Karlsruher Institut für Technologie

Institut für Angewandte Materialien

# Modeling of the temperature dependent deformation of ODS steels

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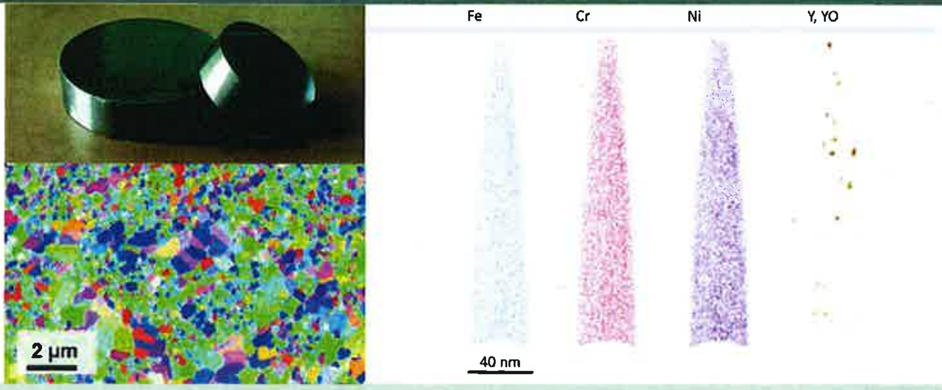
Institut für Angewandte Materialien - Werkstoffkunde



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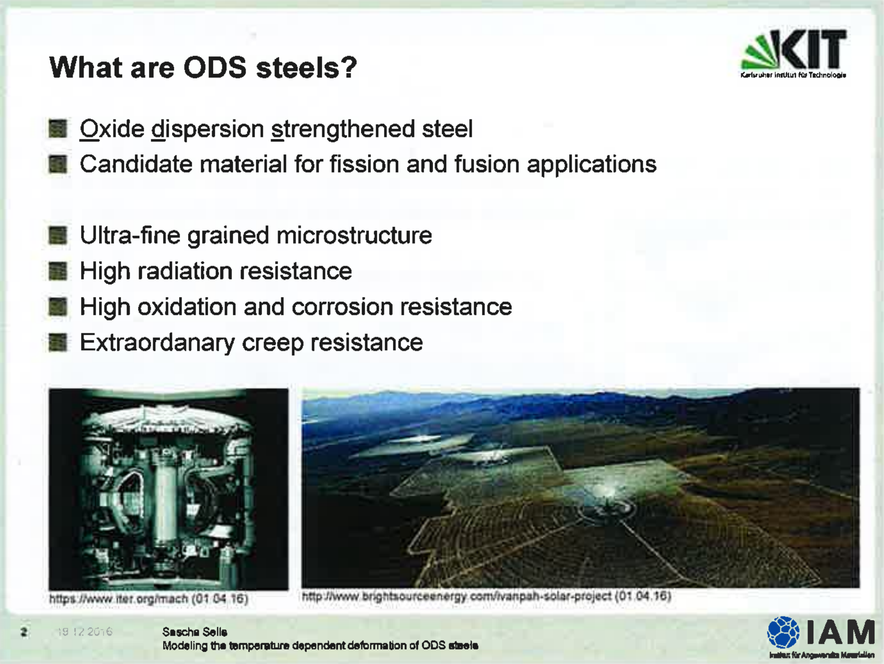

## What are ODS steels?

- Oxide dispersion strengthened steel
- Candidate material for fission and fusion applications

- Ultra-fine grained microstructure
- High radiation resistance
- High oxidation and corrosion resistance
- Extraordanary creep resistance

https://www.iter.org/mach (01.04.16)

http://www.brightsourceenergy.com/ivanpah-solar-project (01.04.16)

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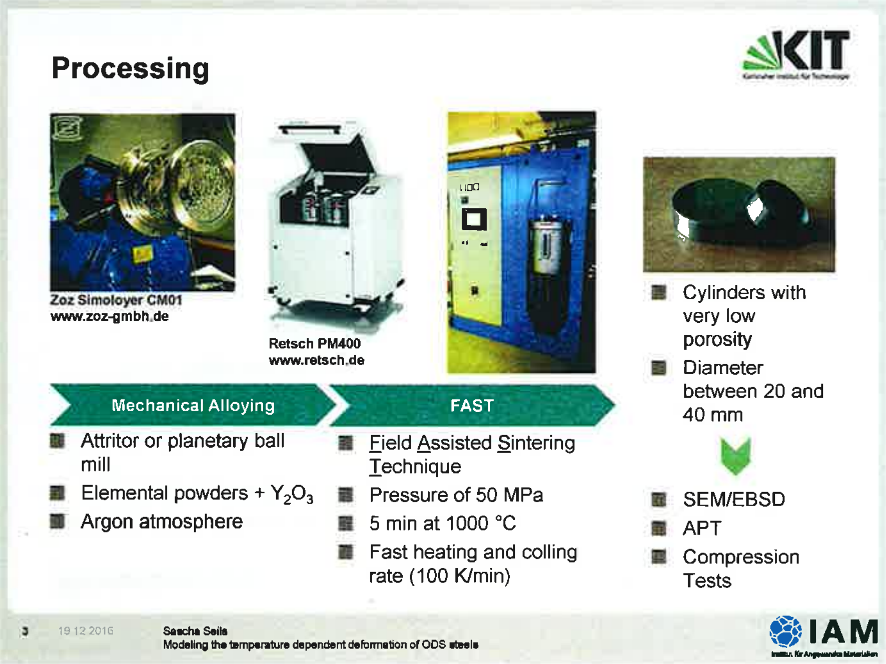


## Processing

Zoz Simoloyer CM01
www.zoz-gmbh.de

Retsch PM400
www.retsch.de

**Mechanical Alloying**

- Attritor or planetary ball mill
- Elemental powders + $Y_2O_3$
- Argon atmosphere

**FAST**

- Field Assisted Sintering Technique
- Pressure of 50 MPa
- 5 min at 1000 °C
- Fast heating and colling rate (100 K/min)

- Cylinders with very low porosity
- Diameter between 20 and 40 mm

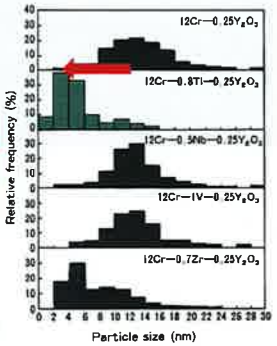
- SEM/EBSD
- APT
- Compression Tests

## Materials

- Analysis of ferritic and austenitic ODS steels
- Improved high temperature properties of austenitic (fcc) ODS steel expected

| in wt.% | Fe | Cr | Ni | Ti | $Y_2O_3$ |
|---|---|---|---|---|---|
| **FNC 14** | Bal. | **14** | | 0.4 | 0.25 |
| **ANC 25/20** | Bal. | **25** | **20** | 0.4 | 0.25 |

- Formation of oxide particles by addition of $Y_2O_3$
- Decreasing particle size by titanium additions
- Formation of Y-Ti-O-containing nanoclusters (~ 4 nm)



S. Ukai et al., J. Nucl. Mater. 307 (2002)

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## Microstructure

- Exclusion of thermally induced changes of the microstructure during compression tests

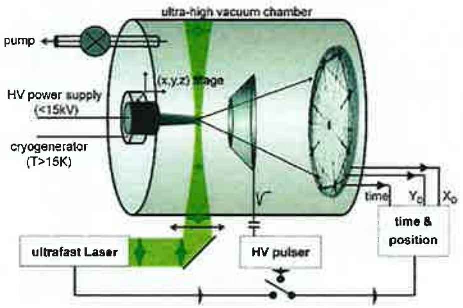
Nanoclusters ? Grain size

- Expections
  - High thermal stability of Y-Ti-O nanoclusters
  - Pinning of grain boundaries by nanoclusters
  - Low grain growth in temperature range of experiments

➢ Annealing at air between 1000 °C and 1200 °C up to 1000 h

# Analysis of nanoclusters

- Atom Probe Tomography (APT)
- Tips produced by lift out method at SEM/FIB dual beam microscope



Gault, B. et al.: Atom Probe Microscopy, Springer Series in Materials Science, Vol. 160, 2012

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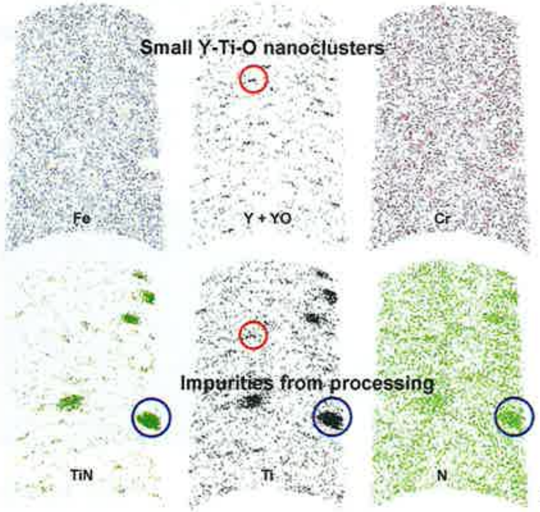
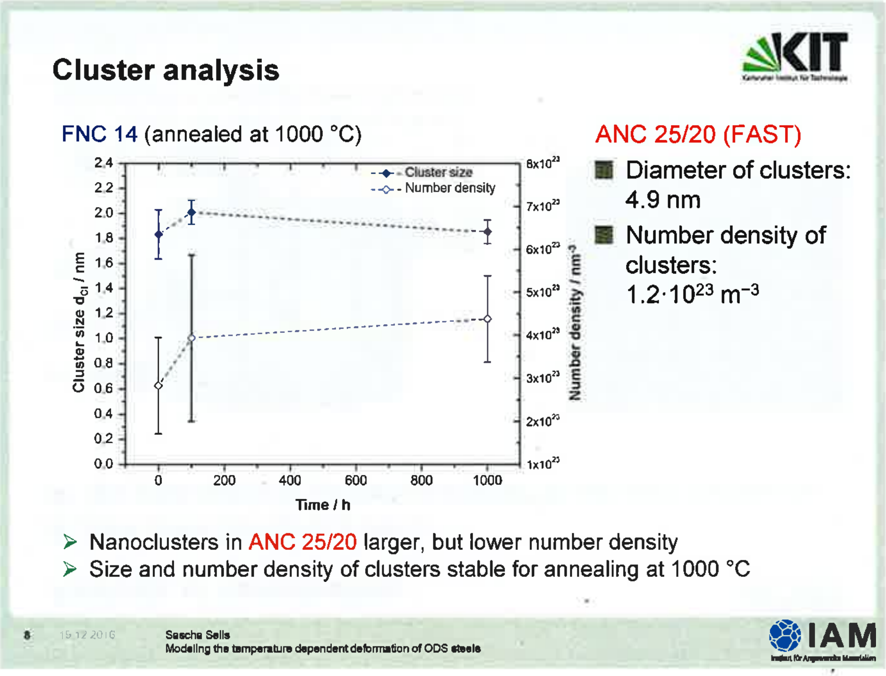
# Reconstruction of a FNC 14 tip (FAST)



- 5 nm thick, 2-dimensional longitudinal section of the reconstruction
- Homogenous distribution of iron and chromium

➢ Proof of cluster formation in FNC 14 and ANC 25/20

➢ Main elements in clusters: Y, O, Ti and Cr



## Cluster analysis

FNC 14 (annealed at 1000 °C)

ANC 25/20 (FAST)

- Diameter of clusters: 4.9 nm
- Number density of clusters: $1.2 \cdot 10^{23}$ $m^{-3}$

- Nanoclusters in ANC 25/20 larger, but lower number density
- Size and number density of clusters stable for annealing at 1000 °C

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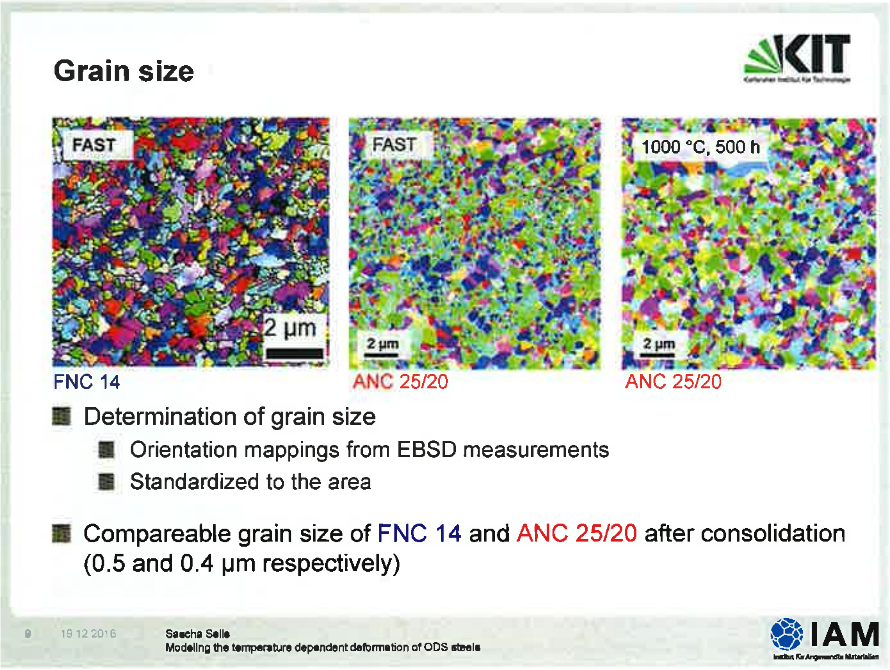
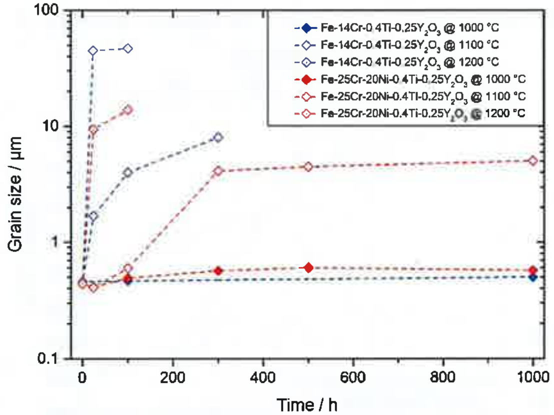


## Grain size

FNC 14 ANC 25/20 ANC 25/20

- Determination of grain size
  - Orientation mappings from EBSD measurements
  - Standardized to the area
- Compareable grain size of FNC 14 and ANC 25/20 after consolidation (0.5 and 0.4 µm respectively)

## Grain size



- 1000 °C:
  No grain growth
- 1100 °C:
  Grain growth of **FNC 14** faster compared to **ANC 25/20**
- 1200 °C:
  Fast grain growth for both steels

- Compression testes perfomed between RT and 1000 °C
- No change of microstructure in important temperature range

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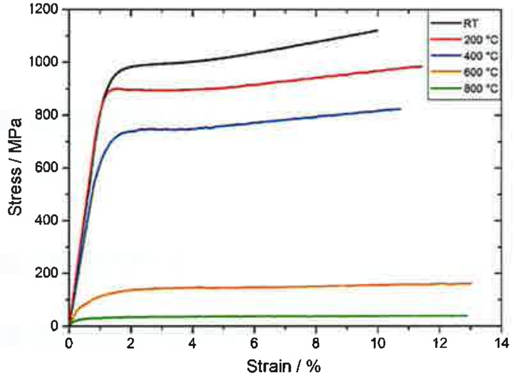
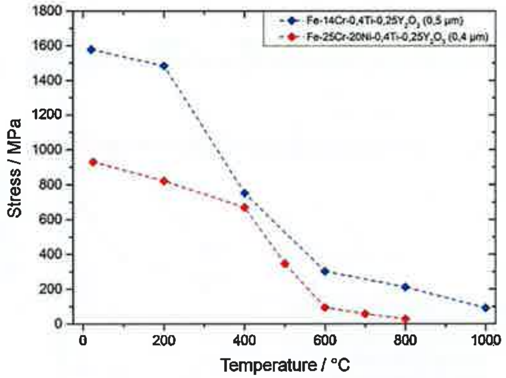
## Compression tests on ANC 25/20



- Constant strain rate: $10^{-4}$ $s^{-1}$
- Tests between room temperature and 800 °C

- Strain higher than 10 % (tests stopped manually)
- Strength at 0.2 % plastic deformation at 930 MPa at RT
- Strong decrease of strength above 400 °C

# Temperature dependence of strength



- Constant strain rate: $10^{-4}$ $s^{-1}$
- Tests between RT and 1000 °C

> Strength of FNC 14 > ANC 25/20 at all temperatures
> Drop of strength for ANC 25/20 at higher temperature
> Slight decrease of strength above 600 °C

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# Strength in low temperature range

- Linear superposition of hardening contributions

$$\sigma_{ys} = \sigma_0 + \sigma_{HP} + \sigma_{OR} + \sigma_{MK}$$

- Non-linear interaction cannot be excluded
- Contribution of fine grain hardening (Hall-Petch) [1]

$$\sigma_{HP} = \sqrt{\frac{G(T)}{G(300\,K)}} \cdot \frac{k_{HP}}{\sqrt{d}}$$

- Contribution of particle hardening (Orowan)

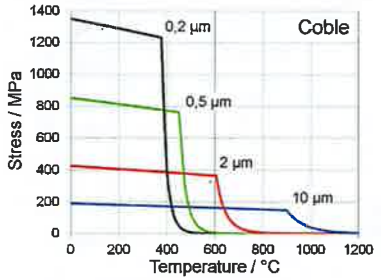
$$\sigma_{OR} = M\sqrt{\frac{6f}{\pi}} \cdot \frac{Gb}{d_p}$$

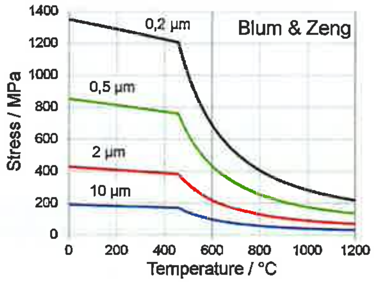
[1] Hirth und Lothe, Theory of Dislocations, John Wiley & Sons, Inc (1982), S. 789

## Strength in high temperature range

- Drop of strength due to creep
- Coble (diffusional creep) [1]: $\sigma_c = \frac{kTd^3\,\dot{\varepsilon}}{47\Omega\delta_B D_{B0}}\, exp\left(\frac{Q_B}{RT}\right)$
- Blum & Zeng (dislocation based creep [2]):

$$\sigma_{BZ} = G\left[\frac{\pi(1-\nu)\,M^9}{1{,}2^4}\right]^{1/8} \cdot \alpha\left[\frac{1-c+c^3}{c^3}\right]^{1/2} \cdot \left[\frac{kT\dot{\varepsilon}}{GbD_{B0}}\right]^{1/8} \cdot exp\left(\frac{Q_B}{8RT}\right) \cdot \left(\frac{d}{b}\right)^{1/2}$$





[1] R. L. Coble, J. Appl. Phys. (US) 34 (1963)
[2] J. H. Schneibel et al., Acta Mater. 59 (2011)

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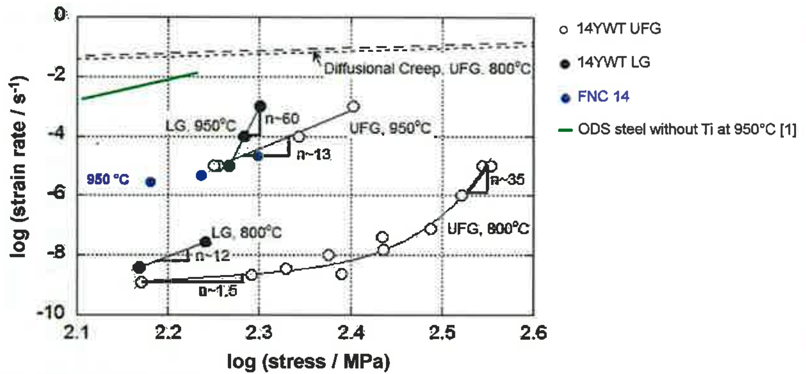
## Diffusional Creep

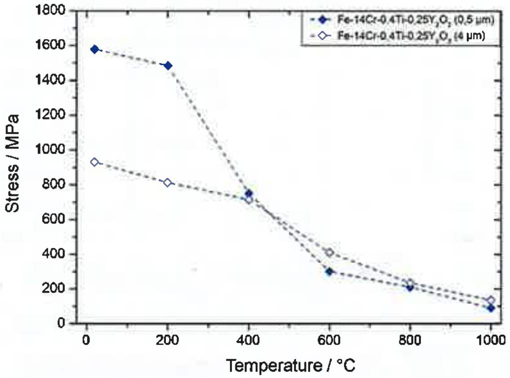


Schneibel et al., Script. Mater. (2009)

- Nanoclusters influence creep mechanism
  - Strain rate lower than expected for Coble creep

[1] Susila et al , MSE A528, 2011

## Grain size dependency of the compression strength of **FNC 14**



- Annealing at 1100 °C for 100 h
- Grain growth to 4 µm
- Constant strain rate: $10^{-4}$ s$^{-1}$

- ➢ Strength of **FNC 14** decreases from 1600 MPa to 900 MPa at RT
- ➢ Drop of strength at higher temperature for larger grain size?

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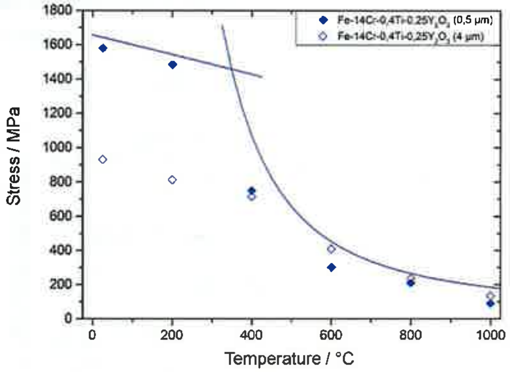
## Modeling the compression strength of **FNC 14**

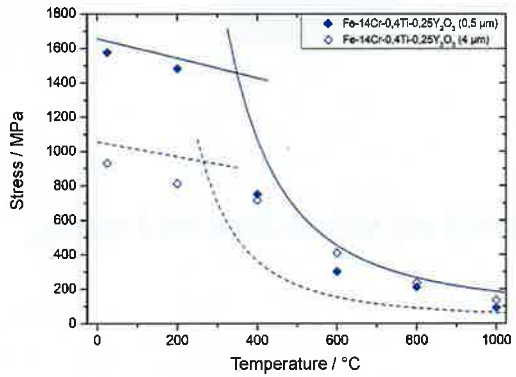


- Low temperature range
- $\sigma_0 + \sigma_{MK}$ from Kanthal A1 [1]
- Assumptions for $\sigma_{OR}$ [1]: grain boundary vs. grain interior
  - Number density smaller
  - Cluster size the same

- ➢ High temperature range: Use of Blum & Zeng model
- ➢ Very good agreement for small grain size

[1] J. Schneibel et al., Acta Mater. 35 (2011) 1300

## Modeling the compression strength



- After annealing at 1000 °C for 100 h
  - Drop of strength not well depicted

- ➢ Model has to be adjusted for larger grain size
- ➢ Not analyzed yet: Transferability to austenitic ODS steel

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## Summary

- Production of ferritic and austenitic ODS steels
- Large number of Y-Ti-O-rich nanoclusters (< 5 nm) by APT measurements
- Very small grain size after FAST process (~ 0.5 µm)
- Stability of the microstructure up to 1000 °C

- Performing compression tests between RT and 1000 °C
- Strength of ferritic ODS steel higher at all temperatures
- Drop of strength between 400 °C and 600 °C observed

- Modeling of the strength in progress
- Low temperature: Good agreement for ferritic ODS steel
- High temperature: Blum & Zeng model promising

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## Thank you very much for your attention!

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