JP11.00098

Detection of trace rhenium in tungsten by laser-induced breakdown spectroscopy

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Background

- Tungsten is useful as a PFM.(high melting point, high thermal conductivity)
- When absorbing neutrons, tungsten changes to rhenium after beta decay.
- In order to safely operate the fusion reactor, it is necessary to evaluate the neutron irradiation dose.

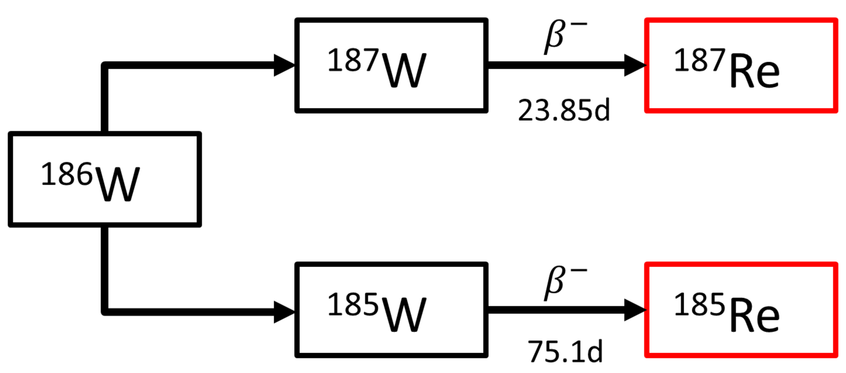

Fig.1. Reaction chains of ^{186}W[1].

Table1. Power-plant transmutation response summary for W[2].

Starting composition (at%)	Transmutation products	Concentration of product in bracket(at%) after irradiating for	
		1year	5year
W(100)	W	98.9	94.0
	Re	0.91	3.80
	Os	0.06	1.38

- **The neutron irradiation dose can be estimated from trace rhenium in tungsten**

Purpose

- **Detection of trace amount of rhenium in tungsten by LIBS**
  -Neutron irradiation dose is evaluated from the composition ratio of rhenium in tungsten.

Conclusion

- **In situ diagnostic method using CF-LIBS is suitable for the environment like fusion reactor.**
- **Detecting trace amount of rhenium in tungsten was achieved by CF-LIBS method.**
- **Improvement of accuracy and sensitivity and investigation of detection limit are necessary.**

Experimental

Set up

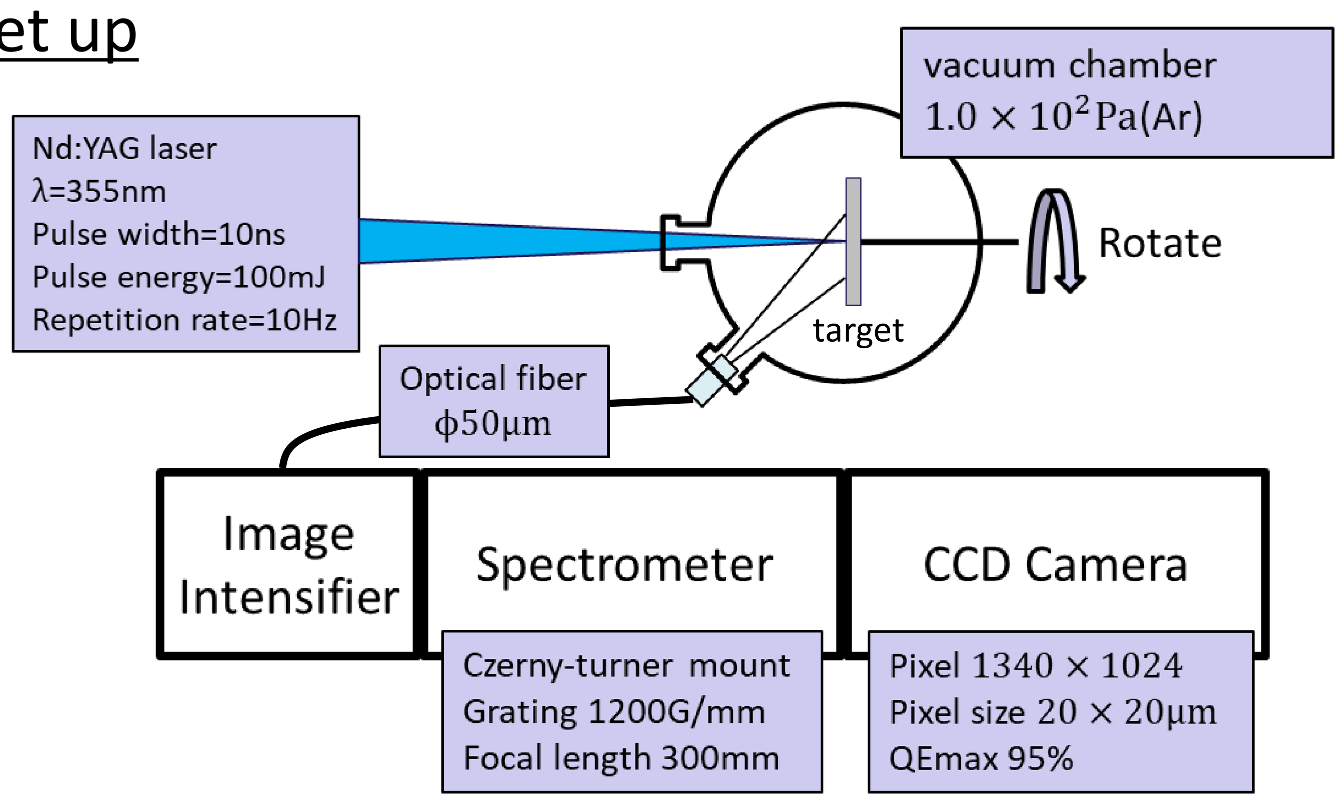

Fig.2. Experimental set up

- Nd:YAG laser is used for generating plasma.
- Emitted light is transmitted by fiber optic cable and observed through the spectrometer.

CF-LIBS method

Ideal intensity

$$I_\lambda^{ki} = N_s A_{ki} \frac{g_k e^{-(E_k/k_BT)}}{U_s(T)} \cdots (1)$$

Observed intensity

$$\bar{I}_\lambda^{ki} = F C_s A_{ki} \frac{g_k e^{-(E_k/k_BT)}}{U_s(T)} \cdots (2)$$

From NIST database

Boltzmann plot

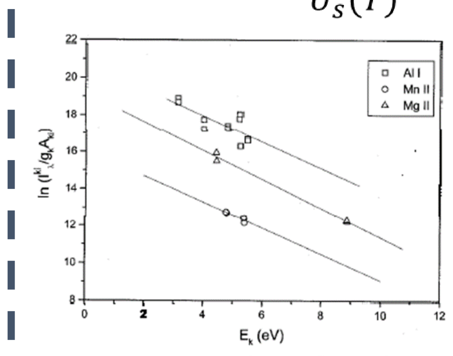

Fig.3. Example of Boltzmann plot[3].

I_λ^{ki}:ideal intensity
$\bar{I}_\lambda^{ki}$: measured intensity
F:experimental parameter
λ: wavelength
N_s: density
C_s: concentration
A_{ki}: transition probability
g_k: k level statistcal weight
K_B:Boltzmann constant
T: plasma temperature
$U_s(T)$:partition function
q_s: intercept

$$C_s = \frac{U_s(T)}{F} e^{q_s} \cdots (3) \qquad \sum_s C_s = \frac{1}{F} \sum_s U_s(T) e^{q_s} = 1 \cdots (4)$$

F factor can be determined

- The line slope corresponds to the T_e.
- F factor is determined by normalization of concentration.
- Drawing calibration curve for each concentration is not necessary.

Continuous spectra removal

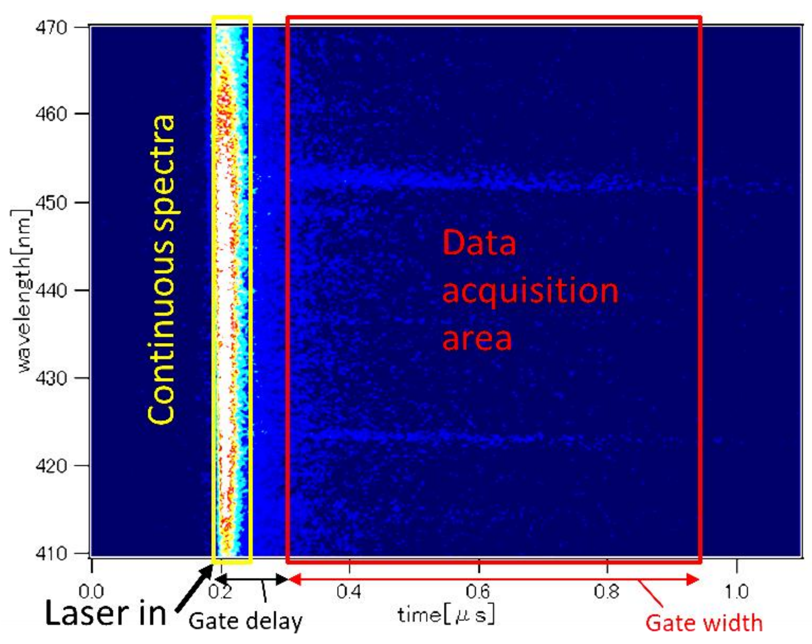

Fig.4. Streak camera image

- To remove continuous spectra, the gated detector is used.
- In our Experiments, gate width is 2μs, and gate delay is 200ns.

Results & Discussion

Observed spectra

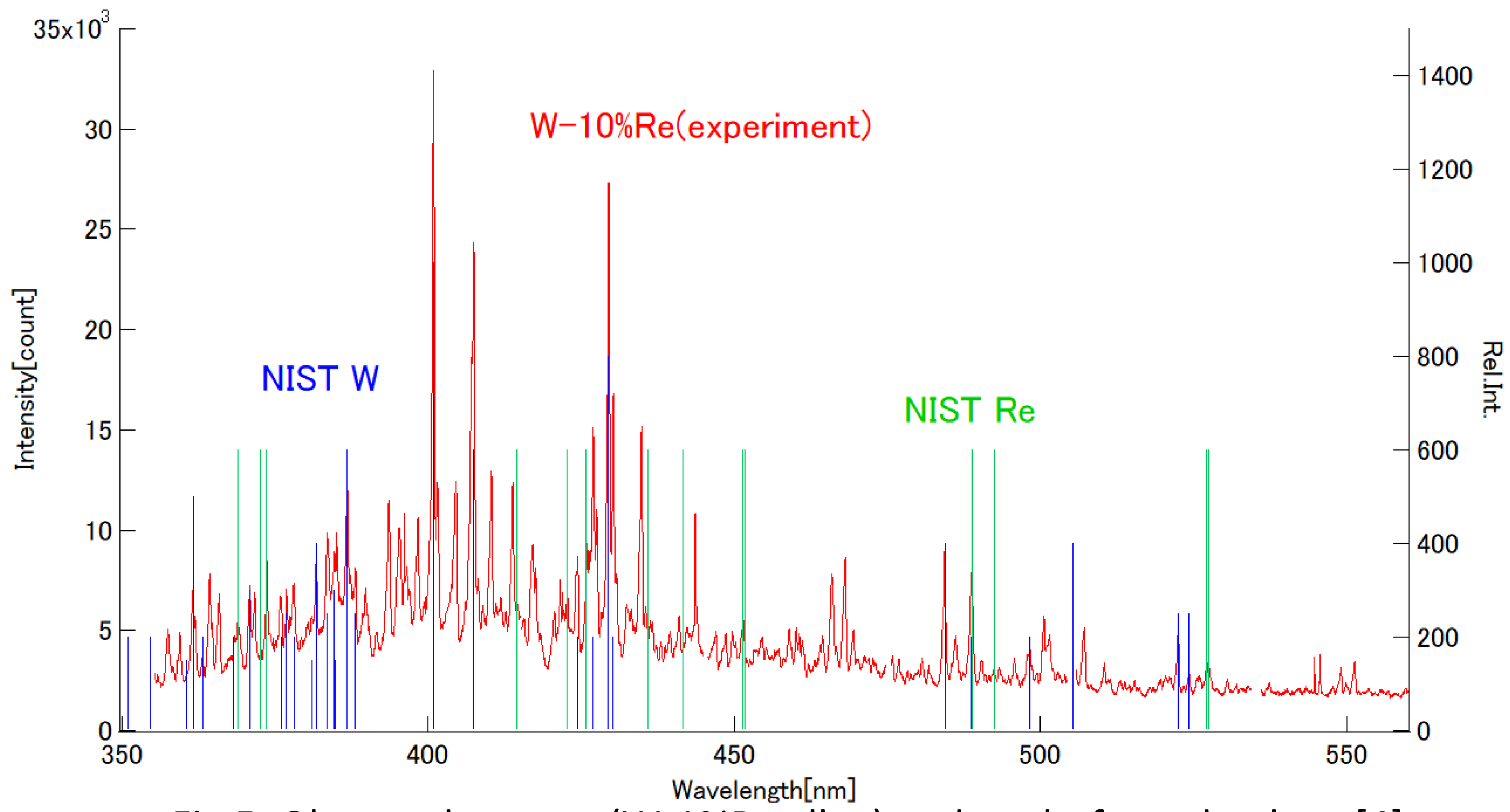

Fig.5. Observed spectra (W-1%Re alloy) and peaks from database[4].

Table2. Peak information[4][5].

species	Wavelength [nm]	Transition probability [1/s]	Upper level [eV]	Lower level [eV]	g_k	g_i
W I	400.875	8.3e+5	3.221556	0.000000	9	7
W I	407.436	1.00E+7	3.408091	0.365913	7	7
W I	429.461	3.04E+6	3.252077	0.365913	5	7
W I	488.670	8.1E+5	3.307462	0.771099	11	9
Re I	368.951	1.96E+8	5.122162	1.762666	6	8
Re I	373.531	1.15E+6	6.248273	2.929972	10	10
Re I	488.9136	1.15E+6	2.535204	0	8	6
Re I	527.1001	1.47E+8	5.281512	2.929972	8	10
Re I	527.5549	3.03E+5	2.349513	0	6	6

- Each peak well matches the NIST database.

Re concentration measurement

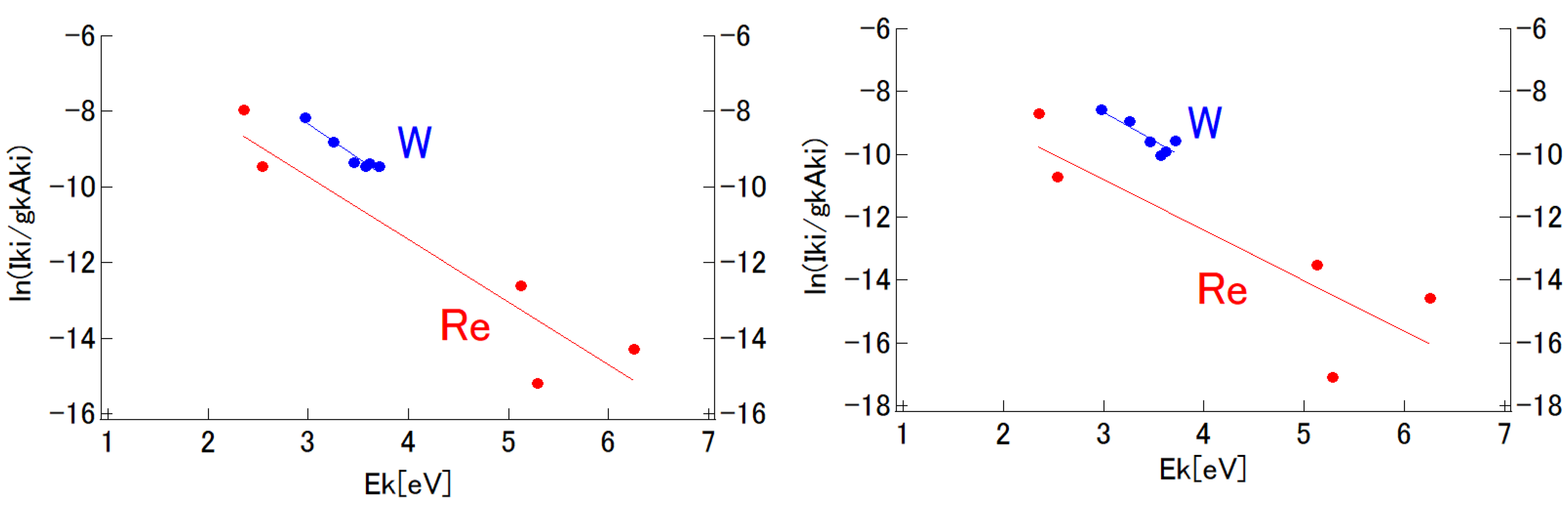

Fig.6. Boltzmann plot (left W-10%Re, right W-1%Re)

Table3. Result of CF-LIBS

sample	Electron temperature[eV]	Measured rhenium concentration[at%]
W-10%Re	0.55±0.07	8.3
W-1%Re	0.58±0.14	3.1

- Dividing the data acquisition into several times may affect the data variation.
- Improvement of accuracy and investigation of detection limit are necessary

SEM & EDS

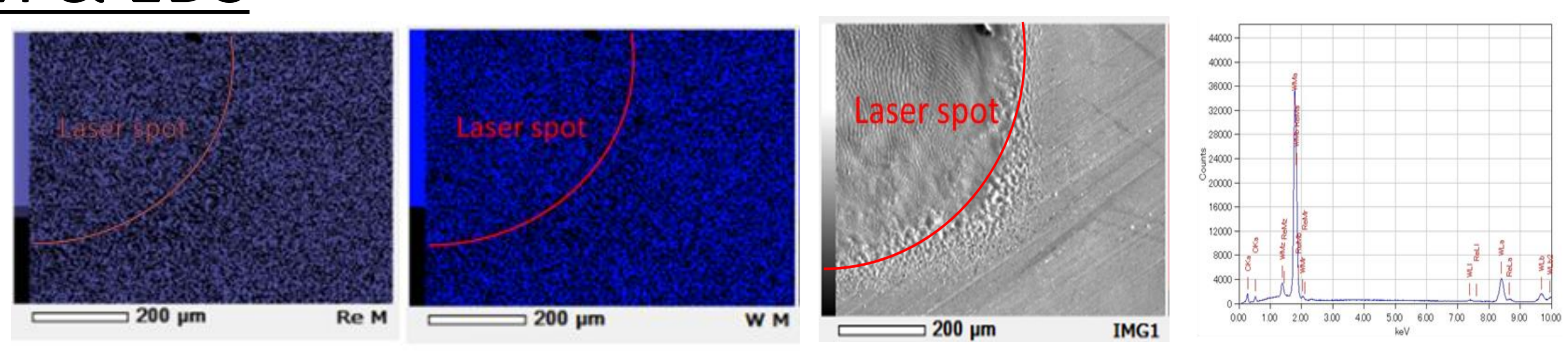

Fig.7. SEM image & EDS analysis

- No difference in composition ratio was observed between the laser irradiated part and the non-irradiated part.
  - ➢ the composition ratio of the sample and the generated plasma is equal.

Table4. Result of CF-LIBS & EDS

sample	CF-LIBS(at%)	EDS(at%)
1%Re	3.1	1.7±0.86
10%Re	8.3	9.0±0.31

Reference

[1] T. Noda et al. Journal of Nuclear Materials 258-263 (1998) 934-939
[2] M.R.Gilbert et al. Nucl. Fusion 51 (2011) 043005
[3]A.CIUCCI, M.CORSI, et al. Applied Spectroscopy, vol. 53, issue 8, pp. 960-964
[4] https://www.nist.gov/pml/atomic-spectra-database
[5] https://www.cfa.harvard.edu/amp/ampdata/kurucz23/sekur.html

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『60th Annual Meeting of the APS Division of Plasma Physics』, Monday-Friday, November 5-9, 2018