

2022/09/06

**The 13th International Symposium of Advanced Energy Science
~Research Activities on Zero-Emission Energy Network~**

ZE2022C-01

***Evaluation of thermal resistance at the
interface of candidate materials for
fusion reactor divertor***

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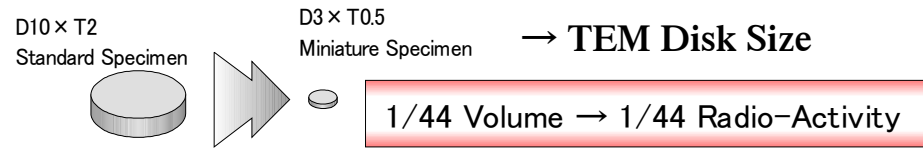
Kiyohiro Yabuuchi (Kyoto Univ.)

Tatsuya Hinoki (Kyoto Univ.)

Miniature specimens

Small specimen for thermal diffusivity measurement is strongly required to **reduce radio activity** and volumetric heating during the neutron irradiation.

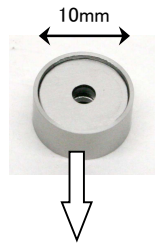
PHENIX Project: 2013–2018 **6Year**
PIE cannot wait the cooling.



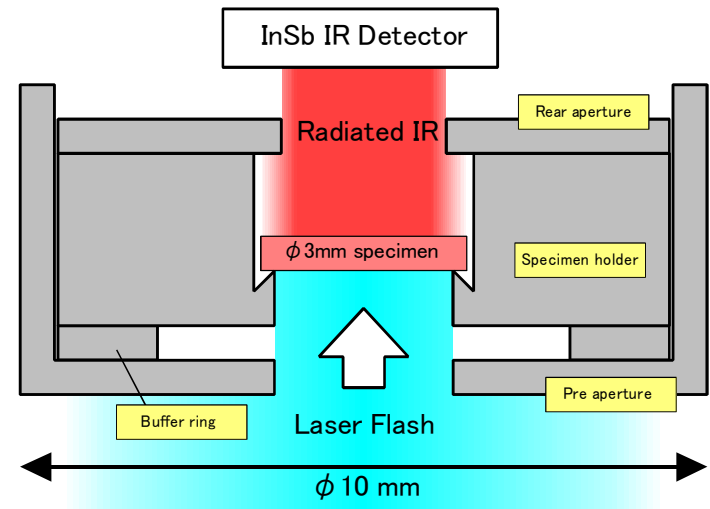
In the **PHENIX project**, irradiation in HFIR have been performed with the specimen form of **Diameter 3mm × Thickness 0.5mm (D3TH)** for thermal diffusivity measurement. Specially manufactured specimen holders enable the measurement of this **D3TH miniature specimens**.



D3 Specimen holder manufactured by Mo-TEM alloy.

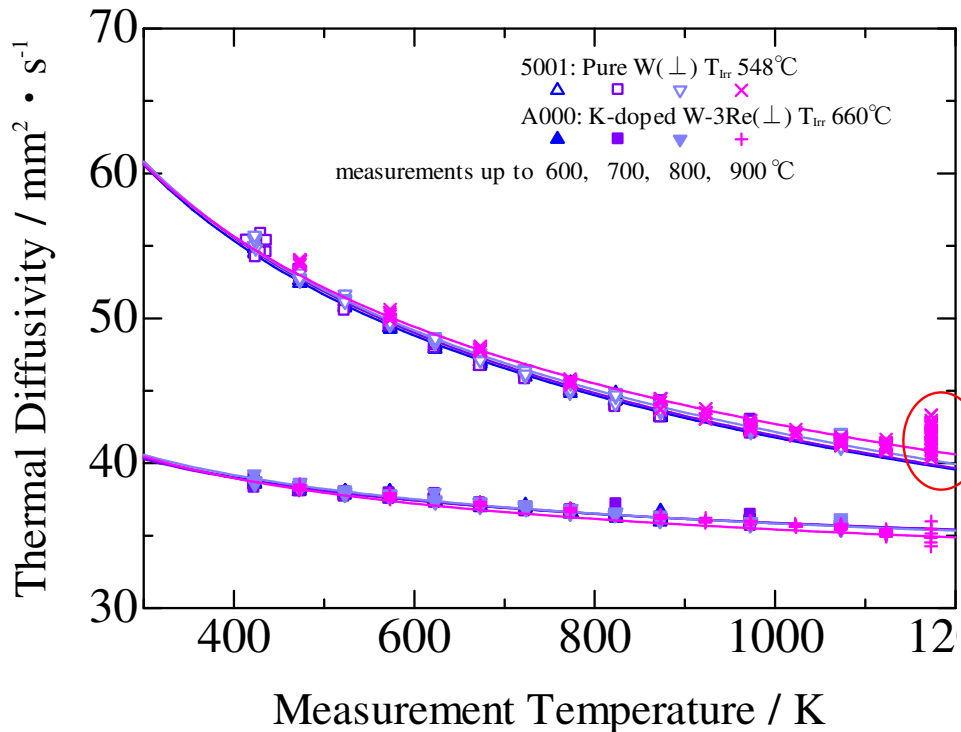


Netzsch LFA-467HT
 Standard Specimen Holder
 (For 12.7mm specimen,
 with 10mm Conversion ring)



Oscillation problem at high temperature

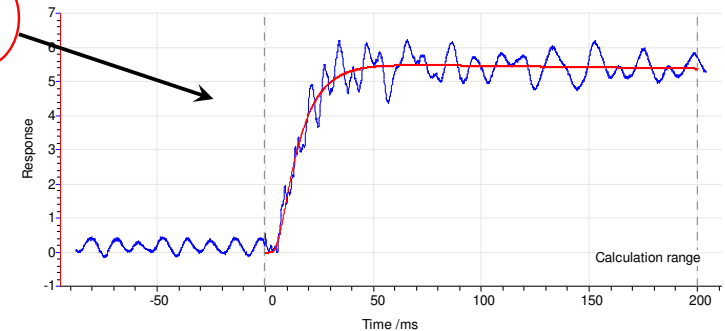
The annealing effect is estimated by isochronal annealing procedure. In addition, if there is some annealing recovery, isothermal annealing behavior is evaluated from the measurement performed in every 1min at aimed annealing temperature for 1h.



- The annealing at 800°C showed no recovery on the specimens irradiated at 548°C or 660°C .

- The measurement at 900°C showed cyclic noise on the IR signals, that is said the **Oscillation problem**.

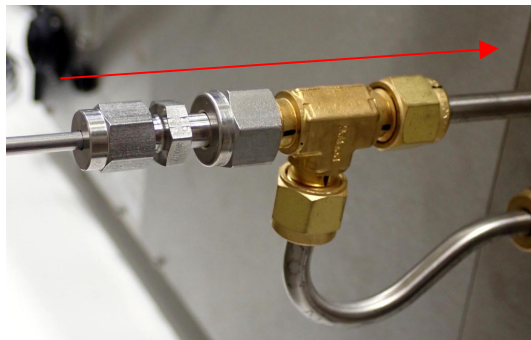
Therefore, we stop the annealing procedure on the irradiated specimens.



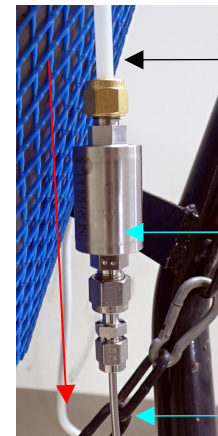
Ultra High Purity Compact Gas Purifier

GP-05/-Ar-Ar-02SW was attached to LFA-467HT at Uji.
(Pureron Japan, gas flow:0.04~0.2L/min)
<http://www.pureron.co.jp/english/product/gp/index.html>
In addition, ultra high purity Ar gas was used to purge.

The graphene coating was oxidized and vaporized to CO/CO₂ gas at poor vacuum or low purity inert gas.



To LFA-467HT
Purge2 /
Protective port



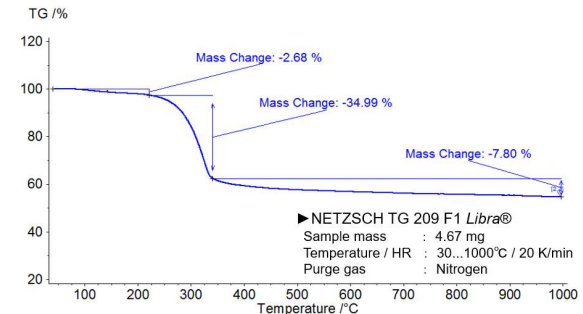
Inlet: 1/4 inch
Teflon tube

GP-05/-Ar-Ar-02SW

Outgo: 1/8 inch
SUS tube

Thermal stability of Graphene nanoplatelets
TG : RT ~1000°C...20 K/min

NETZSCH



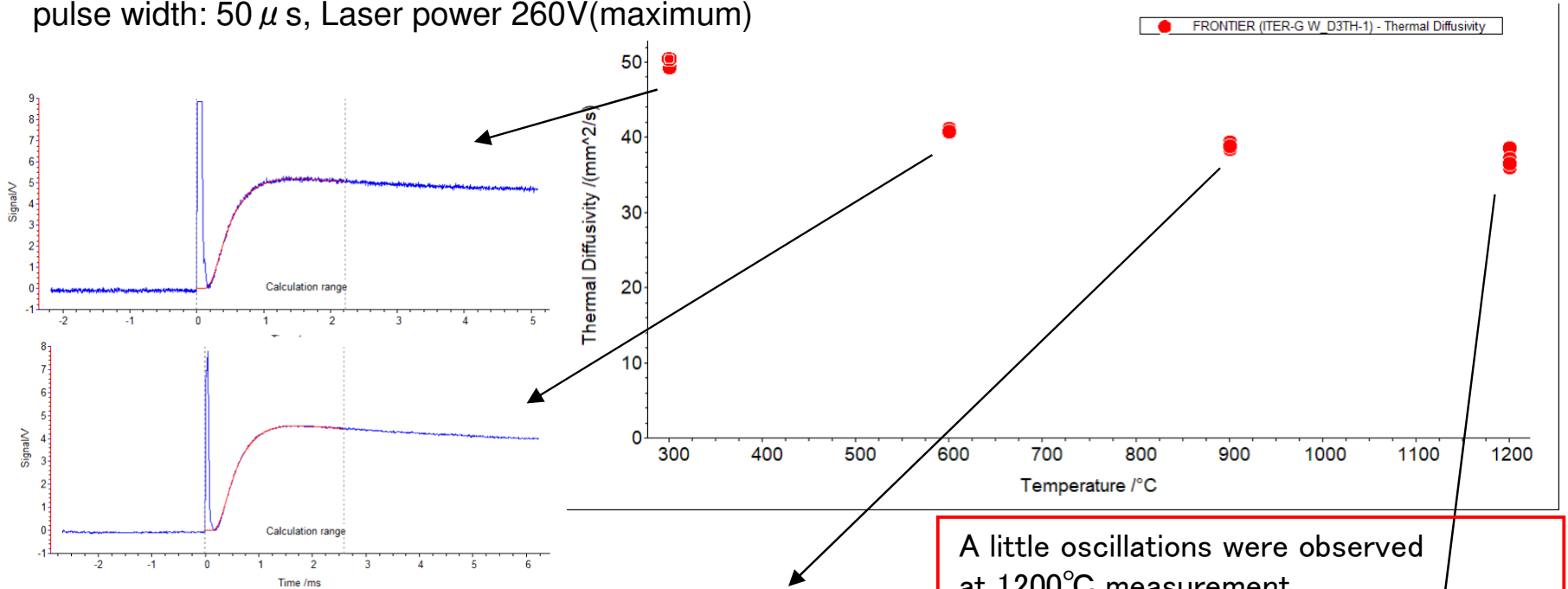
FIX the oscillation at higher temperature



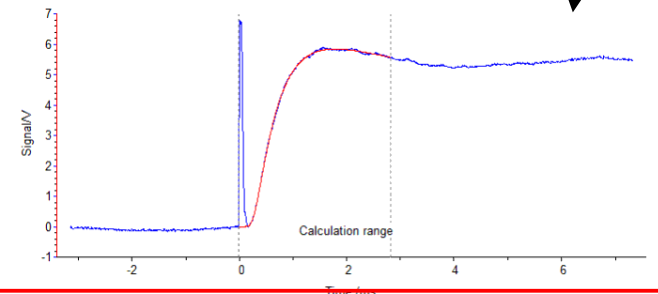
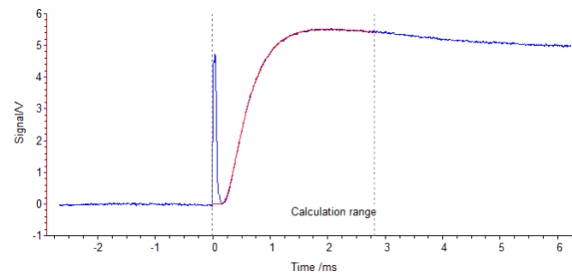
Thick Graphene coat (20 push of spray) on the **D3TH** specimen gives good S/N ratio on IR signals.

Oscillation at high temperature arisen from the oxidation was almost inhibited with the gas purifier.

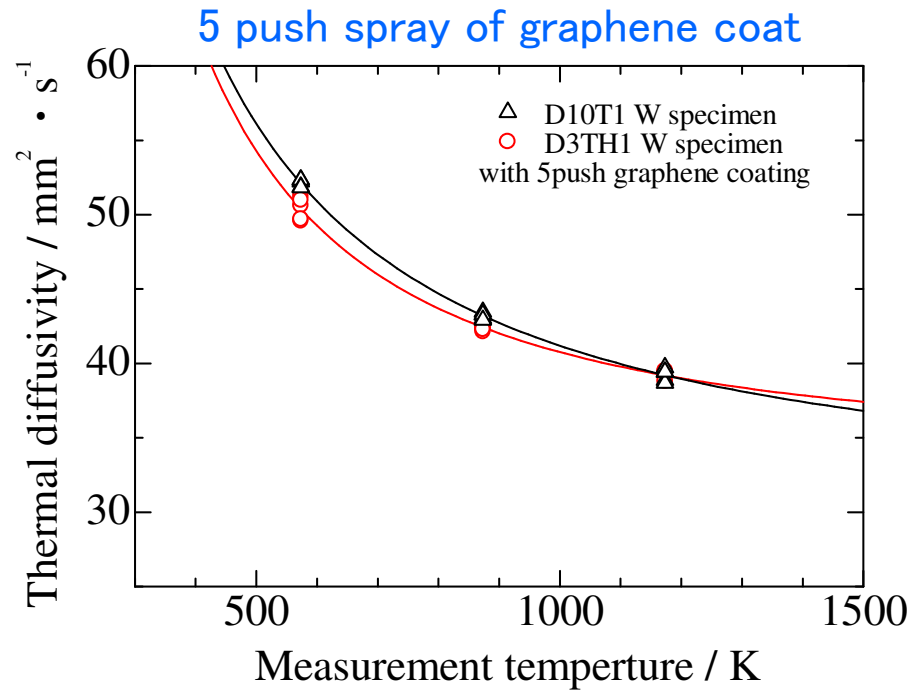
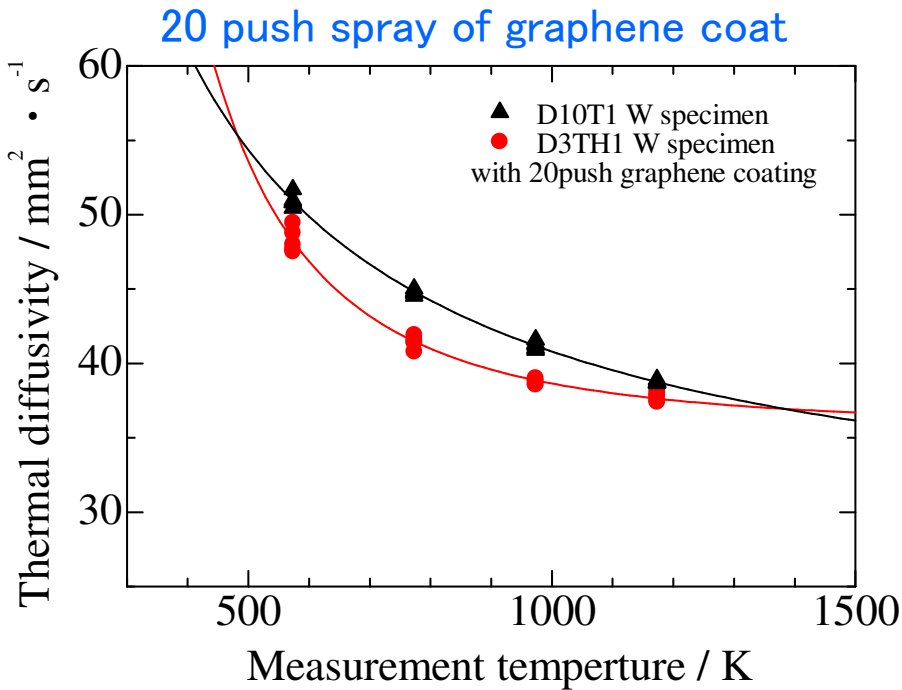
pulse width: $50 \mu\text{s}$, Laser power 260V(maximum)



A little oscillations were observed at 1200°C measurement.

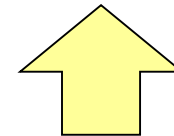


Too thick coating effect



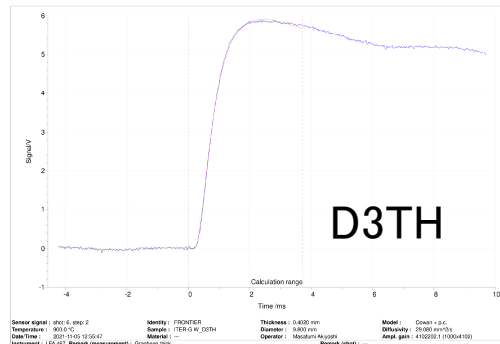
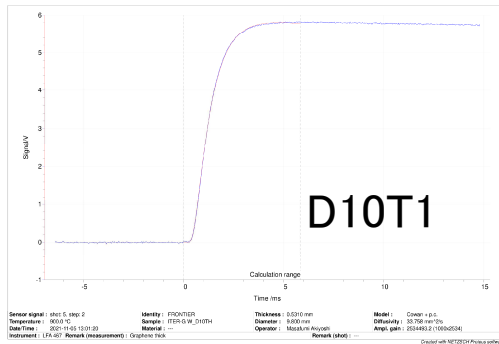
Too thick coated D3TH specimen showed lower thermal diffusivity than the standard specimen.

In the previous study, thin coating specimens showed **low S/N IR curve** with the flash measurement. In addition, at high temperature, these thin coating specimens showed **oscillation from low temperature**.



How to achieve this measurement?

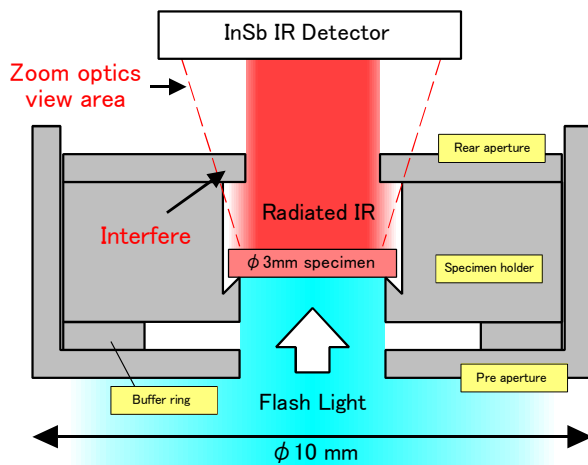
Effect of the D3 specimen holder



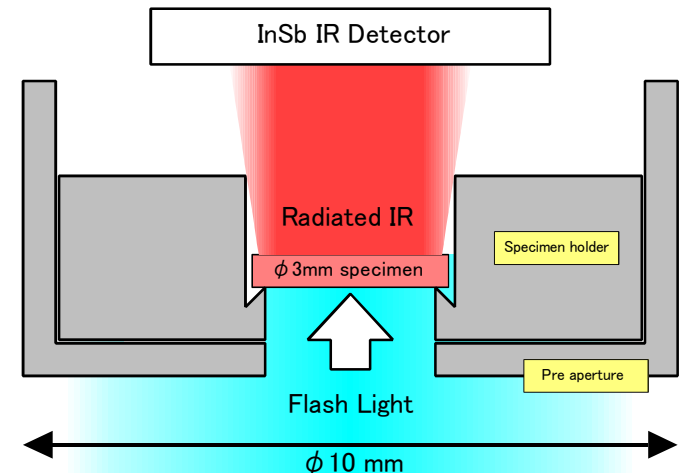
The Netzsch LFA-467HT have the **zoom optics system** at infrared measurement detector on the back of the sample that enable to measure small specimen.

Measurements at 900°C on D10T1 and D3TH W specimens with 20 push graphene coating showed different IR curve **even the detective area of the zoom optics was set to D2.8 mm for both specimens.**

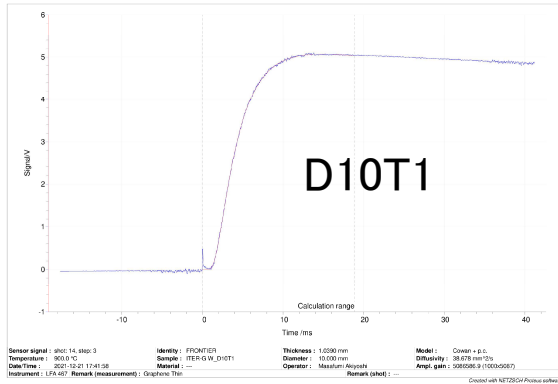
The reason for this difference was that this **zoom optics system** and the **special specimen holder for the D3 sample interfered with each other**, resulting in a decrease in the amount of infrared light.



Remove the buffer ring and the rear aperture



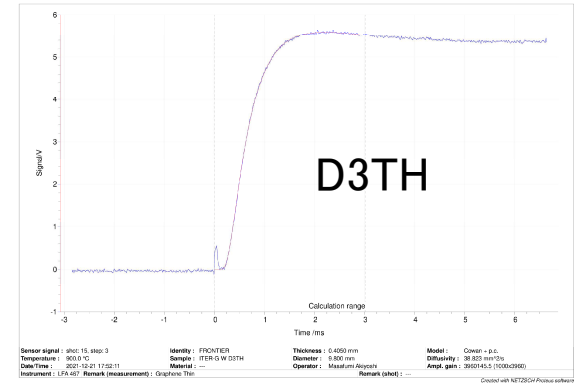
Final fix the oscillation problem



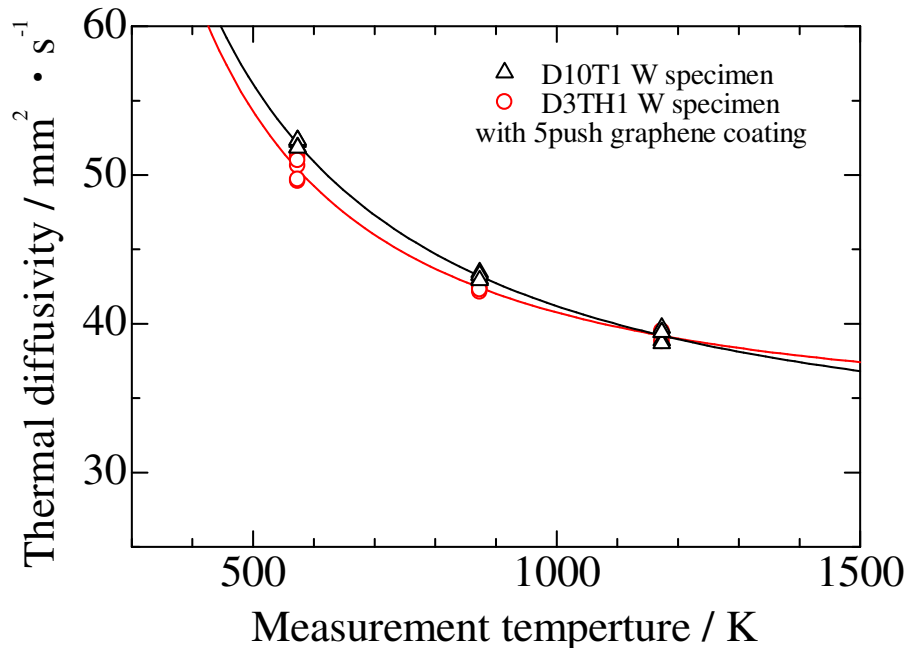
Pure W specimens
with 5push graphene
coating

Measured at 900°C

Using gas purifier



Using D3 specimen holder
without the buffer ring and
the rear aperture.



**D3TH specimen showed
good agreement with
D10T1 specimen and
showed no oscillation.**