IWSMT-10
October 18-22, 2010, Beijin, China

Current Status of Toughness Enhanced W-1.1%TiC Development

Hiroaki KURISHITA

International Research Center for Nuclear Materials Science, IMR, Tohoku University, Oarai, Ibaraki 311-1313, Japan

Outline

1. Introduction
2. Features of TFGR W-1.1TiC
   （Toughness-enhanced, Fine-Grained W-1.1%TiC in the Recrystallized state)
3. Processing technology for adequate sample dimensions
4. Summary and future work

Contributors

H.Arakawa[a], S.Matsuo[a], M.Kajioka[b], T.Sakamoto[b], S.Kobayashi[b], K.Nakai[b], V.Yardley[c], S.Tsurekawa[c], T.Takida[d], M.Kato[d], M.Kawai[e], N.Yoshida[f]

[a] International Research Center for Nuclear Materials Science, IMR, Tohoku University, Oarai, Ibaraki, Japan

[b] Department of Materials Science and Biotechnology, Ehime University, Matsuyama, Japan

[c] Department of Materials Science and Engineering, Graduate School of Science and Technology, Kumamoto University

[d] A. L. M. T. Corp., Toyama, Japan

[e] Institute of Material Structure Science, KEK, Tsukuba, Japan

[f] Institute for Applied Mechanics, Kyushu University, Kasuga, Fukuoka, Japan

Ultra-fine grained (UFG) W-(0.25-1.5)%TiC

- **Tungsten (W) is the most promising for use in advanced nuclear energy systems, such as the high power neutron source solid target, plasma facing materials/components, etc, because of its many advantageous properties.**
- **However, its applicability crucially depends on the degree of mitigation of serious embrittlement caused by recrystallization and by proton, helium and neutron irradiations.**
- **Nanostructures are preferable for the mitigation of embrittlement, and ultra-fine grained (UFG), densified W-(0.25-1.5)%TiC compacts with grain sizes of 50~200nm and fine TiC dispersoids at grain boundaries (GBs) were developed in 2005 by powder metallurgical methods utilizing mechanical alloying (MA) and HIP.**

Superplasticity-based microstructural modification (SPMM)

- **The UFG compacts exhibited good resistances to fast neutron and 3MeV He irradiations.**
- **On the other hand, the compacts even in the unirradiated state did not exhibit any ductility below 500C mainly due to mostly weak GBs of random orientations, extremely high yield strength (5~6GPa) and presence of residual pores (RD:~99%).**
- **Recently, a new microstructural modification method for removal of residual pores, adjustment of grain size and strengthening of weak GBs *in the recrystallized state* was developed. The method is based on activation of GB sliding by superplastic deformation that is characteristic of UFG and thus defined as "superplasticity-based microstructural modification (SPMM)".**

TFGR W-1.1TiC

- **Optimized SPMM was applied to UFG W-(0.25-1.5)TiC compacts and successfully produced *toughness enhanced W-1.1%TiC that exhibits an appreciable bend ductility even at room temperature, accompanied with transgranular fracture, suggesting that the GBs are significantly strengthened*.**

  (IWSMT-9: Kurishita et al., J. Nucl. Mater. 398 (2010), 87-92)

- **The toughness enhanced W-1.1%TiC exhibited equiaxed, fine-grained structures in the recrystallized state (GS: 0.5~1.5μm depending on MA atmosphere (H_2, Ar): no more UFG) and is designated as TFGR W-1.1TiC; W-1.1TiC/H_2 (MA:H_2), W-1.1TiC/Ar (MA: Ar).**

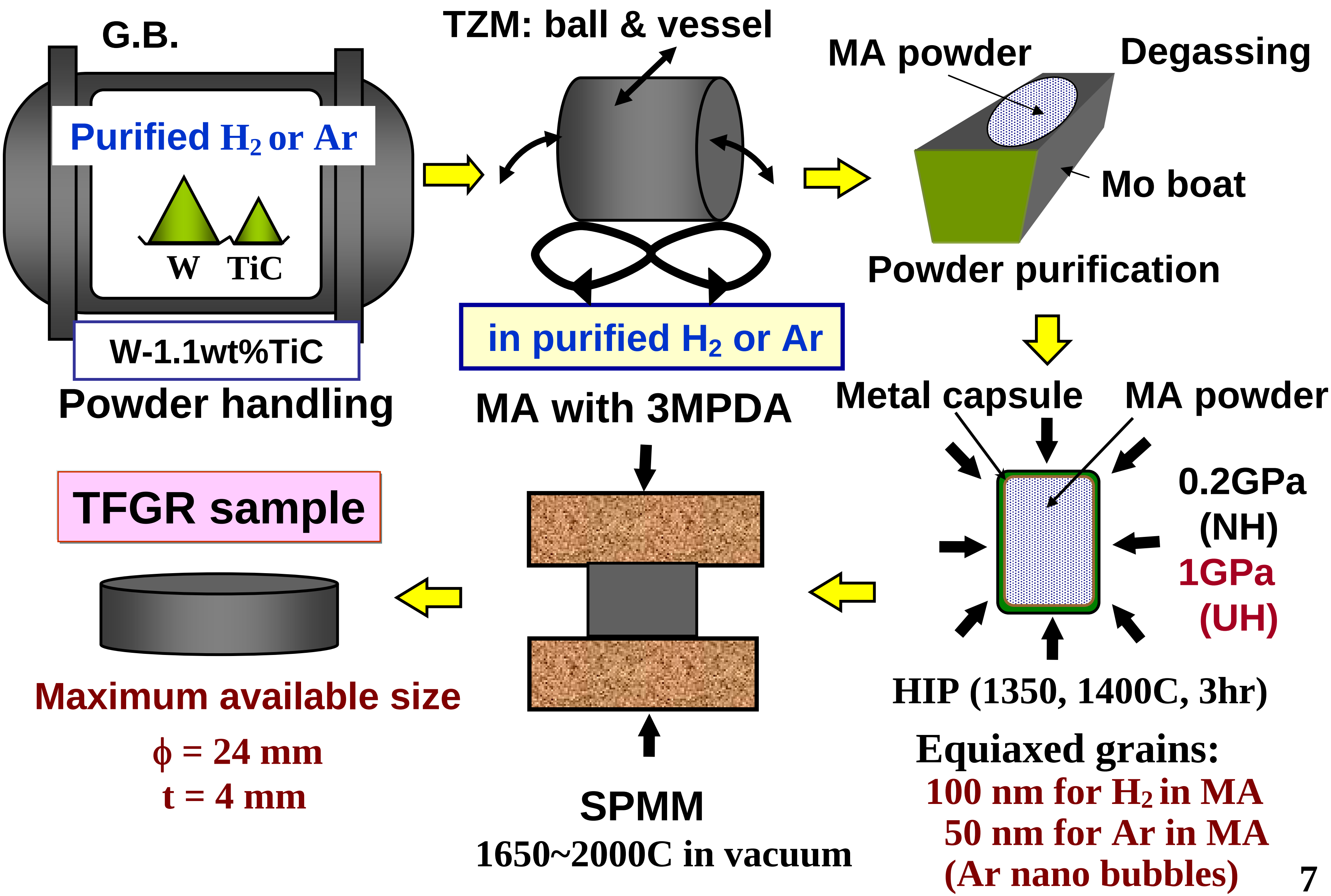
- **However, the features of TFGR W-1.1TiC are not fully clarified, and the maximum specimen size available by the current processing techniques is limited.**

Objectives

1. **Features of TFGR W-1.1TiC**
   - **Microstructures**
     Grain size, GB orientations
     TiC dispersoids at GBs and grain interior
   - **Mechanical properties**
     Vickers microhardness, bend properties
     tensile properties
   - **Resistance to 1650~2000℃ heating in vacuum**

2. **Development of a new processing technology to meet the suitable sample dimension requirement for the aimed applications.**

Processing of TFGR W-1.1TiC



Features of TFGR W-1.1TiC

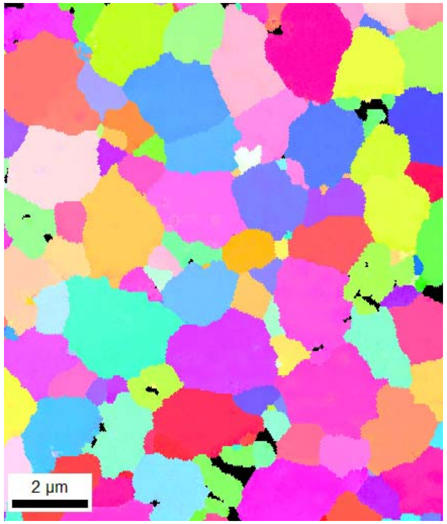
- **Microstructures**
- **Mechanical properties**
- **Resistance to 1650~2000℃ heating in vacuum**

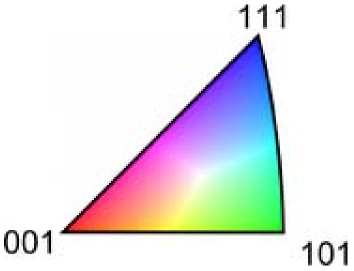
Grain size distribution in TFGR W-1.1TiC
(EBSP: Electron Backscatter Diffraction Pattern)

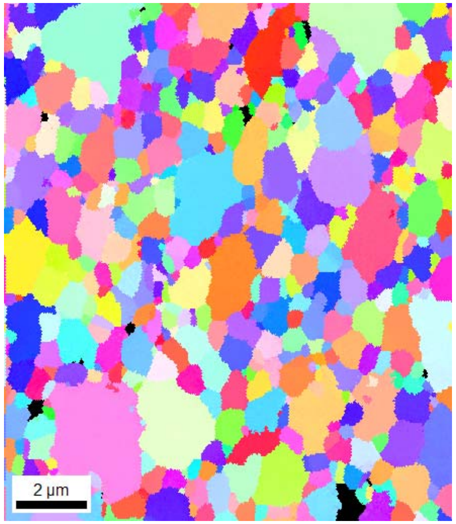
W-1.1TiC/H_2-NH

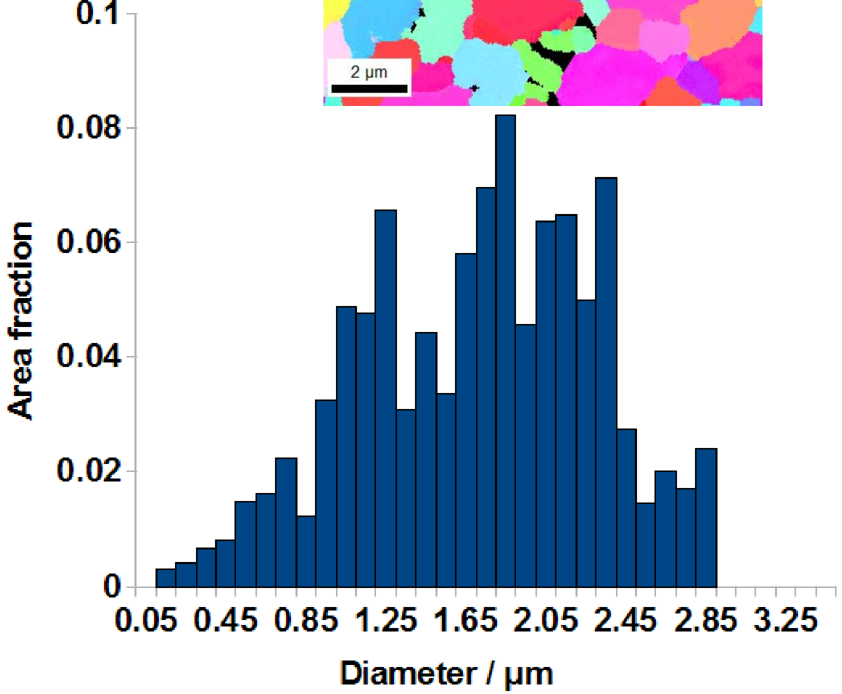
SPMM:1600C

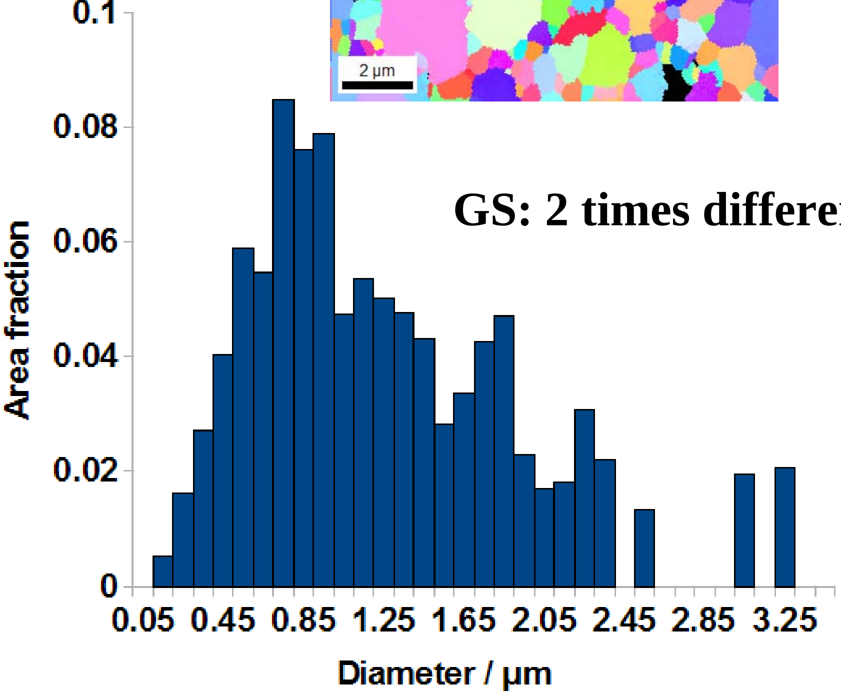
W-1.1TiC/Ar-UH **RD:Ar**





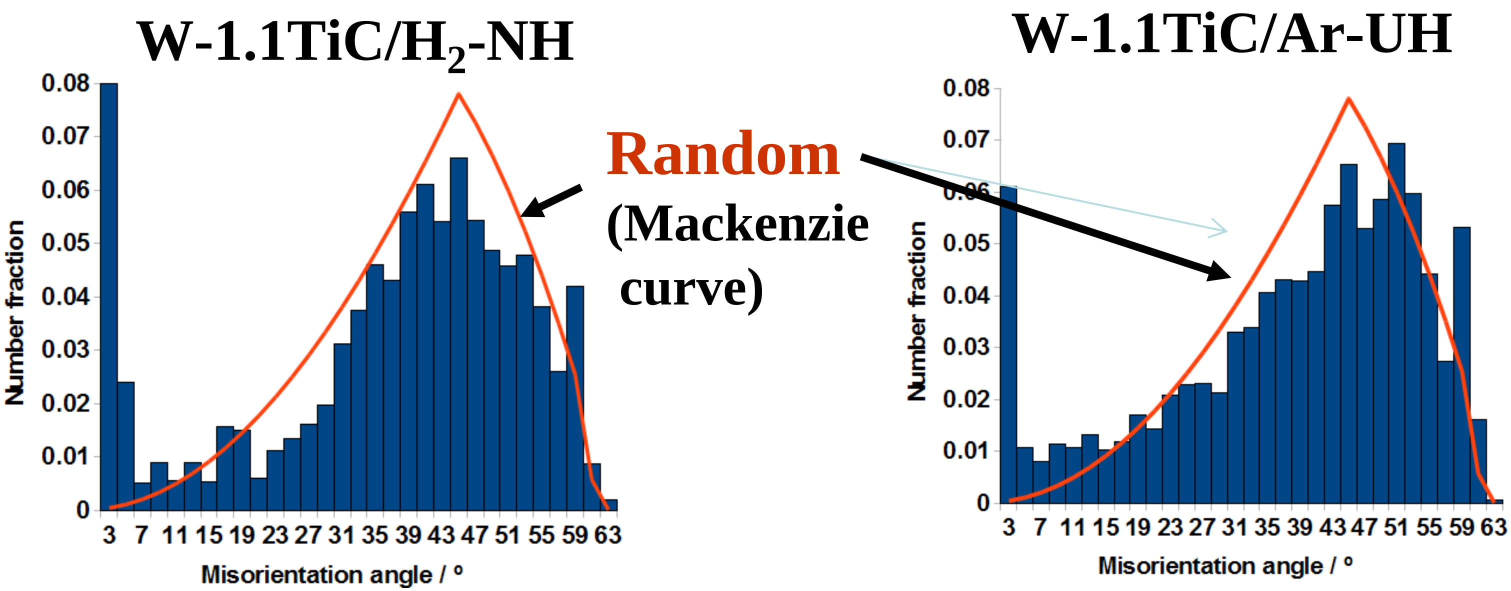


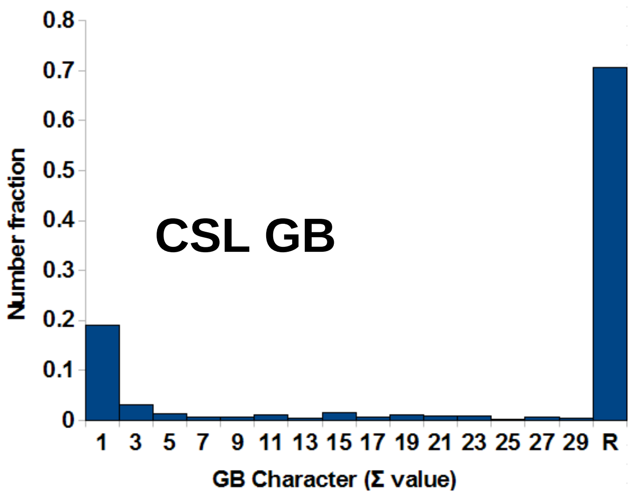


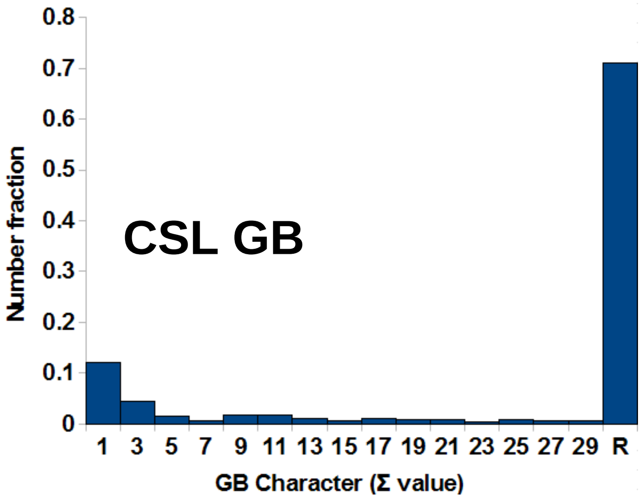


GS: 2 times different

Grain boundary orientation in TFGR W-1.1TiC

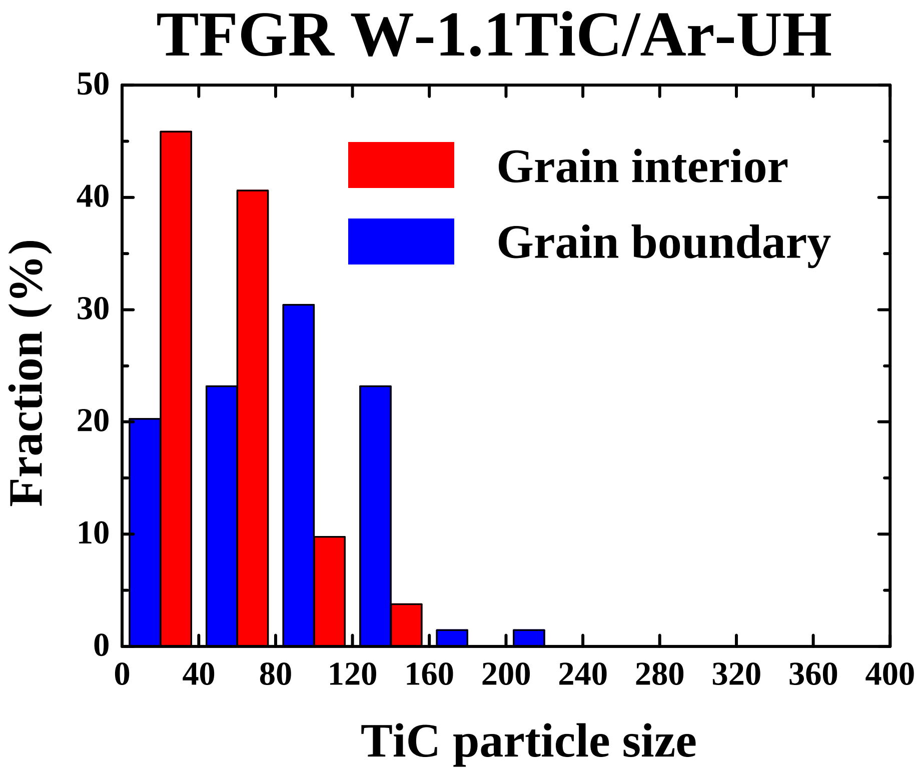


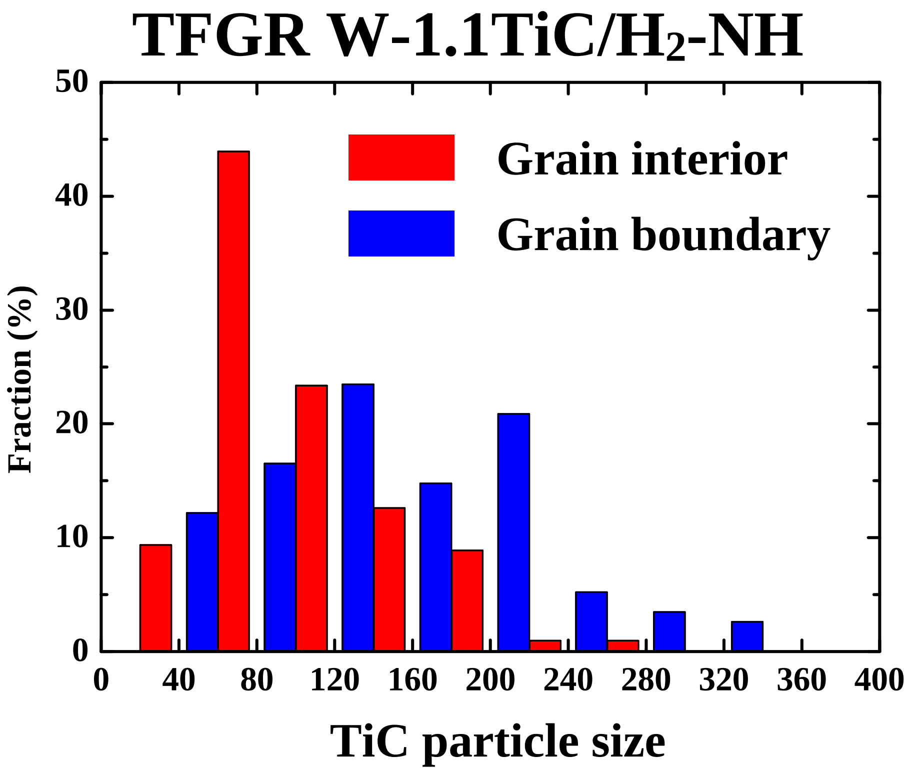




GBs are mostly composed of random orientations (~70%)

Size distribution of TiC dispersoids
(SPMM: 1650C)





- The size of TiC disperoids

  GB >> Grain interior

  W-1.1TiC/H_2-NH >> W-1.1TiC/Ar-UH (GB & grain interior)

Hardness and bend properties

Hardness (RT)

- HV = 656 for W-1.1TiC/Ar-UH
  HV = 570 for W-1.1TiC/H_2-NH

GS effects

Bend properties

- Appreciable plastic deformation occurs at RT for W-1.1TiC/Ar-UH and W-1.1TiC/H_2-NH
- Proportional limit at RT : ~ 2.9GPa for W-1.1TiC/ Ar-UH
  ~ 2.2GPa for W-1.1TiC/H_2-NH
- Fracture strength at RT : ~ 4.4GPa for W-1.1TiC/Ar-UH and ~ 3.3GPa for W-1.1TiC/H_2-NH
- Full bend without fracture at ~250C for W-1.1TiC/H_2-NH

High temp. tensile properties

- **Flow stress**

  Large temperature dependence

  1.3 GPa at 800C

  0.1 GPa at 1600C

- **Strain rate sensitivity of flow stress (*m*)**

  m **: ≤0.2 below 1100C**

  m **: ≥ 0.5 above 1600C**

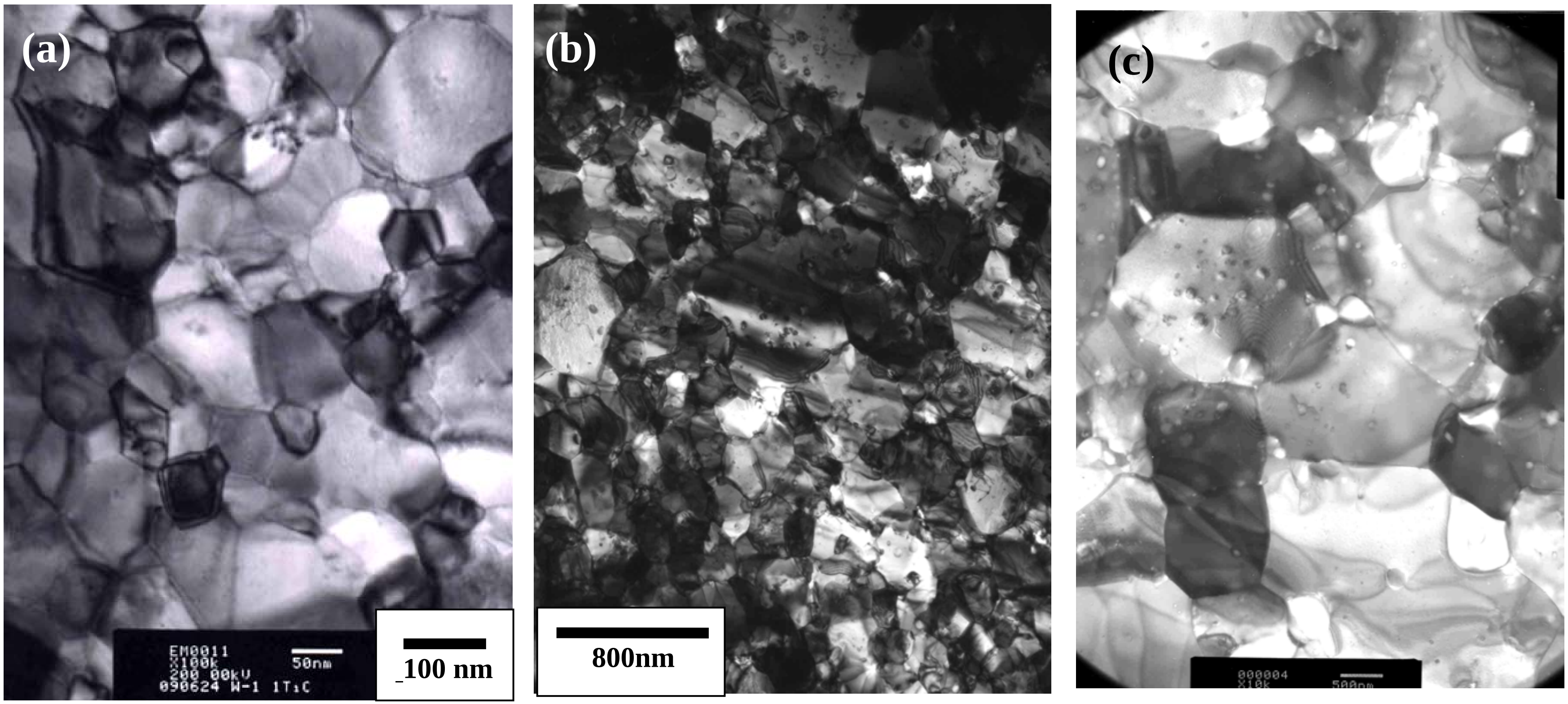
- **Deformation controlling mechanism**

  Recovery control of internal stress below 1100C

  Grain boundary sliding above 1600C

Effect of SPMM temperature on TEM microstructures

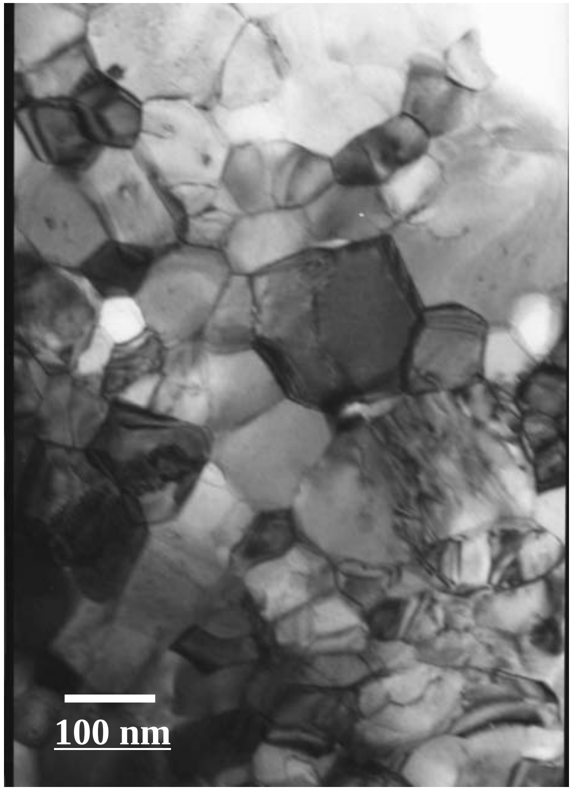
W-1.1TiC/Ar-UH

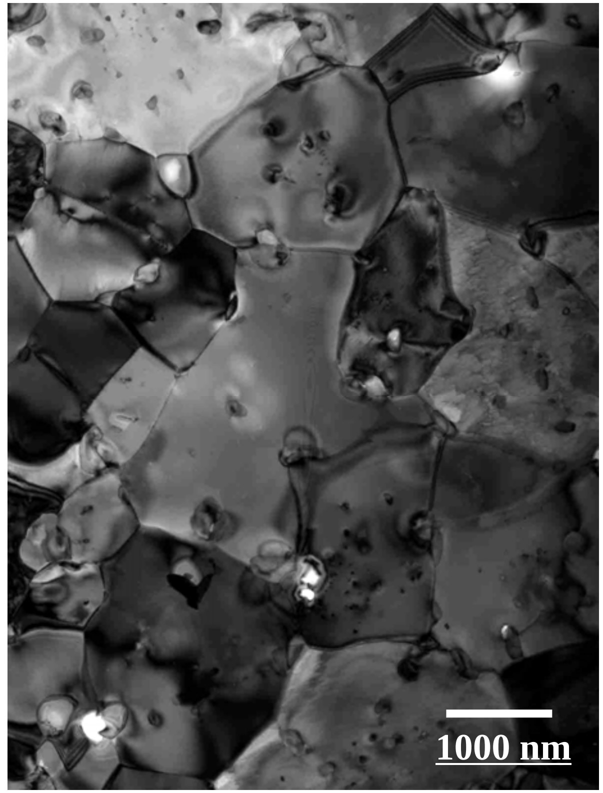


As-HIP (1400C x 3h)	1650C x 3h	1650-2000C x 3h
G.S. = 60 nm	G.S. = 520 nm	G.S. = 1600 nm

Effects of SPMM temperature on TEM microstructures

W-1.1TiC/H_2-NH

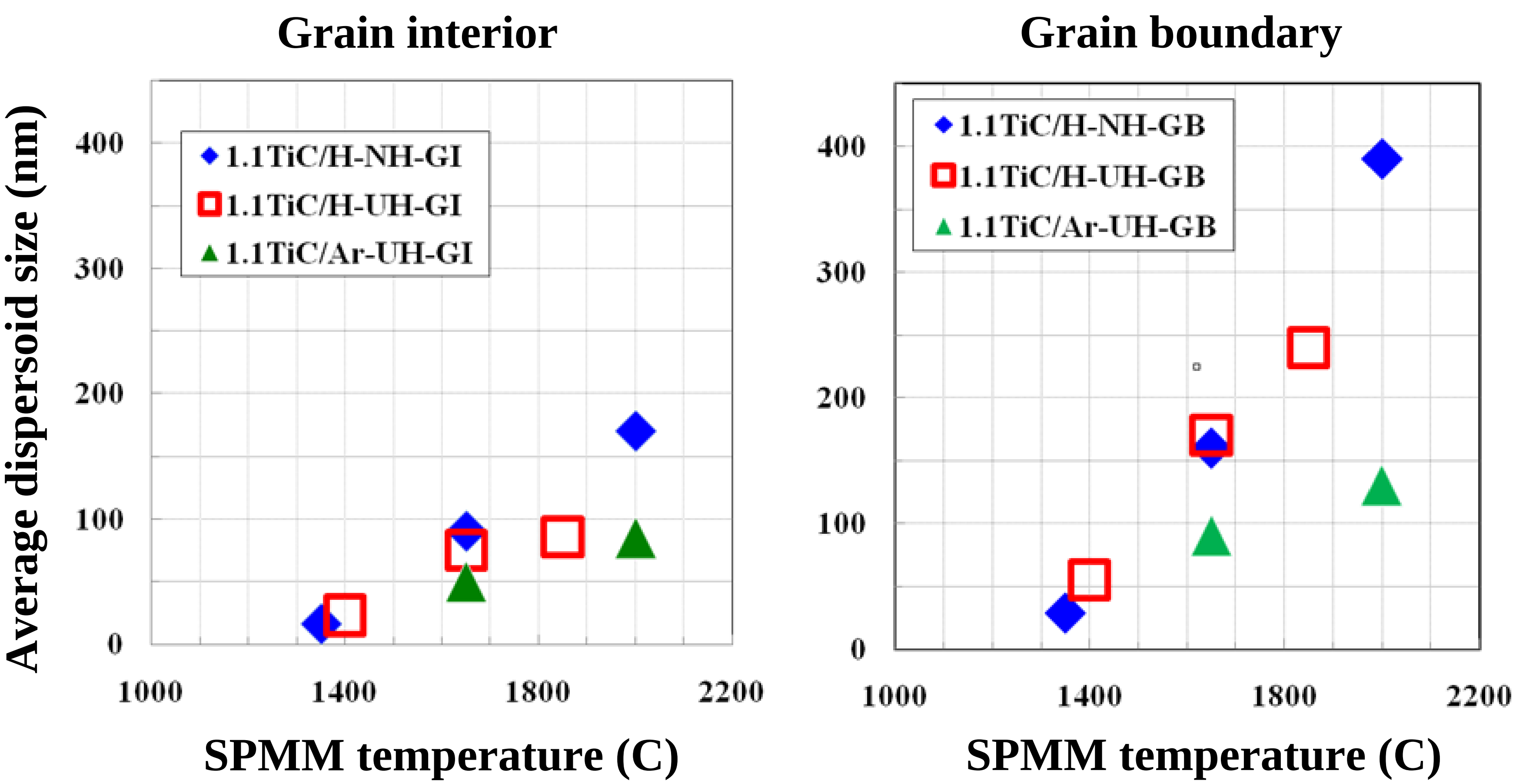




As-HIP (1350C x 3h) | **1650C x 3h** | **1650-2000C x 3h**

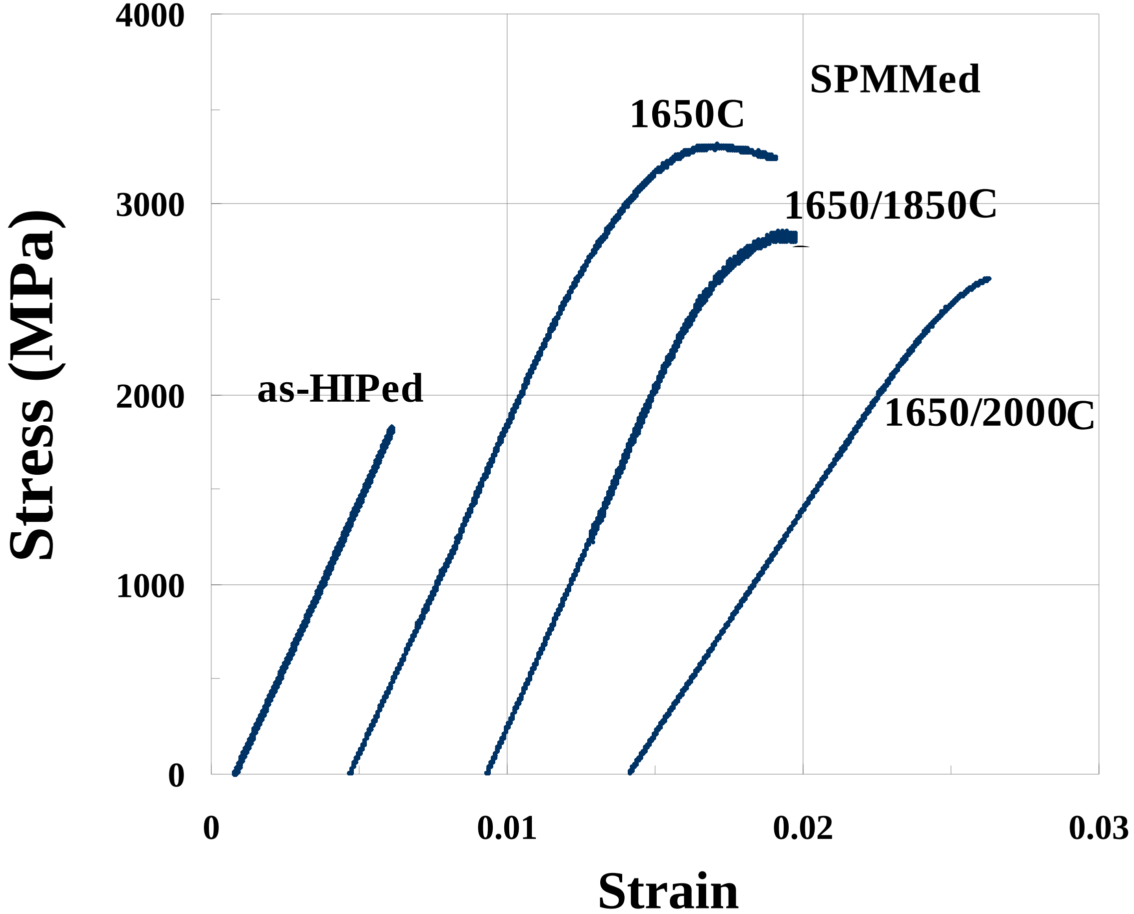
Grain size : 90 nm | **Grain size : 1480 nm** | **Grain size : 2900 nm**

Effect of SPMM temp. on TiC dispersoid size



- The size of TiC dispersoids in grain interior and GBs increases with increasing SPMM temperature.
- W-1.1TiC/Ar exhibits much lower TiC growth rate than W-1.1TiC/H_2.

Effect of SPMM temperature on 3P bend stress strain curve at RT for W-1.1TiC/H_2-NH

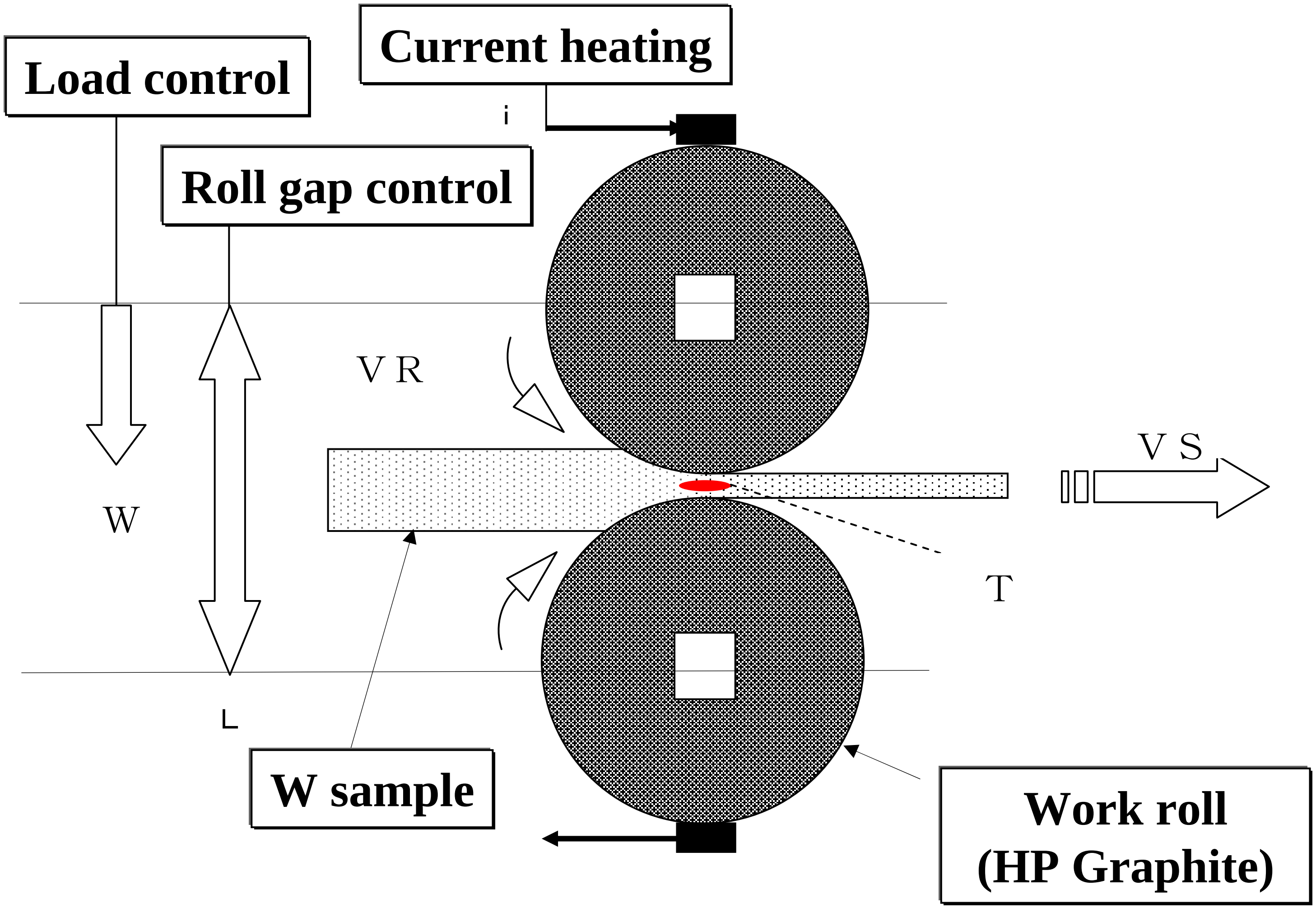


The samples exposed to 1850 and 2000C still exhibit slight RT ductility and much higher strength than as-HIPed, UFG.

Development of a new effective processing technology

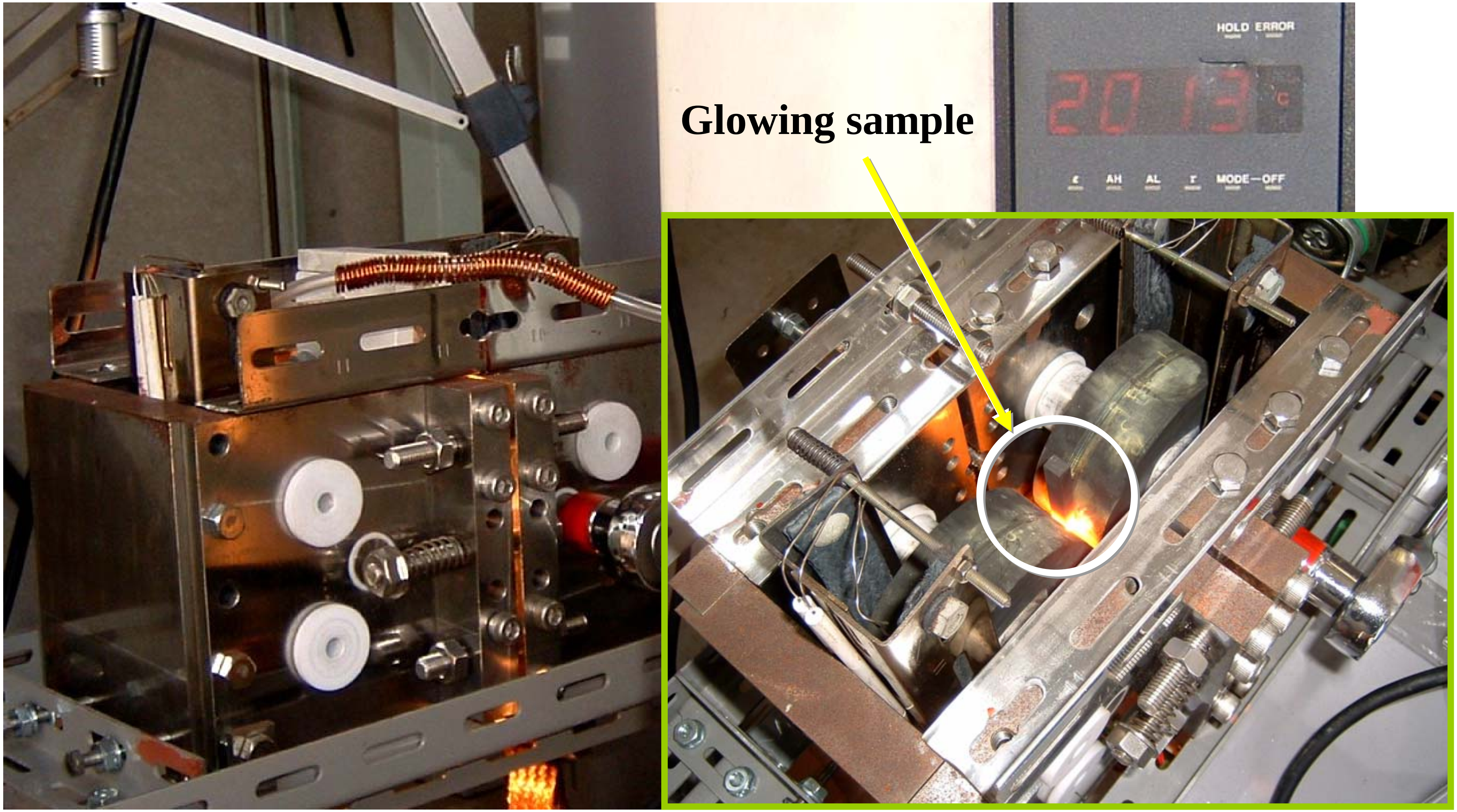
A rolling machine with the following specifications

1. Temperature control: 1500-2000C
2. Local heating of sample
3. Rolling rate control
4. Applied load control
5. Atmosphere control



Conceptional design of the rolling machine

Tungsten sample locally heated at 2013C



Summary

1. **TFGR W-1.1TiC exhibits fully recrystallized, equiaxed grain structures of mostly random GBs with larger TiC dispersoids than those in the grain interior.**
2. **Mechanical properties of TFGR W-1.1TiC including the hardness and bend properties around RT and tensile properties at high temperatures were shown.**
3. **Elevating SPMM temperature up to 2000C increases the sizes of equiaxed grains and TiC dispersoids, but the SPMMed W-1.1TiC/H_2-NH still exhibits a slight bend ductility and much higher fracture strength at RT than the as-HIPed, UFG compact.**
4. **Development of a new effective processing technology to meet the suitable sample dimension requirement for the aimed applications of TFGR W-1.1TiC is in progress.**

Future work

1. **Mechanism of GB strengthening by nanostructural analyses in TFGR W-1.1TiC**

2. **Effects of neutron and proton irradiations and high heat loading on microstructures and physical and mechanical properties of TFGR W-1.1TiC**

3. **Development of TFGR W compacts with TaC additions**

4. **Optimization and establishment of the processing conditions for the new fabrication equipment**

Thank you for your attention!