The new progress of fabrication of oxide dispersion strengthened fine grained tungsten

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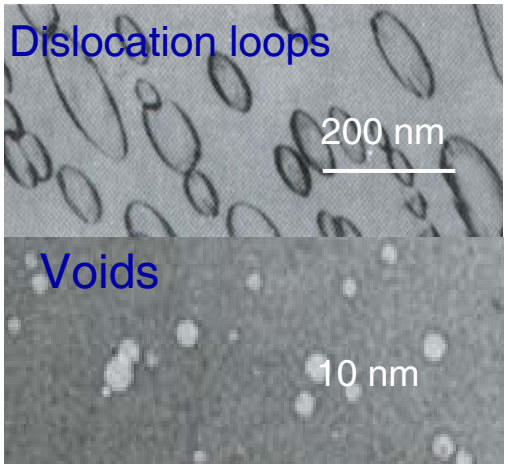
1. Motivation

W is a most promising candidate for spallation target, PFM in fusion reactor.

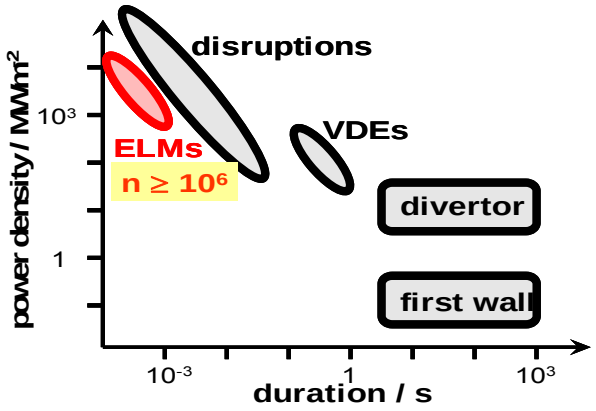
Challenges for W application in extreme enviroment:

- ✓Poor ductility, high DBTT;
- ✓Transient high heat load performance;
- ✓Irridation (H, He, n) effects

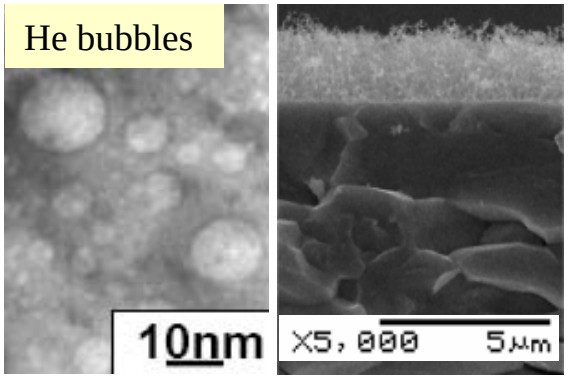


Transient heat loads:

1. Plasma disruptions
2. Vertical displacement events (VDEs)
3. Edge Localized Modes (Type I ELMs)



Irridation effects



From literatures

- **Fabrication of W with ultra fine grain size & particle dispersion strengthened W; New alloy design and new process technology**

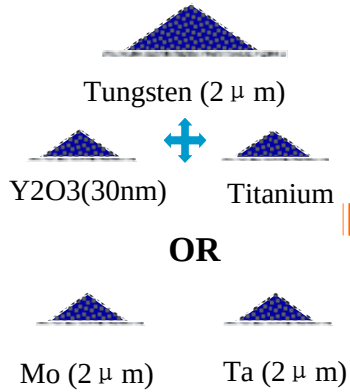
- **UFG (Nano structure)-W,**
- **ODS (CDS)-W**, W_f / W, AKS-W,
- W-Si-Cr alloy,
- MIM-W, SPD-W
- ……

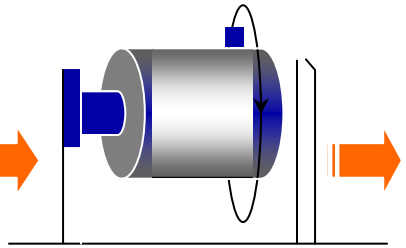
Conventional sintering process for W usually need high T and long time, inducing significant grain growth.

Fast resistance sintering technology has been developed for fine grained ODS W: Spark plasma sintering (SPS)

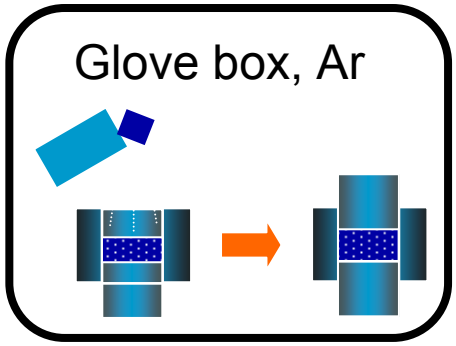
Motivation: invetigation the relationship between grain size of W, second phase, alloy element and its physical property.

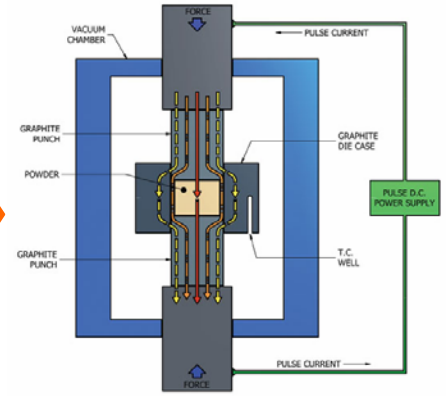
2. Fabrication process





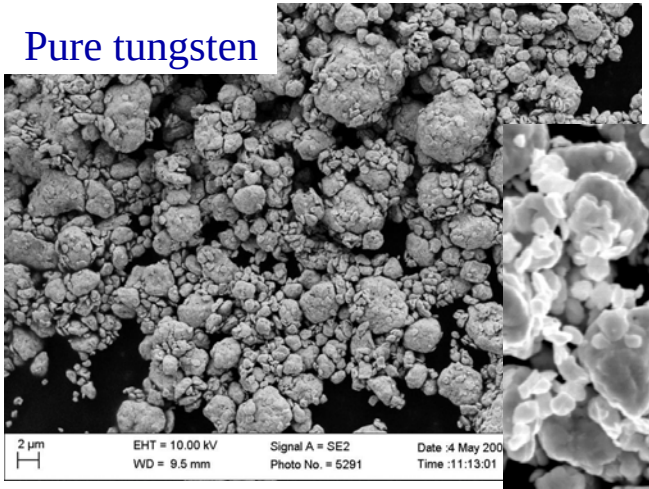
Mechanical alloying,
380rpm, 30h, Ar



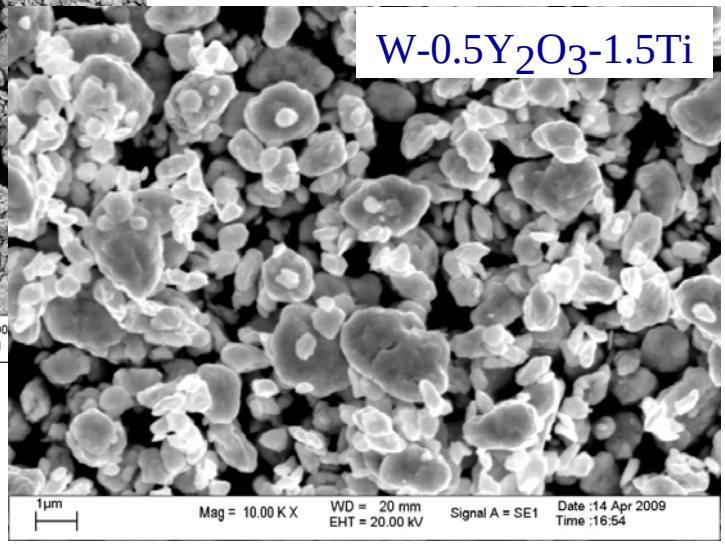


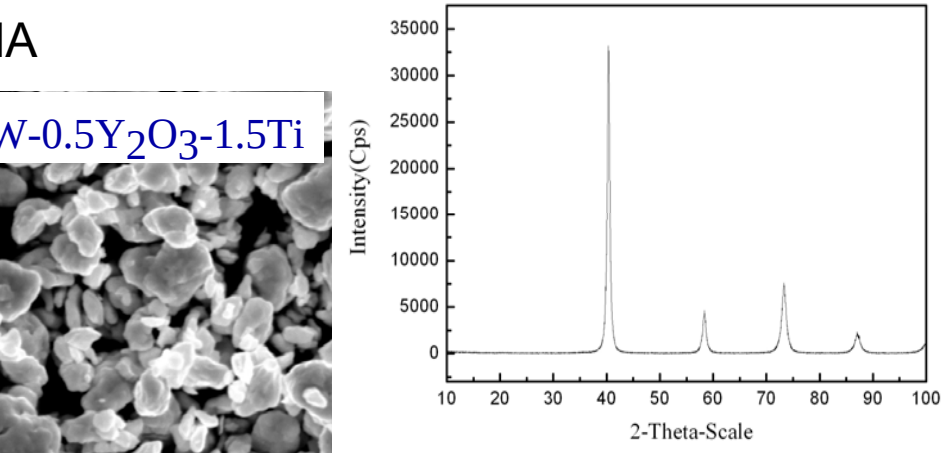
1700℃, 1min, 20 MPa

Pure tungsten



After MA

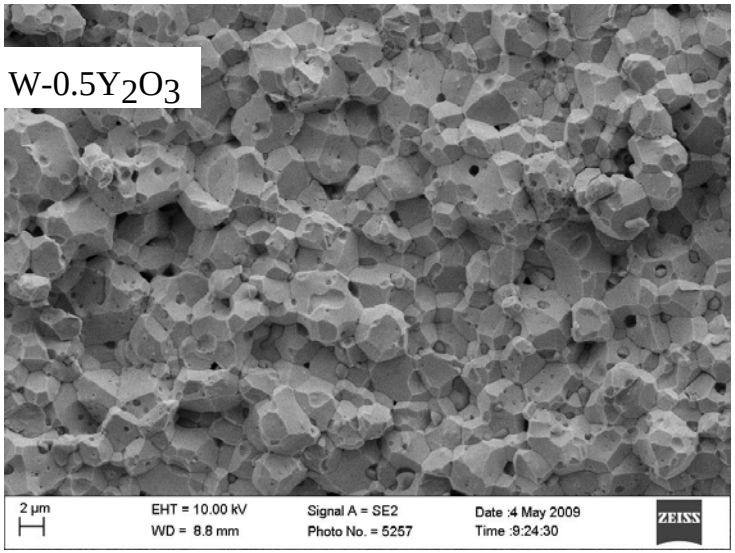


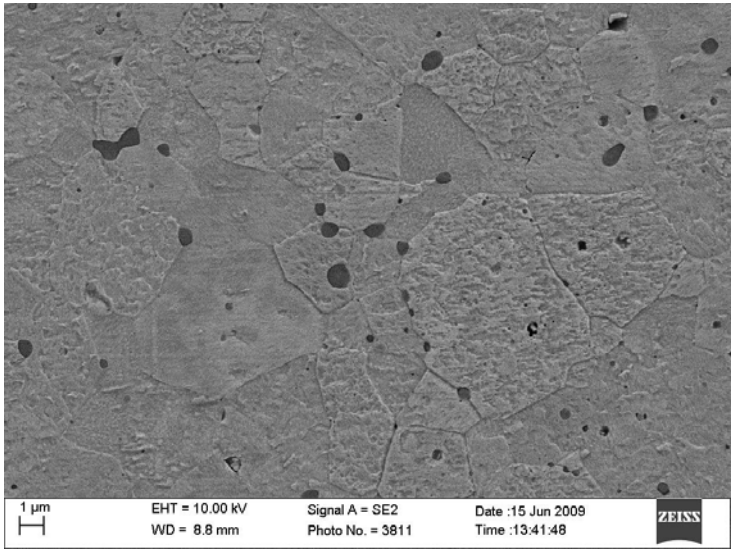


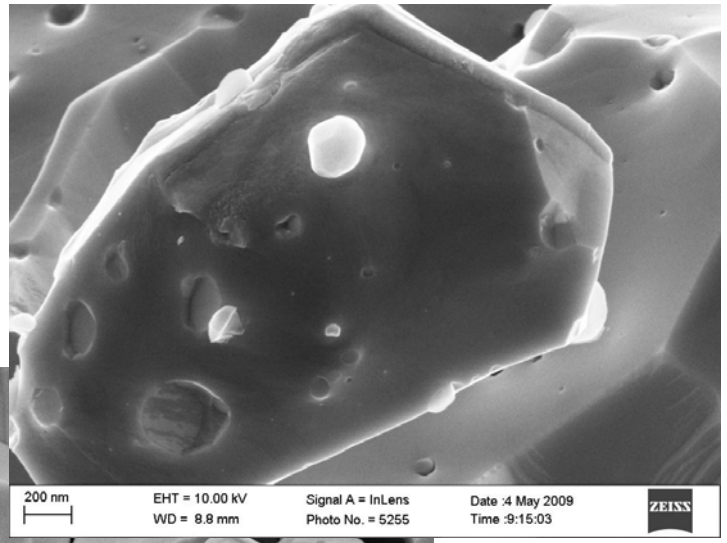
XRD of W-0.5Y2O3-1.5Ti powder after MA

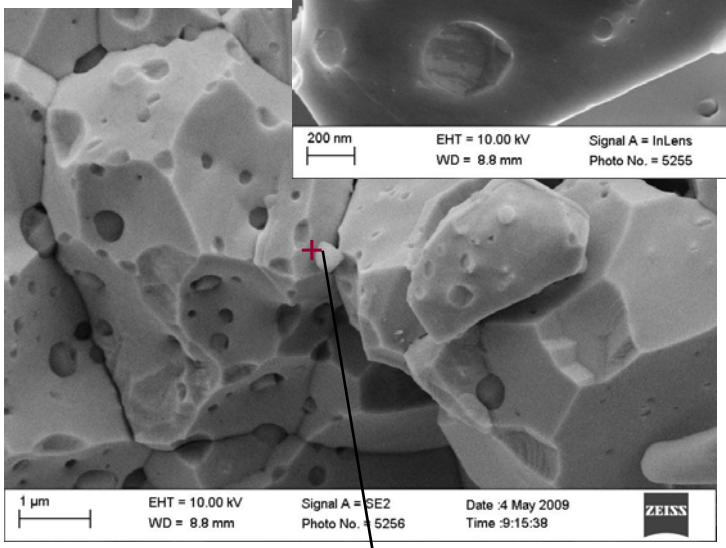
2. Microstructure observation

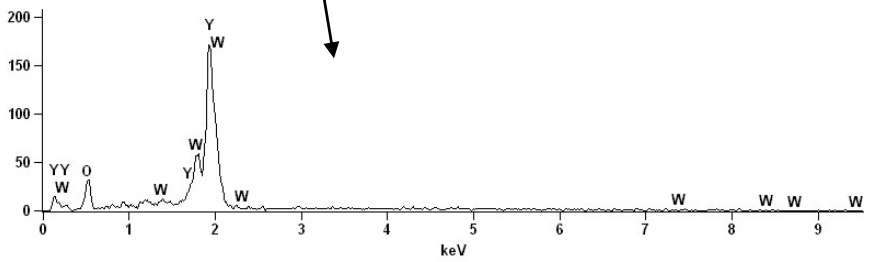
W-0.5Y_2O_3- xTi, x=0.5~2

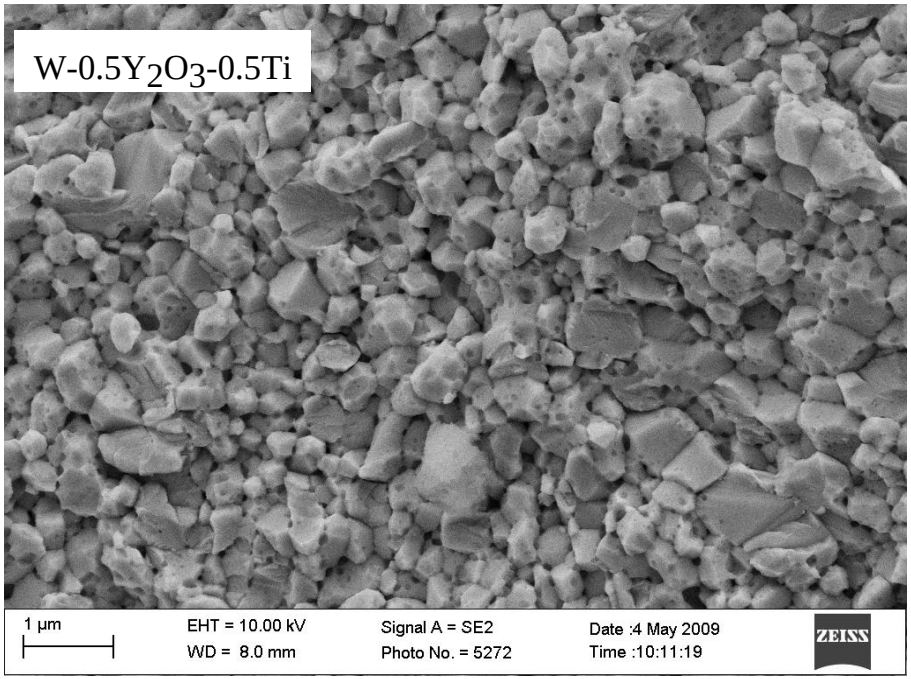


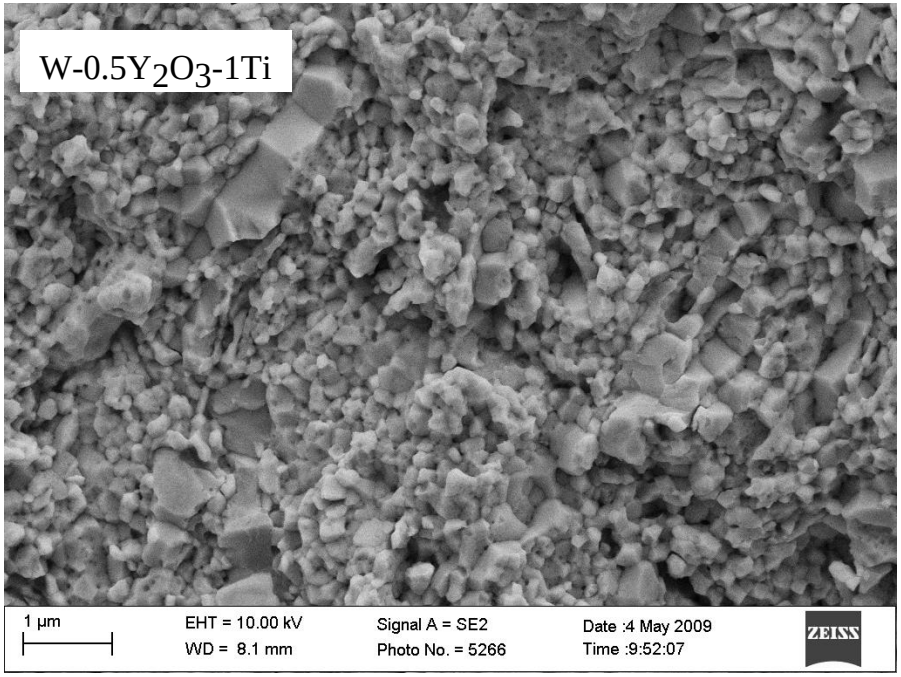









W-0.5Y2O3-0.5Ti
1 µm
EHT = 10.00 kV
WD = 8.0 mm
Signal A = SE2
Photo No. = 5272
Date :4 May 2009
Time :10:11:19
ZEISS


W-0.5Y2O3-1Ti
1 µm
EHT = 10.00 kV
WD = 8.1 mm
Signal A = SE2
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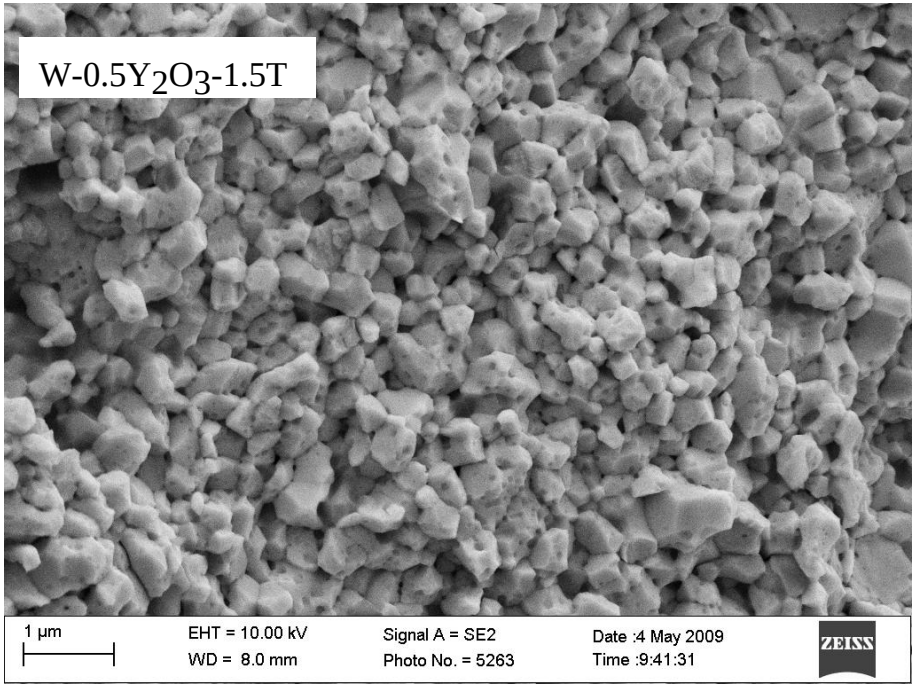
W-0.5Y2O3-1.5T
1 µm
EHT = 10.00 kV
WD = 8.0 mm
Signal A = SE2
Photo No. = 5263
Date :4 May 2009
Time :9:41:31
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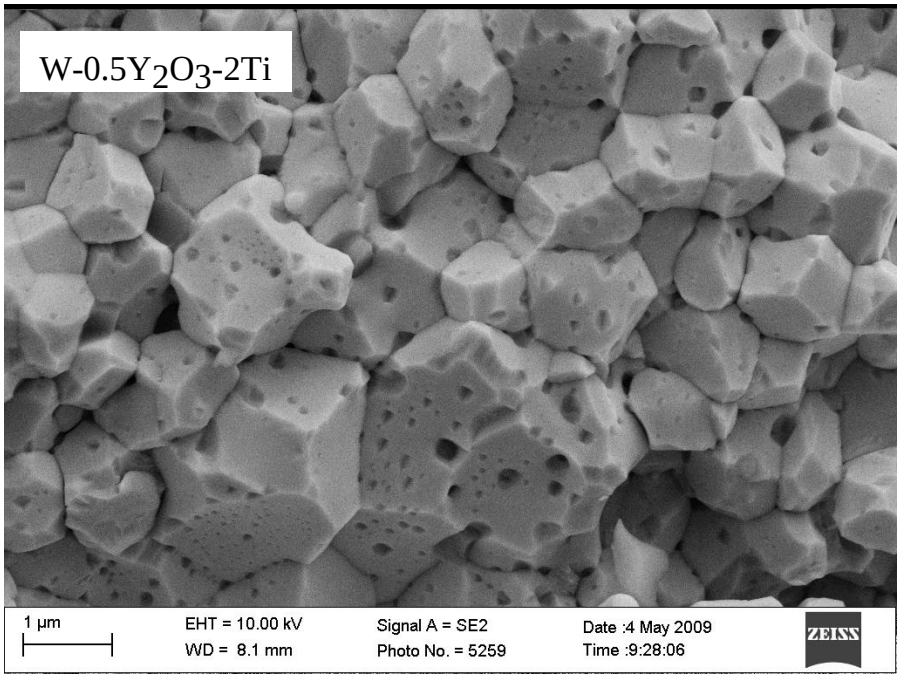
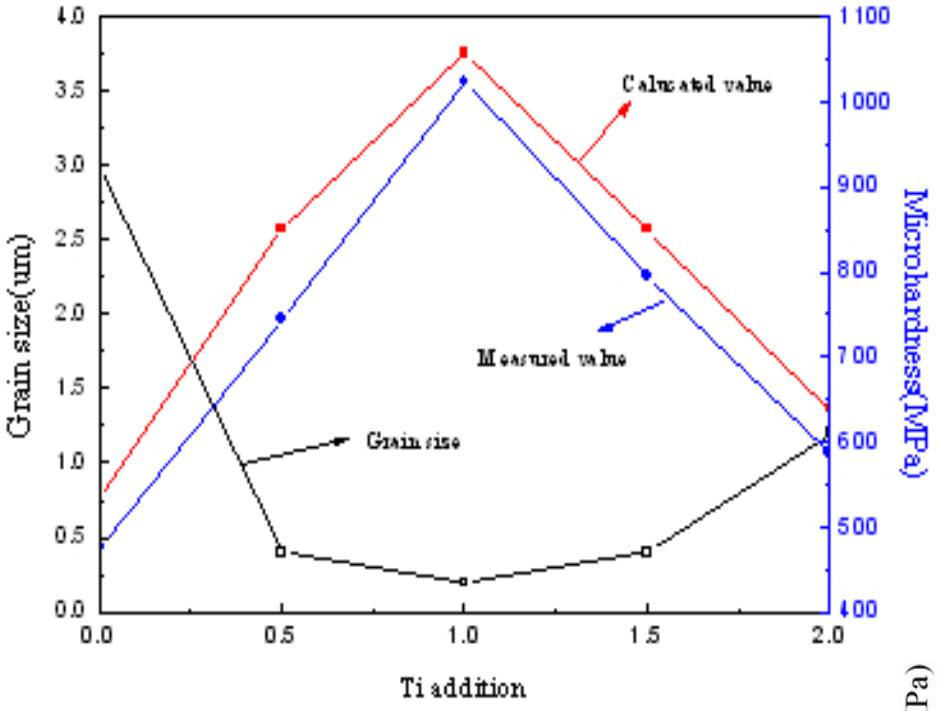
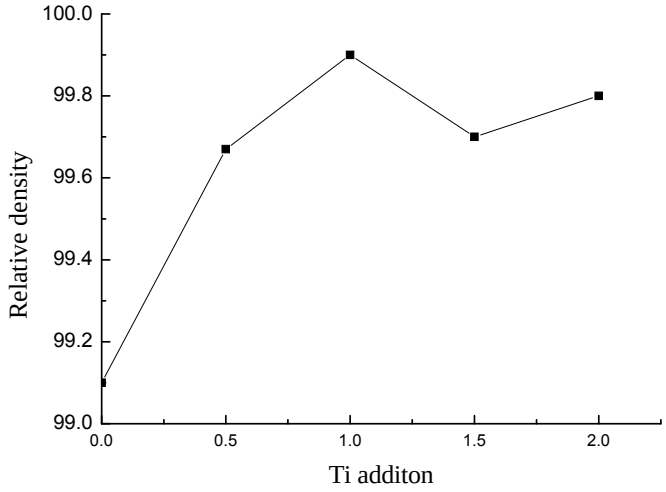
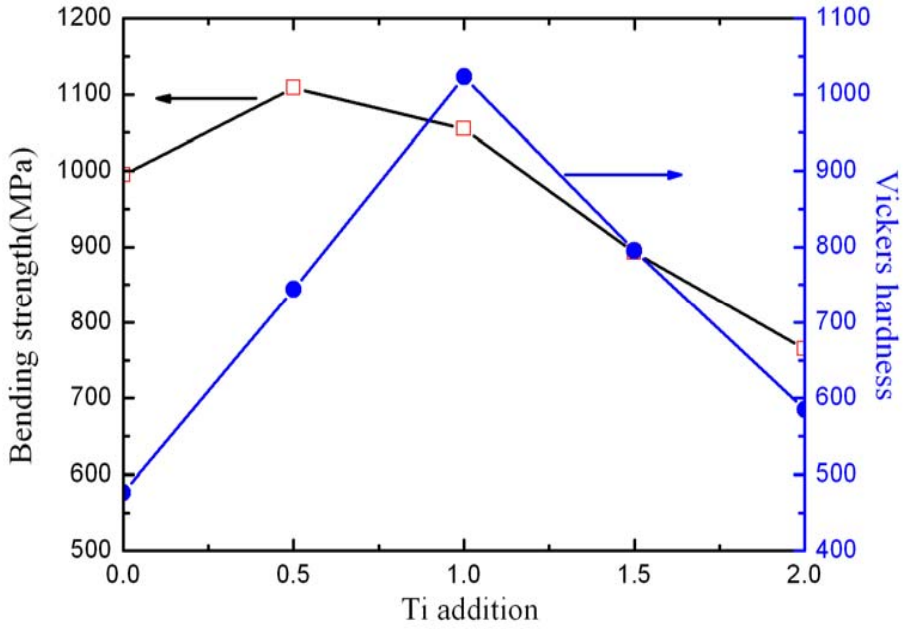

W-0.5Y2O3-2Ti
1 µm
EHT = 10.00 kV
WD = 8.1 mm
Signal A = SE2
Photo No. = 5259
Date :4 May 2009
Time :9:28:06
ZEISS

2. Mechanical properties



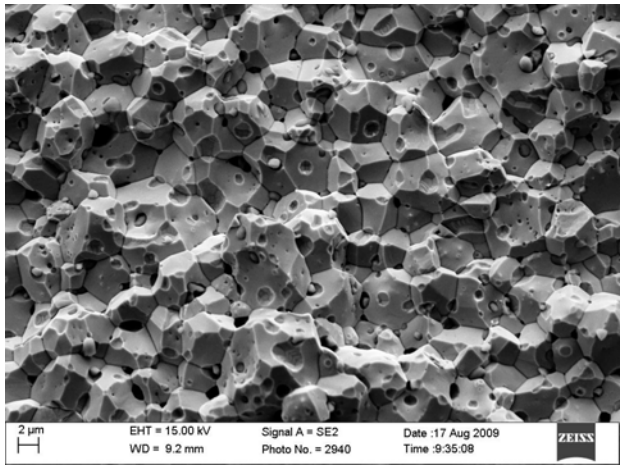


Micro-hardness
Three point bending strength

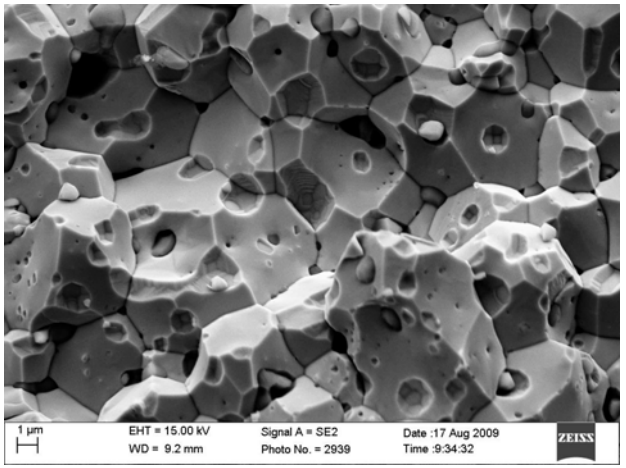


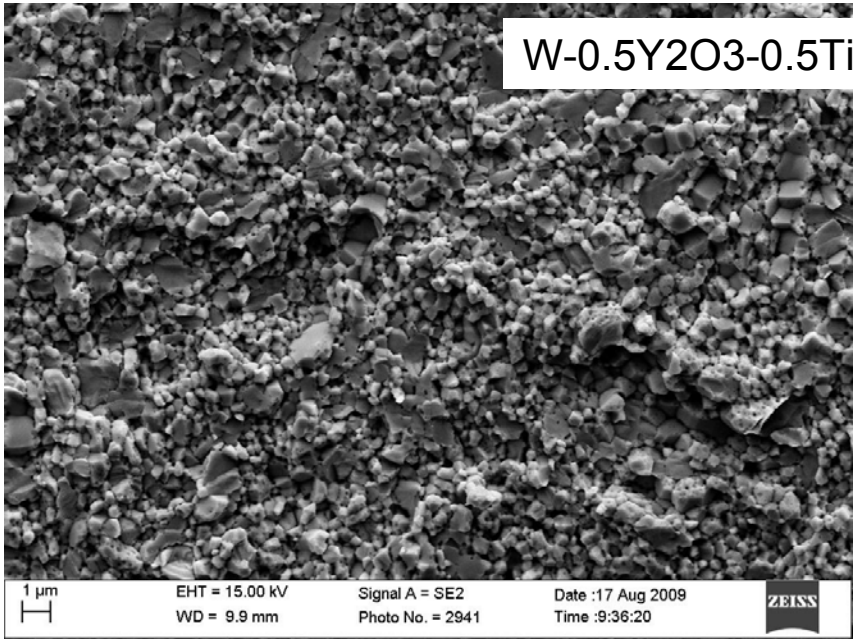
3. Annealing behavior

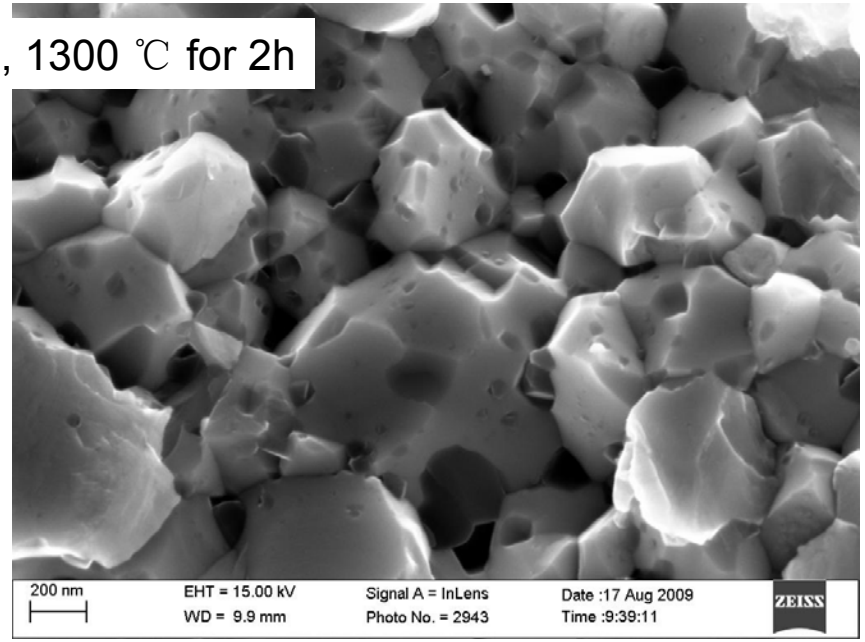
Annealing at 1100℃ for 2h			
sample	Bending strength, MPa	Micro-hardness, HV	grain size, μm
W-0.5Y2O3	1094.47	427.6	4.5
W-0.5Y2O3-0.5Ti	885.83	663.6	0.6
W-0.5Y2O3-1.5Ti	836.97	789.1	0.5
Annealing at 1300 ℃ for 2h			
sample	Bending strength, MPa	Micro-hardness, HV	grain size, μm
W-0.5Y2O3	877.93	433.2	7
W-0.5Y2O3-0.5Ti	949.57	652.1	0.8
W-0.5Y2O3-1.5Ti	726.54	770.0	0.6



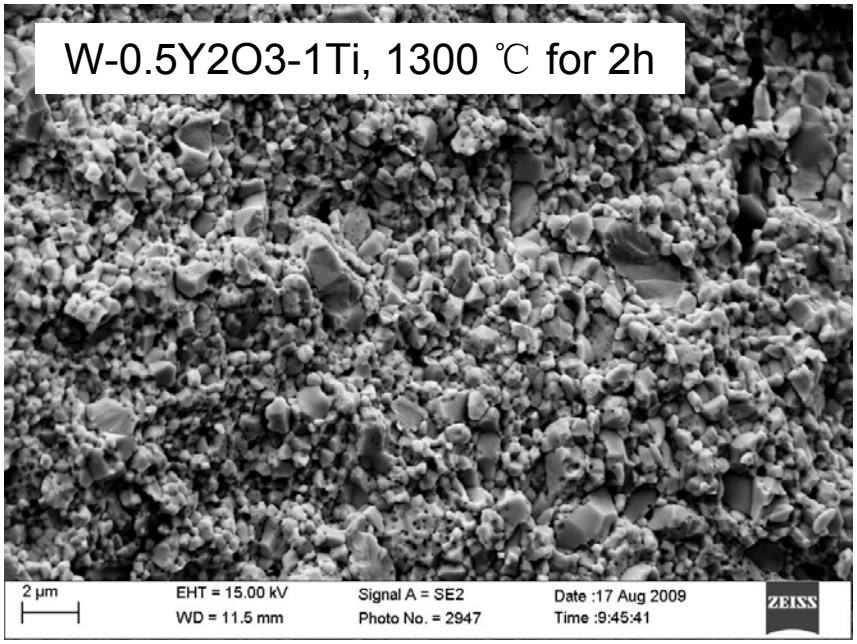
W-0.5Y2O3
after annealing
at 1300 ℃ for 2h

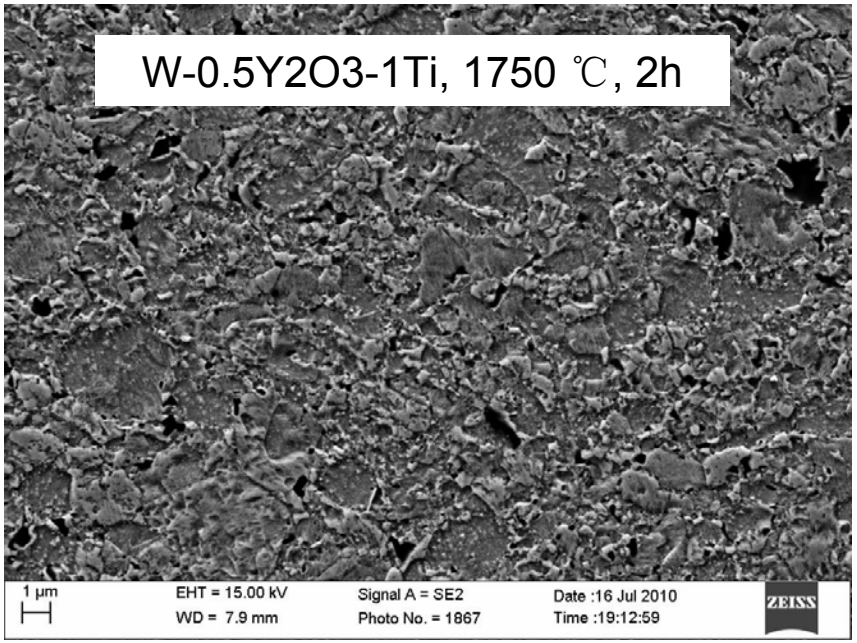




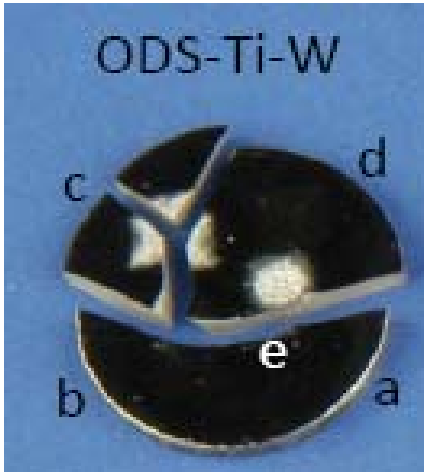


after annealing at 1300 ℃ for 2h

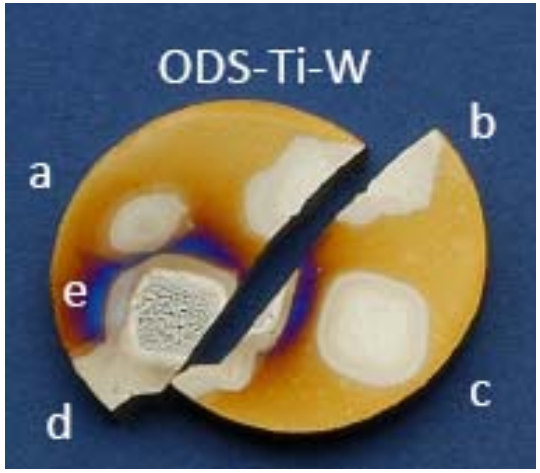




4. Transient HHF test by electron beam



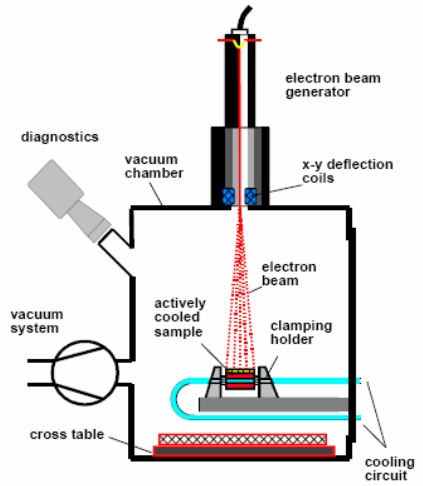
At RT



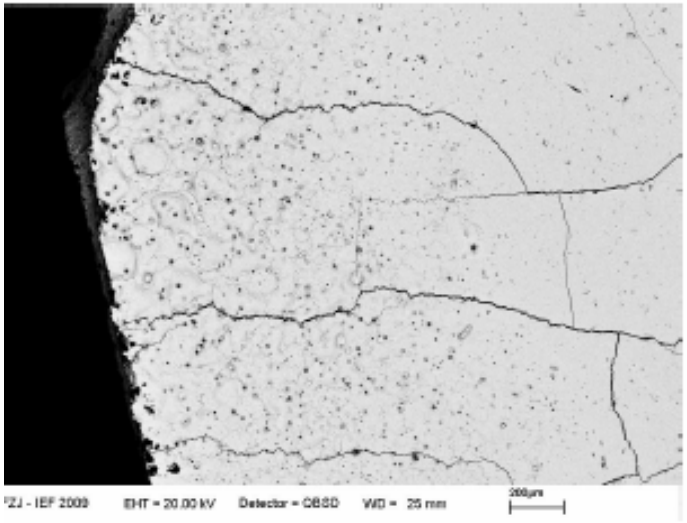
at 500 ℃

a 0.3GWm^{-2} ∆ t=5 ms, 1X
b 0.6GWm^{-2}, Δt=5 ms, 1X
c 0.9 GWm^{-2} Δt=5 ms, 1X
d 0.9 GWm^{-2} Δt=1 ms, 100X
e 1.2 GWm^{-2} Δt=5 ms, 1X

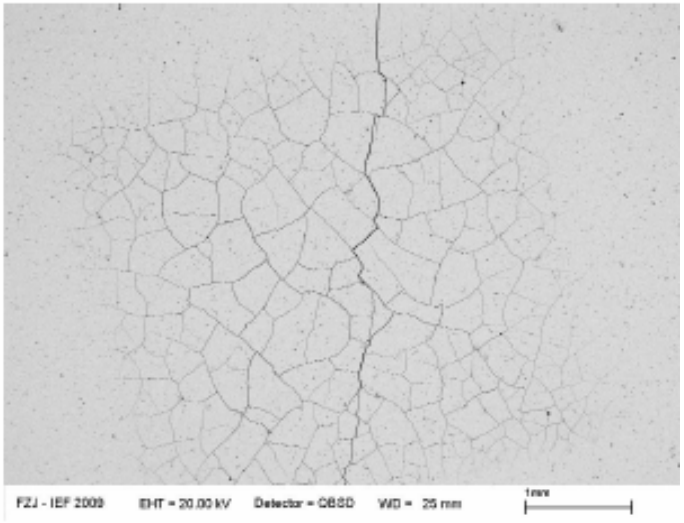
Cracks occurred at all heat flux !!



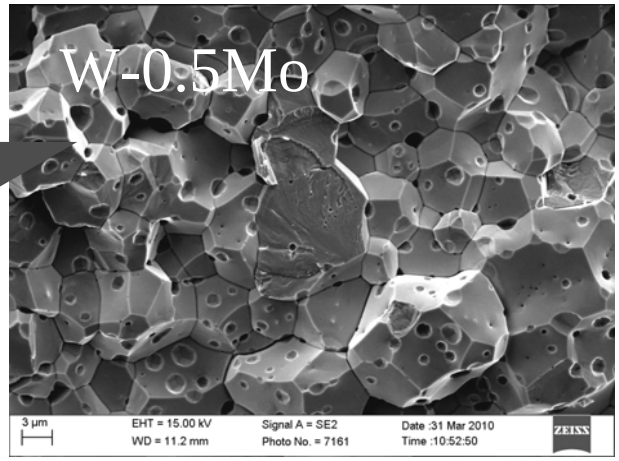
0.9 GW/m², 5 ms, 1 x

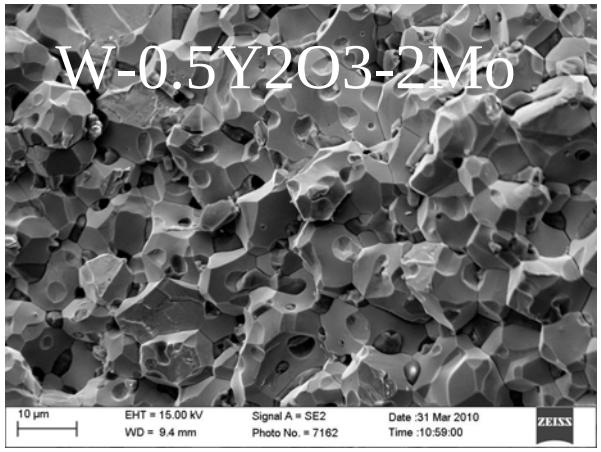


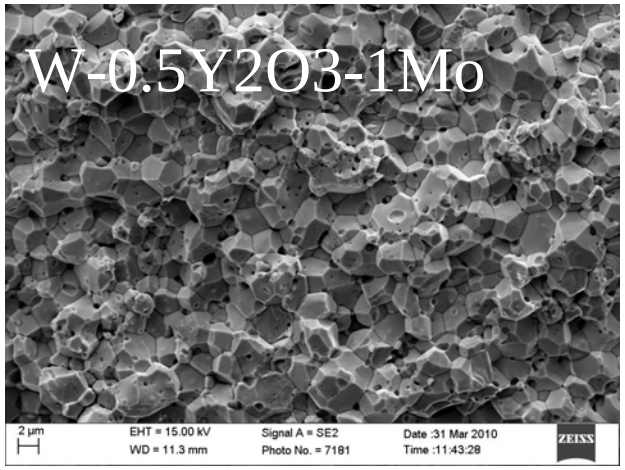
0.9 GW/m², 1 ms, 100 x

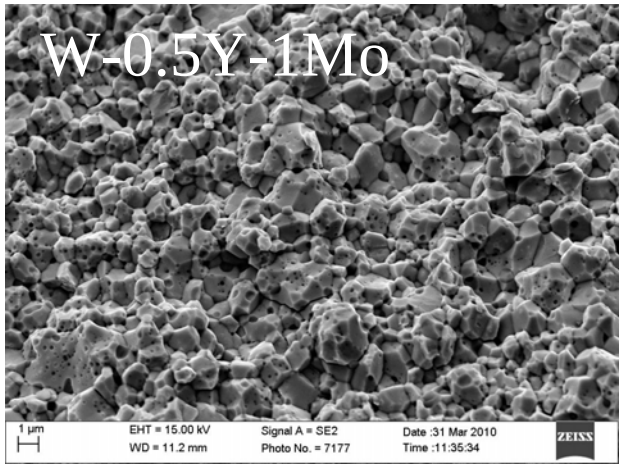


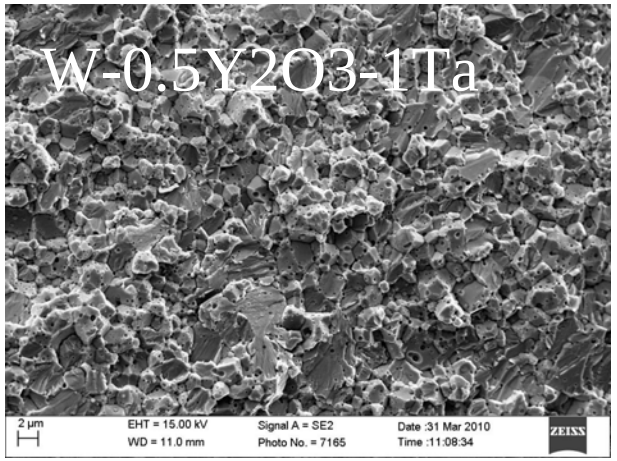
5. Ti→Mo(Ta)











	W	W-2Mo	W-0.5Y-1Mo	W-0.5Y2O3-1Mo	W-0.5Y2O3-2Mo	W-0.5Y2O3-1Ta
RD, %	96.4	96.7	99.1	95.9%	97.2	98.9
BS, MPa	707	669.3	850.16	996.7	1273.7	1061.6
HV	434	355.5	533.8	414.7	433	492.7

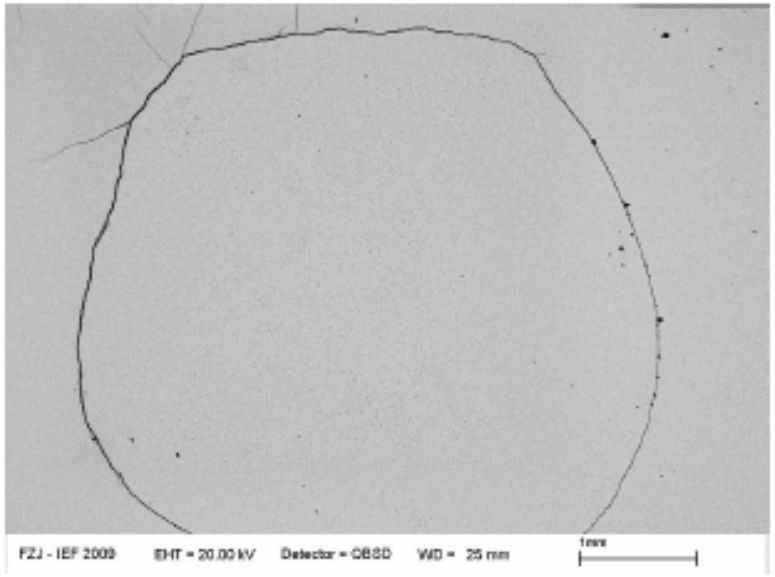
Transient HHF test by electron beam

a 0.3GWm^{-2} Δ t=5 ms, 1X
b 0.6GWm^{-2}, Δt=5 ms, 1X
c 0.9 GWm^{-2} Δt=5 ms, 1X
d 0.9 GWm^{-2} Δt=1 ms, 100X
e 1.2 GWm^{-2} Δt=5 ms, 1X

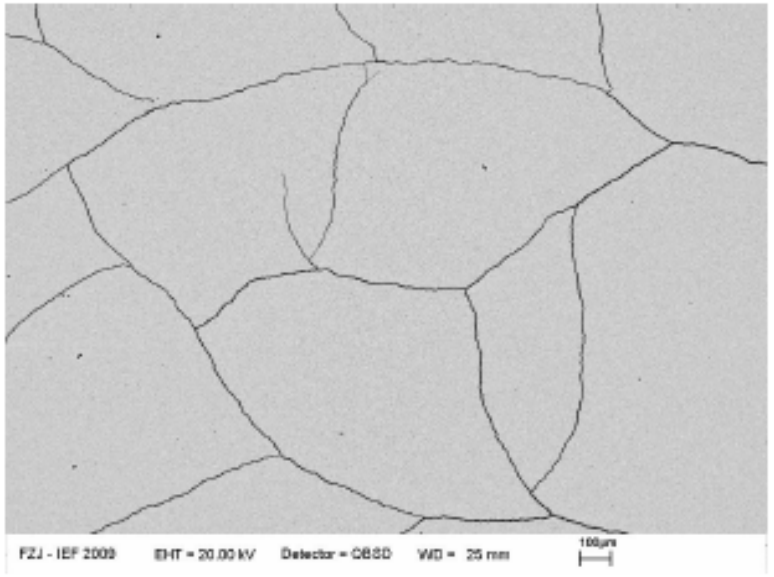
At RT

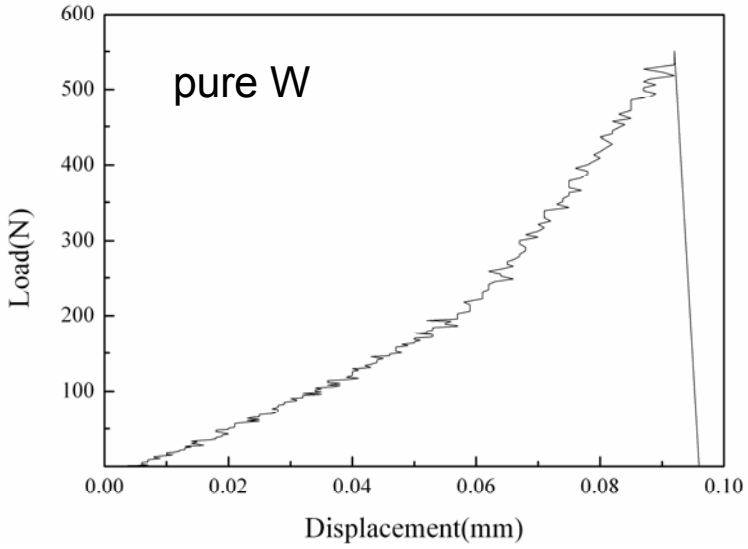
No cracks occurred at 0.3 and 0.6 GWm^{-2} !

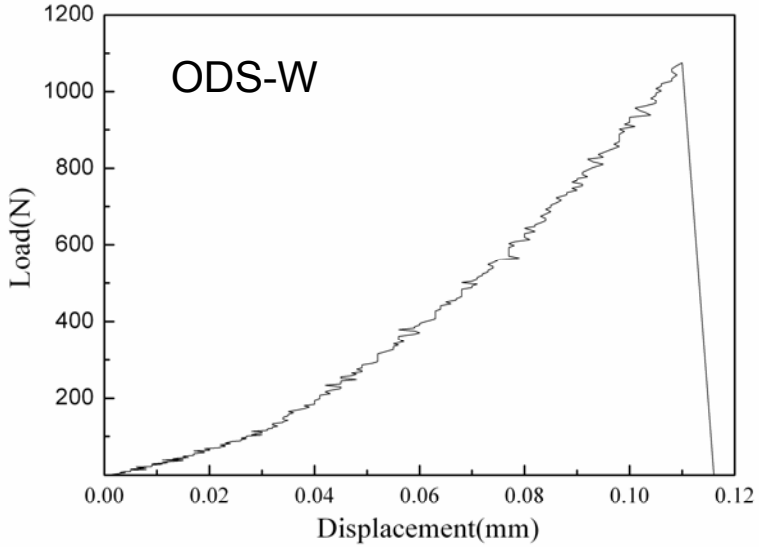
0.9 GW/m^2, 5 ms, 1 x

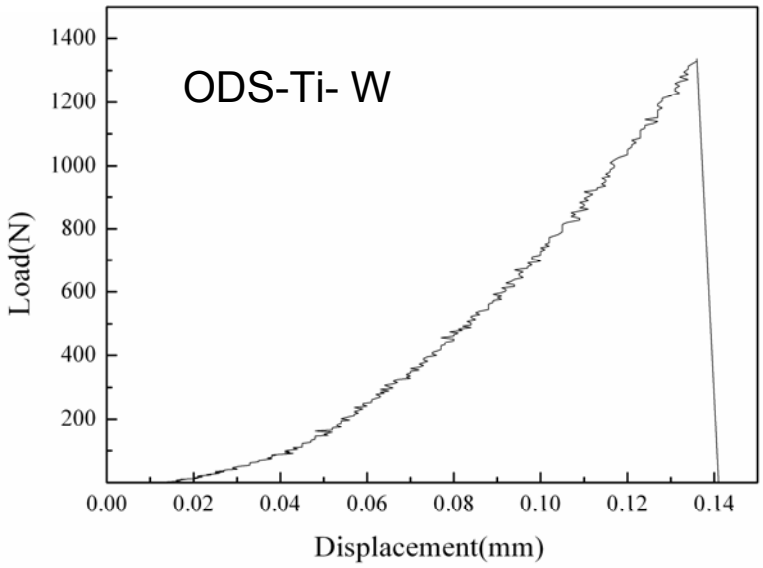


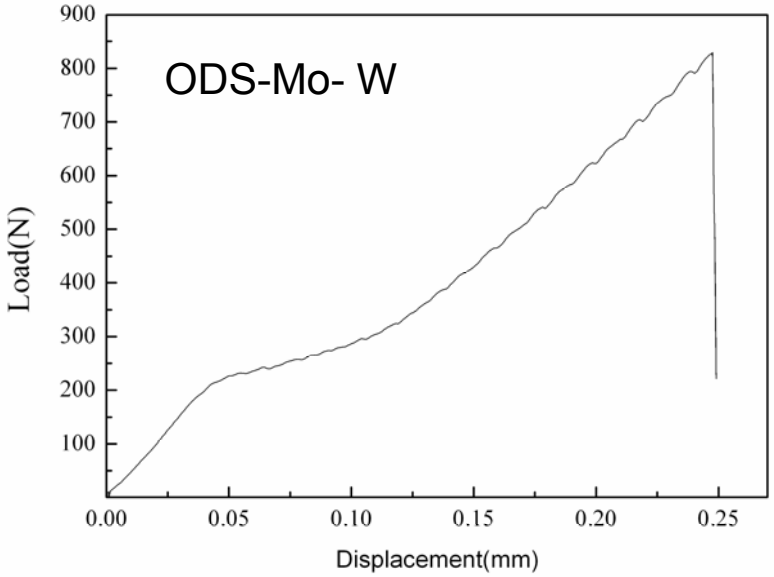
0.9 GW/m^2, 1 ms, 100 x




pure W
Load(N)
Displacement(mm)
600
500
400
300
200
100
0.00
0.02
0.04
0.06
0.08
0.10


ODS-W
Load(N)
Displacement(mm)
1200
1000
800
600
400
200
0.00
0.02
0.04
0.06
0.08
0.10
0.12


ODS-Ti- W
Load(N)
Displacement(mm)
1400
1200
1000
800
600
400
200
0.00
0.02
0.04
0.06
0.08
0.10
0.12
0.14


ODS-Mo- W
Load(N)
Displacement(mm)
900
800
700
600
500
400
300
200
100
0.00
0.05
0.10
0.15
0.20
0.25

6. conclusions

- ODS-W samples with grain sizes from the sub-micron range to several microns have been fabricated by MA-SPS. The addition of Ti will increase the densification and refine the grain size of W significantly. But when the content of Ti is higher than 2 wt. %, the grain size of W will coarsen and deteriorate the hardness and bending strength.
- The effect of Mo on the densification and refinement of the grain size of W is much less compared with Ti. However, the transient high heat loading performance of W-0.5 wt. % Y2O3-1 wt. % Mo was much better than that of W-0.5 wt. % Y2O3- 1 wt. % Ti with a power density cracking threshold that is at least 2-3 times higher.

- Next step works:;
- TEM microstructure investigation.
- Fabrication of samples with larger size for miniature mechanical testing (DBTT, etc).
- Irridation test (H, He, & n) and further THHF test.

The control of impurity during process for new grade W.

The balance of properties for new grade W (two sides):

TC and strength; thermal resisance and irridation resisance.

Thanks for your kind attention!

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