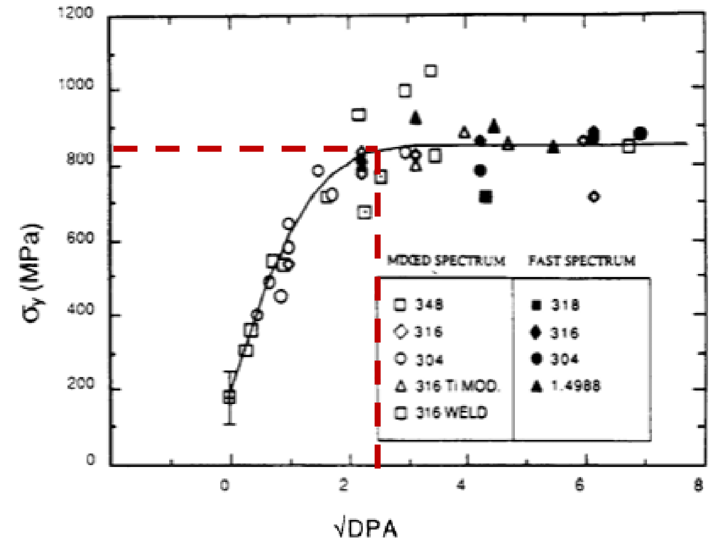



Fig. Mechanical property changes due to radiation [3]

- Neutron irradiation induces hardening and embrittlement in materials through defect formation, resulting in elevated yield strength and reduced elongation.

- This hardening effect compromises fracture toughness, heightening the material's vulnerability to stress corrosion cracking.

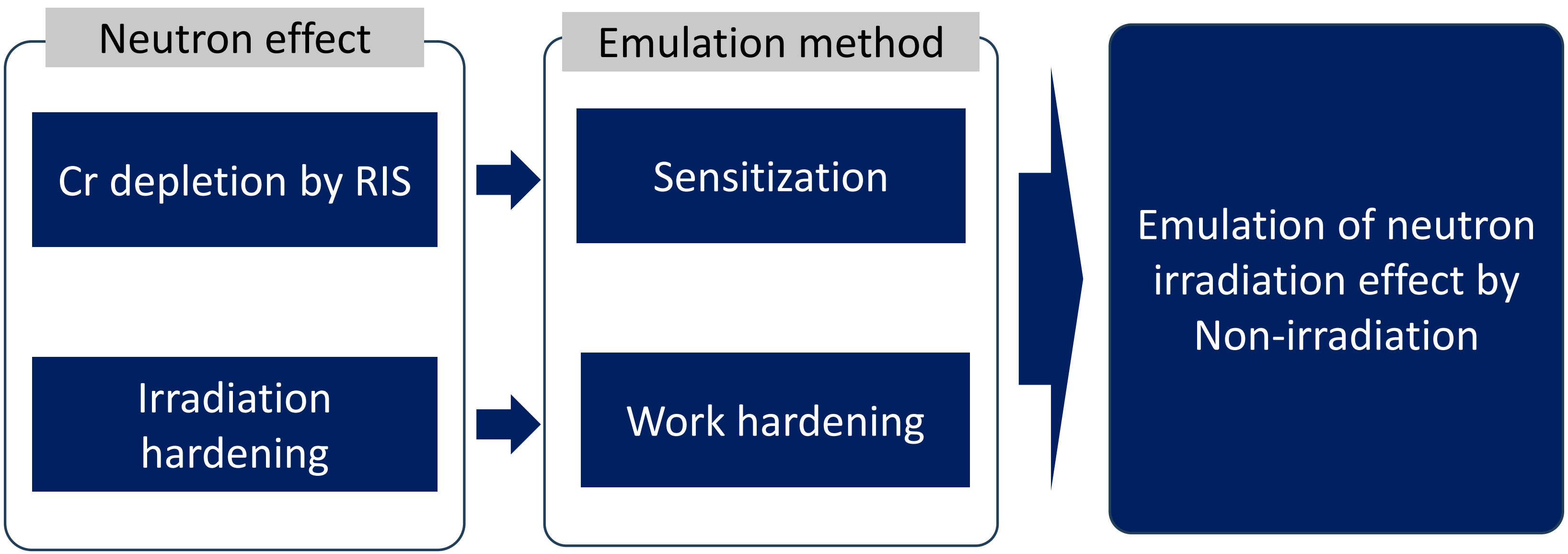
- Upon surpassing 5 dpa of irradiation, the yield strength tends to stabilize, reaching around 800 MPa.


*1. A. Etienne et al., Journal of Nuclear Materials (2010)*
*2. Sangjoon Ahn et al., Journal of Alloys and Compounds (2016)*
*3. G. R. Odette et al., Journal of Nuclear Materials (1991)*

# Objective

- Several methods for emulating neutron irradiation effects are under investigation, and I aim to propose a methodology for **<u>emulating neutron irradiation without actual irradiation</u>**.

- To emulate Cr depletion, sensitization heat treatment will be employed, while mechanical embrittlement will be induced through work hardening via rolling. The objective is to **<u>emulate mechanical strength reaching saturation at 5 dpa</u>**.



# Prediction of Cr Depletion through Prior Research

Prior research → Cr depletion tendency

- Sensitization time
- Temperature
- Chemical composition

→ Amount of $M_{23}C_6$ formation using JmatPro

→ Selection of sensitization conditions for Emulation of RIS effect

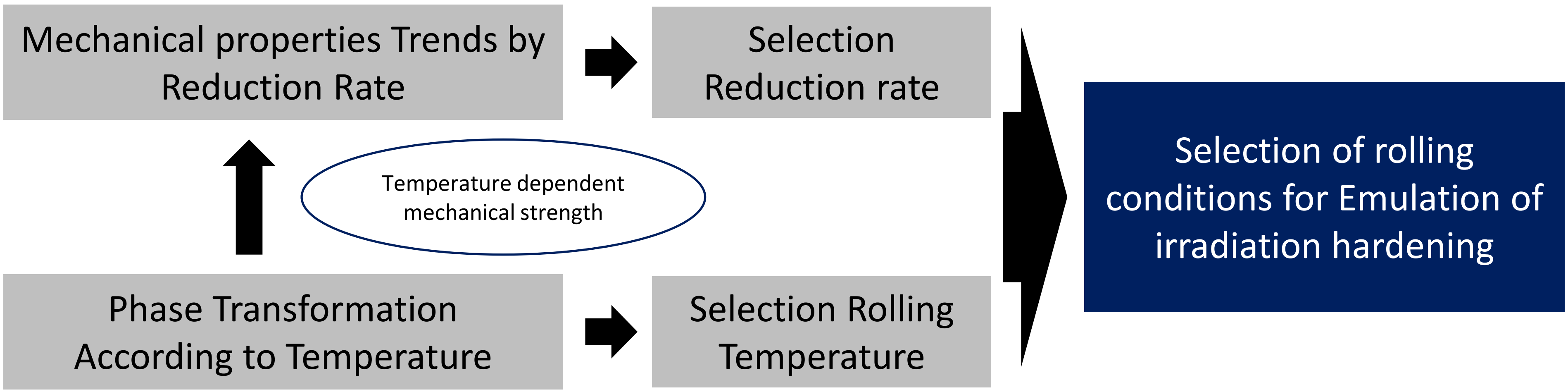
- Analyze the trend of Cr depletion and establish sensitization temperatures by referencing phase diagrams.

- Validate the pattern of Cr depletion across different sensitization durations through existing literature to replicate Cr depletion accurately.

- Furthermore, utilize the thermodynamic computational simulation program JmatPro to confirm the formation of chromium carbides in SS 304 based on previous research.

- Compare the outcomes from both approaches to forecast the required heat treatment duration.

# Prediction of Rolling Condition through Prior Research




- Analyzing hardenability trends based on rolling rate via literature review and adjusting hardenability and rolling rate accordingly.
- Confirming the amount of martensite phase formation at various temperatures during rolling.
- Investigating hardenability trends concerning temperature.
- Choosing the appropriate rolling temperature and rate.

# Specimen Processing and Analysis Method

- **Material – SS304H**

Table. Chemical composition of SS 304H [wt.%]

| Fe | C | Si | Mn | P | Cr | Ni | Cu | Co |
|---|---|---|---|---|---|---|---|---|
| Bal. | 0.0542 | 0.0429 | 1.069 | 0.0158 | 18.25 | 8.055 | 0.027 | 0.022 |

- **Material processing method**
  - Heat treatment: Cr depletion
  - Warm rolling: Irradiation hardening

- **Assessment**
  - Cr depletion: Microstructure analysis using SEM, STEM and EDS.
  - Irradiation hardening: Tensile tests (strain rate: **0.015 mm/mm/min**.)

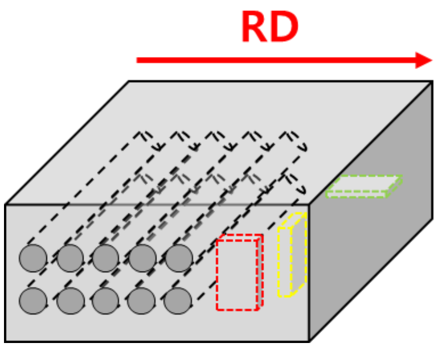



Fig. Rolling direction and specimen direction

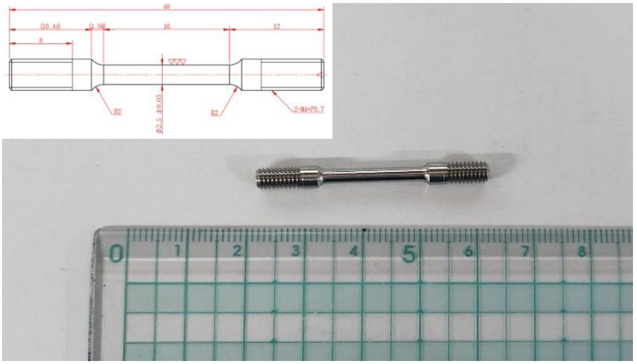


Fig. Tensile test specimen

Fig. Tensile test machine

# Trend of Sensitization and Selection temperature

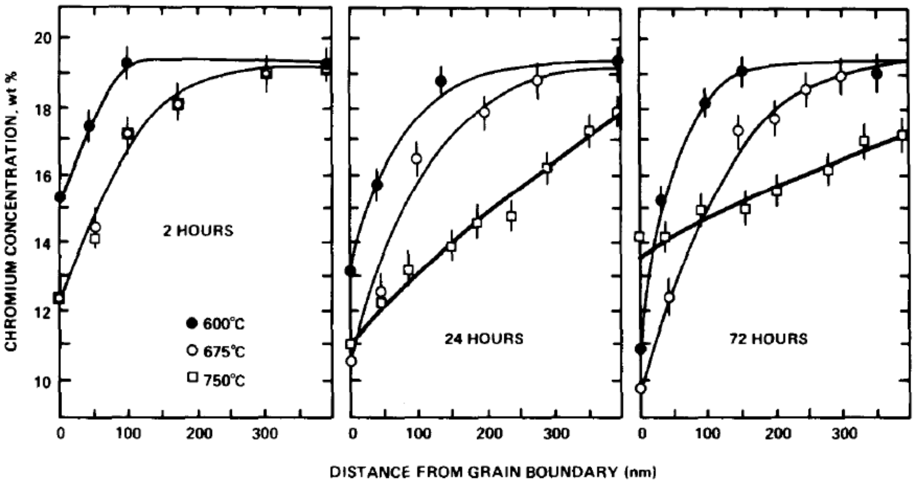



Fig. Heat treatment tendencies of SS304 [1]

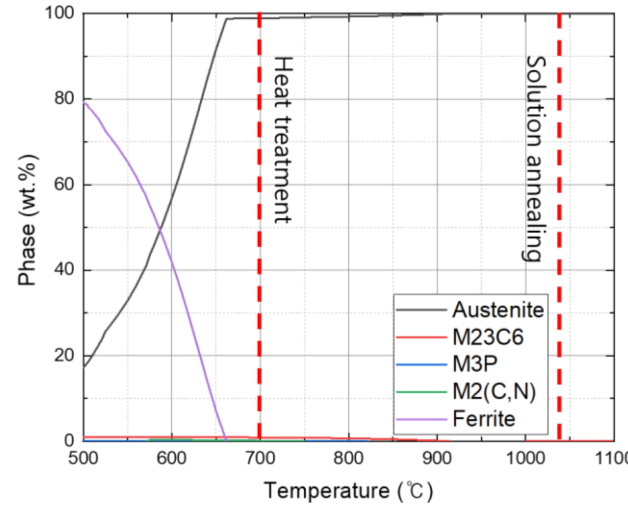



Fig. Phase diagram of SS304H [2]

- During heat treatment, Cr depletion initially experiences a substantial decrease, succeeded by an expansion in the depletion width and a reduction in the rate of depletion.

- Moreover, the extent of Cr depletion and the width of depletion are impacted by the heat treatment temperature, with a higher **sensitivity observed in the depletion width**.

- Given the **narrow Cr depletion width** observed in actual neutron irradiation, it's imperative to minimize the temperature.

- Austenitization is carried out at 1,040°C to **avert undesirable ferrite phase formation and diminish the Cr depletion width**, followed by sensitization at 700°C.

*1. E. P. Butler et al., Acta Metallurgica (1985)*
*2. JmatPro v11.2*

# Selection of Sensitization Time

- Formation of $M_{23}C_6$ ranges from a **minimum of 1 hour to a maximum of 0.52 at.%**.

- To achieve the formation of 0.52 at.% Cr carbide, approximately 12 hours of heat treatment are necessary. Accounting for other variables, this duration can be extended up to a **maximum of 24 hours**.

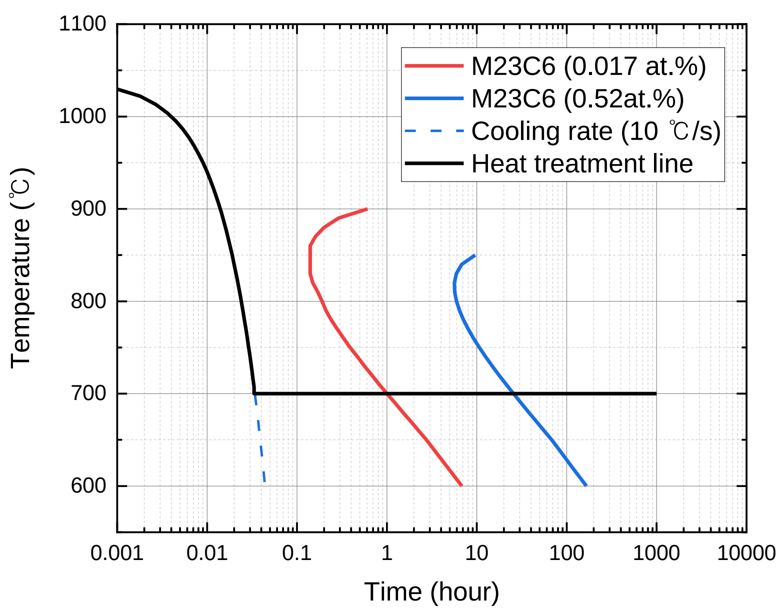



Fig. TTT diagram for $M_{23}C_6$ of SS 304H [5]

Table. In prior research papers, the trend of Cr changes in SS304 was compared with the calculation results of $M_{23}C_6$ using JmatPro [1], [2], [3], [4], [5].

| Author | Temp. (℃) | Time (h) | $M_{23}C_6$ (at.%) [5] | △Cr (wt.%) |
|---|---|---|---|---|
| Bruemmer [2] | 600 | 9 | 0.0298 | 5.38 |
| Bruemmer | 600 | 57 | 0.2266 | 6.58 |
| Bruemmer | 600 | 100 | 0.3986 | 7.08 |
| Bruemmer | 625 | 25 | 0.17 | 7.28 |
| Butler [1] | 600 | 2 | 0.00645 | 3.8 |
| Butler | 600 | 2 | 0.00645 | 1.9 |
| Butler | 675 | 2 | 0.0401 | 6.5 |
| Butler | 675 | 2 | 0.0401 | 6 |
| Butler | 675 | 2 | 0.0401 | 5.5 |
| Butler | 675 | 2 | 0.0401 | 4.9 |
| Bruemmer | 700 | 1 | 0.0226 | 5.98 |
| Bruemmer | 700 | 10 | 0.3 | 7.08 |
| Bruemmer | 700 | 100 | 1.25 | 6.58 |
| Ravindra [3] | 700 | 2 | 0.0436 | 6.011 |
| Ravindra | 700 | 24 | 0.55 | 8.711 |
| Ravindra | 700 | 96 | 0.89 | 8.011 |
| Trillo [4] | 775 | 10 | 0.52 | 4.79 |
| Bruemmer | 800 | 10 | 0.85 | 3.88 |

Table. Test matrix in this paper

| | |
|---|---|
| **Annealing Temperature [℃]** | 1040 |
| **Annealing time** | 2 hour |
| **Sensitization Temperature [℃]** | 700 |
| **Sensitization Time [hour]** | 1, 2, 3, 5, 7, 15, 24 |
| **Cooling** | Water quenched |


*1. E. P. Butler et al., Acta Metallurgica (1985)*
*2. S. M. Bruemmer and L. A. Charlot et al., Scripta METALLURGICA (1986)*
*3. Ravindra V. Taiwade et al., ISIJ International Vol. 52 (2012)*
*4. E.A. Trillo, R. Beltran et al. Materials Characterization (1995)*
*5. JmatPro v11.2*

# Carbide Distribution due to Heat Treatment

*A = Cr carbide area, L = Grain boundary length*

- Verifying the **area of Cr carbides in relation to the length of grain boundaries** over different heat treatment durations using SEM.

- It's important to note that Cr depletion cannot be confirmed via SEM. **Quantitative evaluation of Cr depletion requires STEM.**

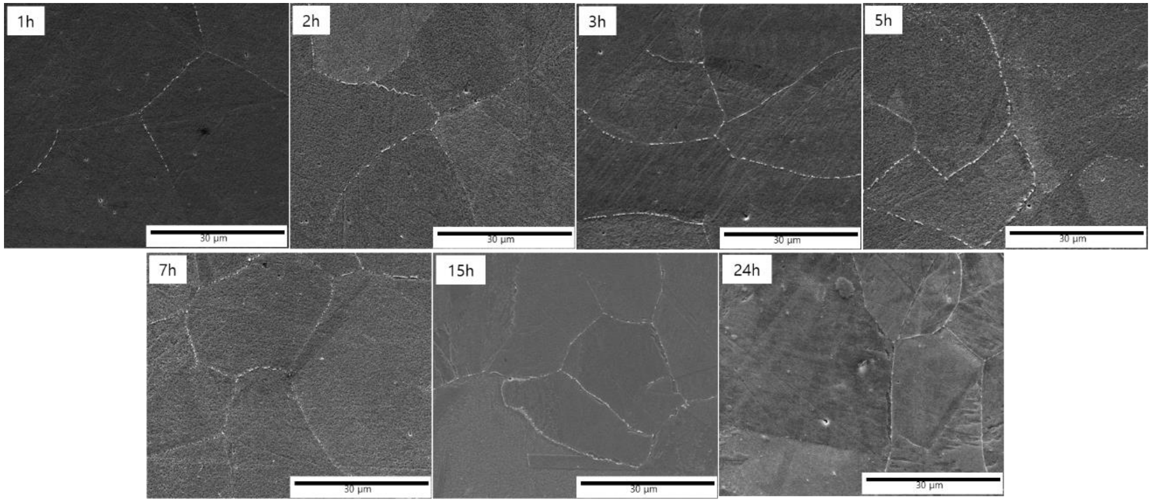



Fig. SEM images of SS304H by heat treatment durations

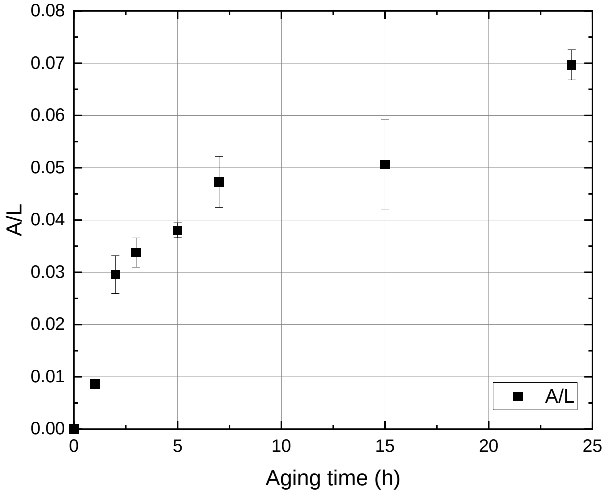



Fig. A/L value of SS304H by heat treatment durations

Table. In Prior research paper, trend of Cr depletion

| Aging time (h) | 1 | 2 | 3 | 5 | 7 | 15 | 24 |
|---|---|---|---|---|---|---|---|
| Area/length(A/L) | 0.0087 | 0.0296 | 0.0338 | 0.0380 | 0.0473 | 0.0506 | 0.0697 |

# Cr Carbide Analysis using STEM

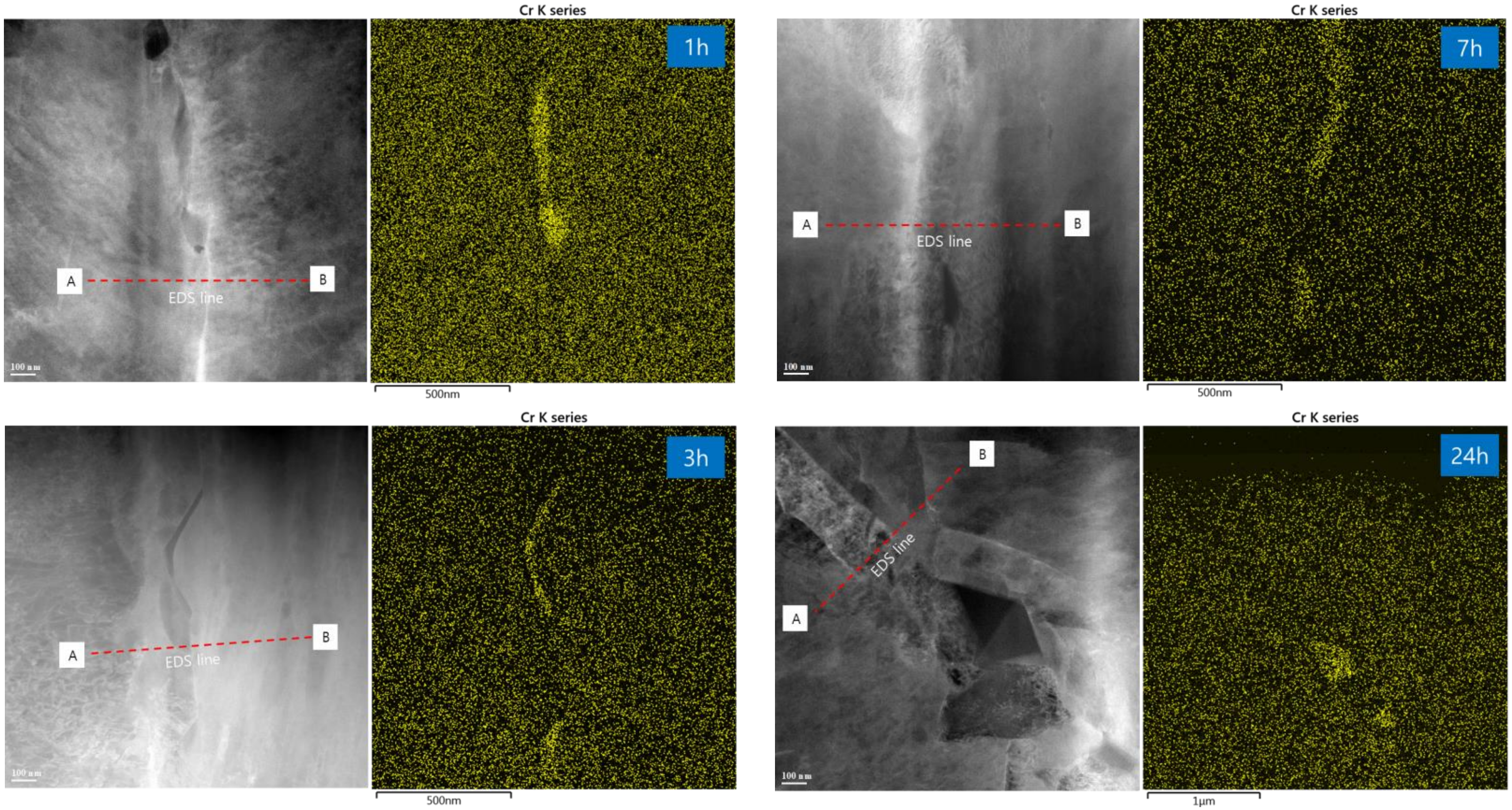



Fig. STEM image with EDS line and EDS map of Cr

- Conducting measurements of the EDS line along the grain boundary, maintaining a consistent distance (100nm to 150nm) from the Cr carbide.

# Comparison of Cr Depletion Trends between Heat Treatment and RIS

- The variation of Cr experiences a sharp change initially with heat treatment time.
- After 7 hours, the variation of Cr stabilizes, while the **depletion width undergoes significant changes after 3 hours**.
- Comparison with actual 5 dpa neutron-irradiated materials indicates that the optimal **heat treatment duration is approximately 3 hours**.

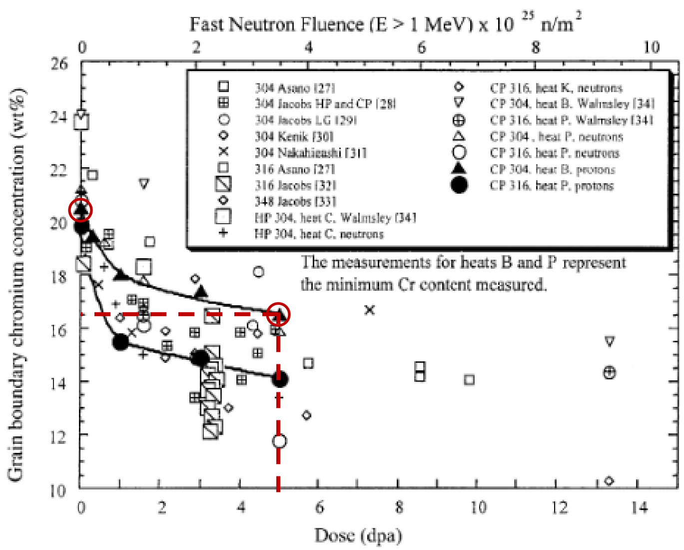



Fig. Cr depletion trend due to RIS [1]

Table. Trend of Cr concentration sensitization time

| Heat treatment time (Hour) | Min. of Cr (wt.%) | Depletion width (nm) |
|---|---|---|
| 1 | 14.76 | 150 |
| **3** | **12.2** | 180 |
| 7 | 11.42 | 250 |
| 24 | 11.14 | 300 |

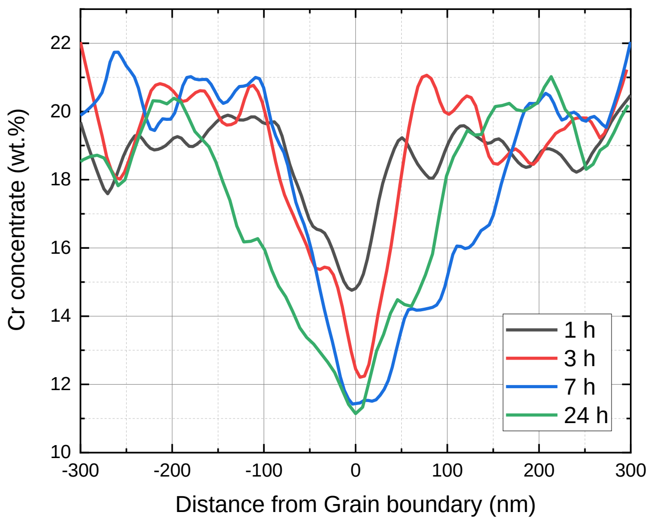



Fig. Trend of Cr concentration by heat treatment time

1. G.S. Was, J.T. Busby et al., Journal of Nuclear Materials (2002)

# Methodology for Selecting Rolling Temperature

- Warm rolling is performed at **high temperatures to mitigate undesired Martensite phase formation**.
- However, during warm rolling, the increment in the original yield strength tends to be diminished, necessitating careful consideration.
- Our objective is to enhance the yield strength by conducting **rolling at 200℃**. To account for temperature effects, we plan to execute approximately **10% more rolling compared to the existing reduction rate**.

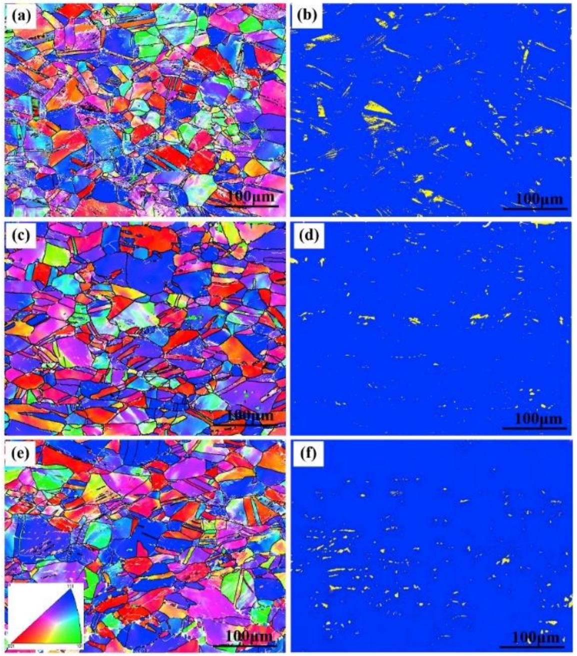



Table. Yield strength by hot rolling temperature (RD, reduction rate = 40%, SS316) [2]

| Temperature (℃) | $\sigma_{0.2}$ (MPa) | UTS (MPa) | Elongation (%) |
|---|---|---|---|
| 500 | 945 | 1080 | 10.5 |
| 700 | 875 | 960 | 11.5 |
| 900 | 720 | 820 | 20.0 |

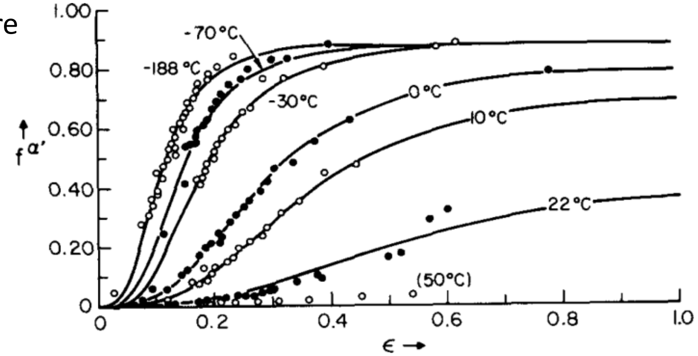



Fig. Martensite Fraction by Temperature [1]

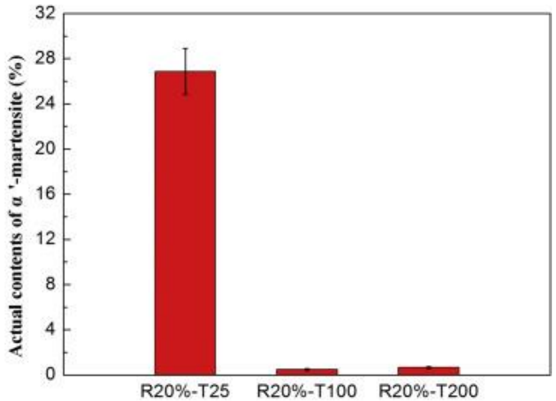



Fig. Martensite Fraction by temperature [3]

*1. G. B. Olson et al., Kinetics of Strain-Induced Martensitic Nucleation, Metallurgical Transactions A (1975)*
*2. Xingyang Chen et al., International Journal of Hydrogen Energy (2018)*
*3. Marina Odnobokova et al., Metals (2022)*

# Methodology for Selecting Rolling Rate

- We emulate strain hardening by inducing defect formation through rolling.

- We attained the desired yield strength of **<u>800 MPa with approximately 25% cold rolling</u>**.

- Considering that the hardening effect diminishes as temperature rises

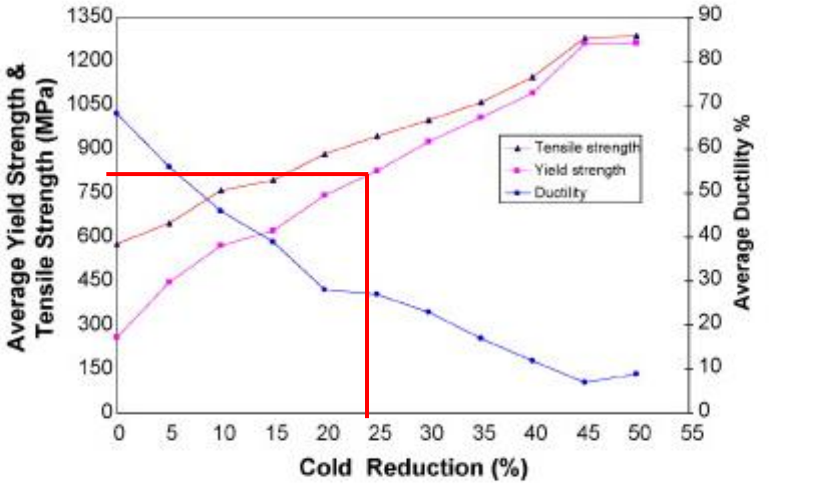



Fig. Yield strength by reduction rate (TD, Room Temperature, SS304) [1]

Table. Test matrix in this paper

| | |
|---|---|
| Temperature [℃] | 200 |
| Reduction rate [%] | 10, 20, 30 |

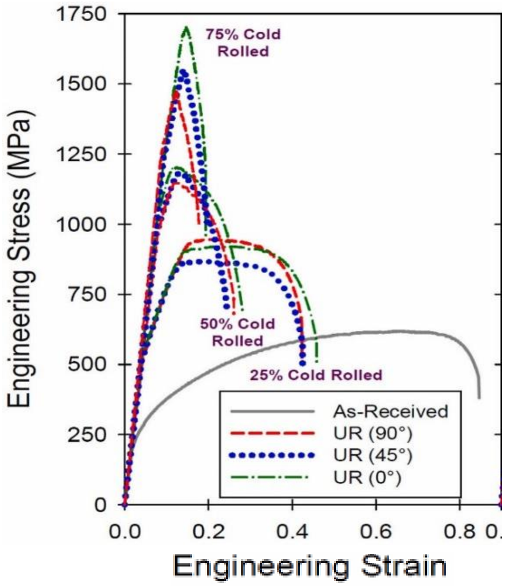



Fig. Stainless Steel 316 Tensile test graph [2]

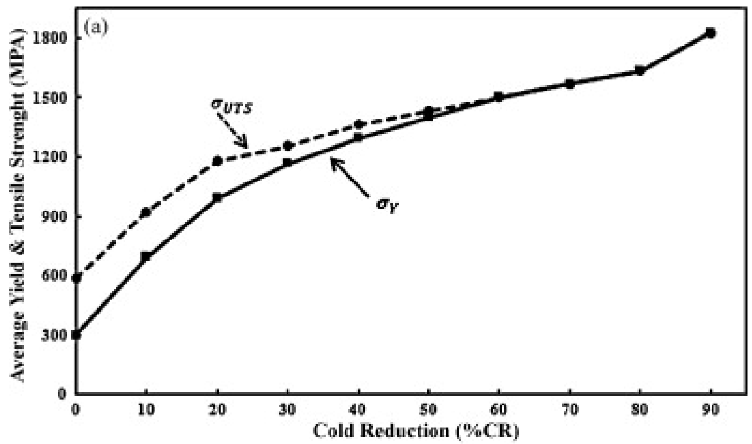



Fig. Yield strength by reduction rate (RD, 0℃, SS304) [3]

*1. M. Milad et al., Journal of Materials Processing Technology (2008)*
*2. Sara Mohammaadzehi et al., Materials Science and Engineering : A (2023)*
*3. C. K. Mukhopadhyay et al., Journal of Materials Science (1993)*

# The Tensile Test Results by Rolling

- As the reduction rate rises, the material's yield strength increases while elongation experiences a notable decrease, mirroring traits observed in neutron-irradiated materials.

- The irradiation hardening phenomenon at 5 dpa was discovered to closely resemble conditions at **approximately 800 MPa**, corresponding to around a **30% reduction rate**.

Table. Mechanical property changes by rolling

| Reduction rate | 3hr, 0% | 3hr, 10% | 3hr, 20% | 3hr, 30% |
|---|---|---|---|---|
| Yield strength (MPa) | 233 | 443 | 568 | 789 |

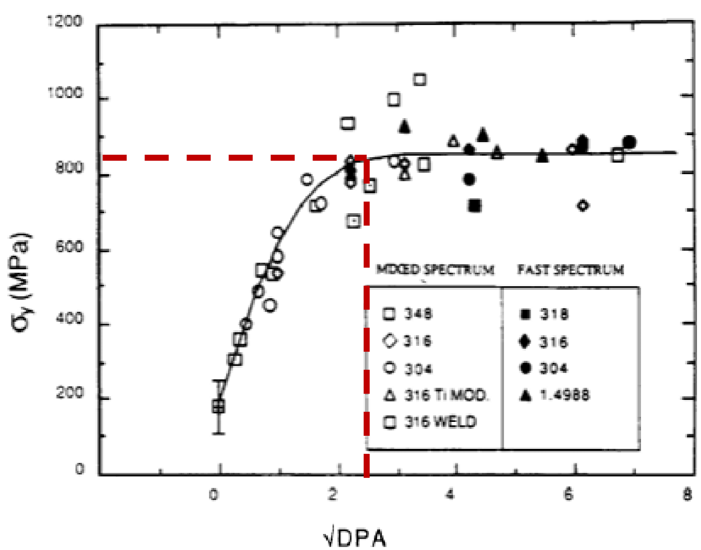



Fig. Mechanical property changes by irradiation [1]

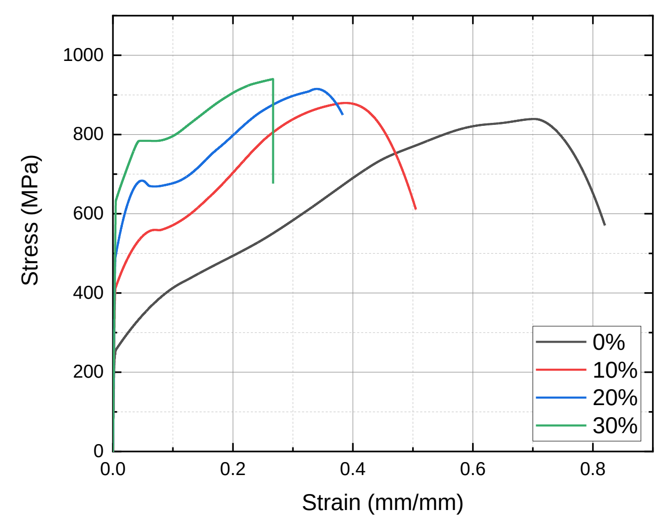



Fig. Mechanical property changes by rolling

*1. G. R. Odette et al., Journal of Nuclear Materials (1991)*

# Conclusion and Future work

## Conclusion

- Neutron irradiation of stainless steel significantly contributes to SCC. **RIS and irradiation hardening play pivotal roles**, crucial in understanding IASCC.

- However, analyzing these factors entails risks and substantial costs. Therefore, our approach focuses on **emulating them using non-irradiated methods**, aiming to achieve a mechanical strength saturation equivalent to 5 dpa. This will be accomplished through **heat treatment to mimic RIS and work hardening via rolling**.

- Utilizing thermodynamic computational simulation programs, we determined optimal heat treatment conditions, concluding that conducting heat treatment at **approximately 700°C for about 3 hours** is most suitable. Similarly, **rolling at a 30% reduction rate at 200°C** was identified as the most appropriate method.

## Future work

- Comparison of corrosion susceptibility according to microstructural changes between heat-treated and neutron-irradiated specimens

- Comparing Neutron-Irradiated Materials with SCC Susceptibility

# Thank you

## for your attention

Hyeonje Ryoo