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Fakulta matematiky, fyziky a informatiky

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Autoreferát dizertačnej práce

Investigation of materials suitable for the walls of the divertor of the fusion reactor using method LIBS

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v odbore doktorandského štúdia:

Kvantová elektronika a optika

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Kvantová elektronika a optika
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Abstract

The presented work deals with the method of Laser Induced Breakdown Spectroscopy (LIBS) and its use in the analysis of plasma facing components within the fusion devices, mainly within the divertor of the fusion reactor.

The thesis is divided into the theoretical and experimental part. Theoretical part summarizes the basic knowledge about plasma, method of Laser Induced Breakdown Spectroscopy as well as possibilities of using the LIBS method for the analysis of studied sample (qualitative and quantitative analysis) and the basic information about the thermonuclear fusion. Connection between the theoretical and experimental part represents the part which deals with the using method LIBS in thermonuclear fusion as well as analysis of current knowledge of the issue.

The experimental part is divided into three parts. The first describes the experimental apparatus. The other two sections are focused on experimental measurements themselves and the presentation of achieved results. During the experimental measurements two types of samples were analysed - lithium- based alloy (sample of *LiSn*) and tungsten- based alloys.

Keywords: Laser induced- breakdown spectroscopy (LIBS), Calibration-Free Laser induced-breakdown spectroscopy (CF LIBS) thermonuclear fusion, plasma wall interaction, *LiSn* alloy, tungsten, tungsten-based alloys, deuterium, fuel retention, depth profile, ablation rate

Abstrakt

Predkladaná práca sa zaoberá metódou Laserom indukovanej iskry (LIBS) a jej využitím pri analýze komponentov, ktoré prichádzajú do styku s plazmou v rámci fúznych zariadení, predovšetkým v rámci divertora fúzneho reaktora.
Práca je rozdelená na teoretickú a experimentálnu časť. V rámci teoretickej časti sú zrhnuté základné poznatky o plazme, o metóde laserom indukovanej plazmy ako aj možnosti využitia metódy LIBS pri analýze skúmanej vzorky (kvalitatívna a kvantitatívna analýza) a základné infromácie o termojadrovej fúzii. Prepojenie medzi teoretickou a experimentálnou časťou tejto práce predstavuje časť, ktorá sa zaoberá využitím metódy LIBS v rámci termojadrovej fúzie a rozborom aktuálneho stavu tejto problematiky.
Experimentálna časť, je rozdelená na tri časti. Prvá z nich predstavuje opis experimentálnej aparatúry. Ďalšie dve časti sú zamerané na samotné experimentálne merania a prezentáciu dosiahnutých výsledkov. V rámci experimentálnych meraní boli analyzované dva typy vzoriek- zliatina založené na báze lítia (vzorka *LiSn*) a zliatiny založené na báze volfrámu.

Kľúčové slová: Spektroskopia laser indukovanej iskry (LIBS), Bezkalibračná metóda spektroskopie laserom indukovanej iskry, termojadrová fúzia, interakcie plazmy so stenami termojadrového reaktora, zliatina *LiSn*, volfrám, zliatiny na báze volfrámu, deutérium, záchyt paliva, hĺbkový profil, ablačná rýchlosť

Introduction

The electric energy represents the basic kind of the energy which is nowadays necessary for existence and for life. At present the world is dependent on the electricity produced primarily from fossil fuels (oil, coal, natural gas) and nuclear power plants. As world energy consumption is still growing and sources of fossil fuel are inadequate, it is necessary to develop new energy sources that are safe and environmentally friendly. One such option is a thermonuclear fusion, which has enormous potential to affect global energy production.

Thermonuclear fusion is process which occurs at high temperature ($\approx$*15 million* $^\circ$ *C* and at the huge pressure inside the Sun. At such high temperatures, the gas becomes plasma. Nowadays, scientific teams are working on the reproduction of the process of thermonuclear fusion on Earth. The process of thermonuclear fusion in such conditions requires two heavier isotopes of hydrogen- deuterium and tritium. During the fusion reaction the plasma facing components are exposed to high temperatures and high energy flows of the particles which come from plasma. It leads to wall erosion and the creation of the layers and fuel retention (deuterium, tritium) in the inner part of the fusion reactors. These factors can shorten the lifetime of fusion devices. Therefore the experimental monitoring of plasma facing components is necessary. One of diagnostic method which is suitable for this monitoring is the method of Laser Induced Breakdown Spectroscopy.

Laser Induced Breakdown Spectroscopy (LIBS) is the method of atomic emission spectroscopy, which has a wide range of application. The analysis of the sample using method LIBS allows to obtain complete information about the qualitative and quantitative composition of the studied sample.

The presented work deals with the using of method LIBS for the analysis of the fusion relevant wall materials. During the analysis, two types of the alloys were studied: lithium-based alloy and tungsten-based alloys.

Experimental part

Lithium-based alloy

Lithium-based alloys seem to be a suitable material for the wall of the fusion devices. The popularity of using this material is increasing due to the fact that the wall remains liquid under operation. The advantage is that the liquid state prevents the creation nano-cracks and local defects and also that the retained fuel in the walls can be easily removed by heating.

The measurements of the Lithium-based alloy were carried out at the Faculty of Mathematics, Physics and Informatics, Comenius University in Bratislava at the Department of Experimental Physics in the laboratory of Prof. Dr. P. Veis, Phd. LIBS experimental setup is shown in Figure 1.

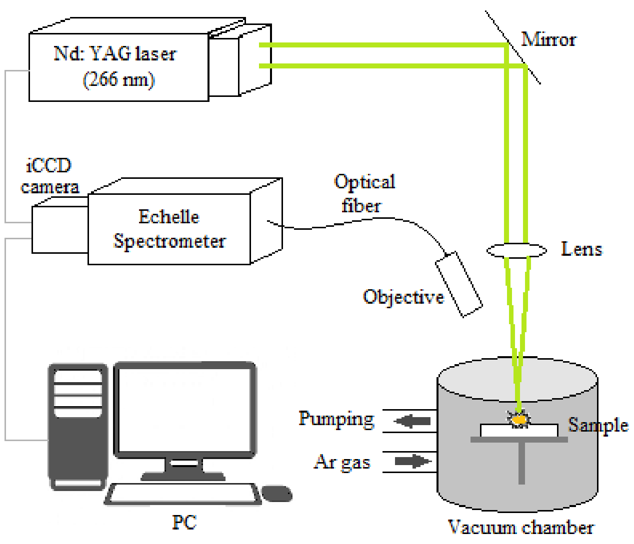

Figure 1: Schematic experimental setup used in our experiment, which contains of laser, mirrors, lens, vacuum chamber with sample and pumping/inserting gas windows, fibre holder (objective), fibre and Echelle spectrometer.

The analysed sample was the alloy of lithium-tin (*Li- 20* % and *Sn- 80* %, from Princeton Corporation). The analysis consisted from two parts.

In first one, the different experimental conditions were used and the CF LIBS approach was applied for the identification of the elemental composition in the studied sample. The analysis were performed under three different pressures (*130, 1 300 and 13 000 Pa*) in argon atmosphere. At each pressure the different gate delay and gate width parameters were used (*100, 300* and *500 ns*). The gate delay and spectral gate width were kept to an identical value. The measured spectra were recorded as an accumulation of *200* laser shots.

In the second part of the experiments, the sample was broken in order to expose its inner structure. The site of this defect is referred to as "the inner part". So in addition of the measurements of surface of the sample, the measurements of the inner part were performed. The measurements were carried out under the low pressure (1 330 Pa) at argon atmosphere. The gate delay and spectral gate width delay were kept to an identical value, *300 ns*. The measurements consisted from *20* laser shots to the both of those location types of which first three were analysed. These measurements show the structure of the sample in different depth.

Tungsten and tungsten-based alloys

Tungsten is material with high melting temperature, low degree of erosion and low ability of retention of light elements (*D, T, He*). Due to this properties, its represents material suitable for plasma facing components in fusion devices. In ITER reactor tungsten will be used for the divertor.

The analysis of tungsten and tungsten-based alloys can be also divided in two main parts. The first part of experiments was carried out in the laboratory of Prof. Dr. P. Veis, Phd. at the Faculty of Mathematics, Physics and Informatics, Comenius University in Bratislava. Experimental apparatus is shown in Figure 1. During these measurements the several tungsten-based samples were studied. Pure tungsten, the layer of the tungsten (*250 nm*) deposited on silicon substrate (prepared by FÚ SAV, Dr. Šifalovič) and mixture of tungsten - aluminium (*W- 80* %, *Al- 20* %) deposited on silicon substrate (prepared within the WPPFC Eurofusion project) were analysed. The first set of measurements were done under the different pressures (atmospheric pressure, *6 kPa* and 130 Pa) and different gate delay and gate width conditions (*100 ns, 200 ns, 500 ns, 1 μs, 2 μs, 5 μs* and *10 μs*) The spectra were recorded as accumulation of *100* laser shots. Subsequently, the measurements of tungsten-based samples with protective carbon layer (*1 μm*) were performed under the different experimental conditions- pressure (*1 330 Pa*), gate delay and gate width parameters (*100, 500 ns*). The second part of the analysis of tungsten-based samples includes the analysis of layers of *AlW* and *BeW* (*Al / Be 67* %, *W- 33*%) without or with deuterium deposition applied on the molybdenum substrate (prepared in Romania, INFLPR). The measurements were carried out using beryllium-compatibile LIBS set-up at VTT Technical Research Centre of Finland. The experimental apparatus is shown in Figure 2.

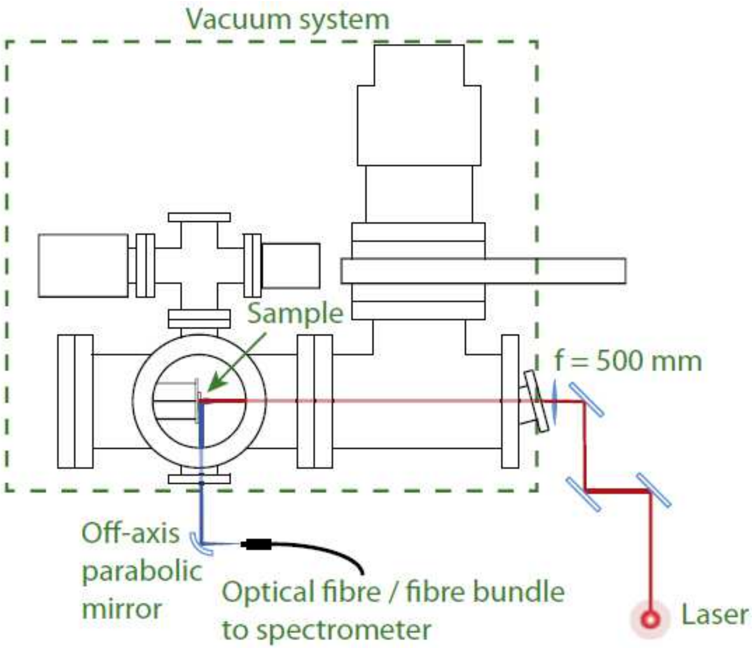

Figure 2: Experimental apparatus contains of laser, vacuum system and optical components at VTT Technical Research Centre of Finland.

The measurements were performed under the low pressure- at vacuum (of the order of 10^{-4} *Pa* in argon atmosphere, in four spectral range- *395, 450, 608* and *656 nm* for *AlW/AlWD* samples and *395, 450, 518* and *656 nm* for *BeW/BeWD* sample. The gate delay time was set to *80 ns* and gate width was set to *500 ns.* The measured spectra consist of *150* spectra recorded as a single scan in each spectral region with aim to observed the shot to shot spectra evolution.

Results and conclusion

Lithium-based alloy

In first stage of the analysisi of *LiSn* alloy, the measured data were used for the qualitative and quantitative analysis of the sample. The electron density was identified by the Stark broadening of H_α (*656.25 nm*) spectral line and the electron temperature was determined using multi-elemental Saha-Boltzmann plot using the neutral and singly ionized spectral lines of *Sn* and neutral spectral lines of *Li*. The ionic spectral lines of *Li* were not used for the analysis because they were not observed in the measured spectra due to the high energy of the upper levels of these lines. After the determination of plasma parameters (electron density and electron temperature) the CF LIBS approach was applied with the aim of the determination of concentration of the elements contained in the sample. The results of this analysis are listed in Table 1.

p (Pa)	GD (ns)	n_e (cm^3)	T (eV)	c(Sn) (%)	c(Li) (%)
130	100	1.79 E+19	1.42	61	39
	300	1.21 E+18	1.05	70	30
	500	5.13E+17	1.03	70	30
1300	100	7.39 E+18	1.39	62	38
	300	1.08 E+18	1.08	69	31
	500	4.09 E+17	0.96	73	27
1300	100	1.21 E+19	1.55	60	40
	300	3.38 E+18	1.29	64	36
	500	2.22 E+18	1.24	65	35

Table 1: Determined values of T_e, n_e and concentration of the elements for the different settings of the experimental apparatus.

In the second stage of the experiment, the analysis of first three shots of surface and of the inner part of *LiSn* sample was done. The electron density (using the Stark broadening of H_α spectral line) as well as the electron temperature (using the Saha-

Boltzmann plot) were identified and the CF LIBS aprroach was used for the determination of the elemental concentration in different layers of the sample. The results are summarized in Table 2.

		n_e (cm^3)	T (eV)	c(Sn) (%)	c(Li) (%)
Surface	1^{st} shot	5.36 E+17	1.91	58	42
	2^{nd} shot	6.1 E+17	1.53	60	40
	3^{rd} shot	8.05 E+17	1.72	59	41
Inner part	1^{st} shot	7.1 E+17	1.16	67	33
	2^{nd} shot	4.35 E+17	1.31	64	36
	3^{rd} shot	2.93 E+17	1.30	63	37

Table 2: Determined values of T_e, n_e and concentration of the elements for the 1^{st}, 2^{nd} and 3^{rd} shots at the surface and at the inner part of the sample

The concentration demonstrates the heterogeneity of *LiSn* alloys. The differences between CF LIBS evaluated composition and producer composition can be caused by using only neutral spectral lines of *Li* (ionic spectral lines of *Li* were not observed in the measured spectra) in CF LIBS analysis. Further investigation and improvement of accuracy of the determination of *Li* concentration is planning.
The results of these experiments are also presented in publication *"Analysis of LiSn alloy at several depths using LIBS"* in Fusion Engineering and Design [1].

Tungsten and tungsten-based alloys

During the first part of the analysis several samples of tungsten and tungsten-based alloys were studied. Pure tungsten, the layer of the tungsten (*250 nm*) deposited on silicon substrate (prepared by FÚ SAV, Dr. Šifalovič) and mixture of tungsten - aluminium (*W- 80 %, Al- 20 %*) deposited on silicon substrate (prepared within the WPPFC Eurofusion project) were studied. Analysis of these data was based on the determination of electron temperature using Boltzmann plot. The parameters (transition probability and energy of upper level) for the calculation of coordinates of Boltzmann plot were obtained from two databases- Harvard and NIST and the electron temperature was determined for both of them.

		Low pressure		Atmospheric pressure		
Database	Ionization degree	100 ns	200 ns	1 μs	2 μs	5 μs
Harvard	W I	0.52	0.45	0.48	0.4	0.38
	W II	0.98	0.83	0.71	0.68	0.54
NIST	W I	0.84	0.53	0.83	0.57	0.54
	W II	3.70	2.83	1.02	0.89	0.65

Table 3: Sample- pure *W*, determined values of electron temperature T_e by Boltzmann plot using *W I* and *W II* spectral lines, for the different settings of the experimental apparatus, using database NIST and Harvard.

		Amospheric pressure		
Database	Ionization degree	1μs	2 μs	4 μs
Harvard	W I	0.44	0.4	0.39
	W II	0.96	0.79	0.56
NIST	W I	0.65	0.54	0.53
	W II	1.53	1.16	0.95

Table 4: The layer of *W* deposited on *Si*, determined values of electron temperature T_e by Boltzmann plot using *W I* and *W II* spectral lines, for the different settings of the experimental apparatus, using database NIST and Harvard.

		Atmospheric pressure	
Database	Ionization degree	1 μs	2μs
Harvard	W I	0.44	0.55
	W II	0.73	0.75
NIST	W I	0.81	0.89
	W II	1.15	1.13

Table 5: The layer of *W-Al* deposited on *Si* ,determined values of electron temperature T_e by Boltzmann plot using *W I* and *W II* spectral lines, for the different settings of the experimental apparatus, using database NIST and Harvard.

The results, which are listed in Table 3, 4 and 5 show that the electron temperature determined using the database Harvard and NIST are different. The electron temperature which is determined using the singly ionized spectral lines of tungsten $(W\ II)$ is greater than the electron temperature determined from neutral spectral lines of tungsten $(W\ I)$. It is also possible to observed the different behaviour of the temperature for the data measured under the atmospheric and under low pressure. While the electron temperature under the atmospheric pressure is substantially constant (electron temperature determined from neutral spectral lines *W I*), under the low pressure is possible to observe decrease of the temperature with increasing time delay (electron temperature determined from neutral and singly ionized spectral lines *W I* and *W II*). Analysis of the measurements points at the fact that the condition of LTE is not satisfied, so the determination of electron temperature cannot be done correctly. This fact may be caused by the effect of self- absorption and repopulation of the selected spectral lines of tungsten which were used for analysis. It can be also seen that by comparing the electron temperature determined using NIST and Harvard database, temperatures are different. From these facts we can conclude, that the selection of the spectral lines using for calculation of electron temperature is important. Further, there are differences between the using of parameters for calculation of electron temperature from database NIST and Harvard. In our analysis we prefer to use NIST database, because it contains the values of the parameters which are obtained also experimentally (not only by calculation as in case of database Harvard).

Due to aforementioned reasons we focused on the improvement of the determination of electron temperature. The more precise determination of plasma temperature can be reached using Saha-Boltzmann plot. In this case sample of *W* with a protective carbon layer (*1* μm) was analysed. For more precise determination of electron temperature in addition to neutral and singly ionized spectral lines of tungsten also doubly ionized spectral lines of tungsten were used. The obtained results were compared with the electron temperature calculated from Boltzmann plot using neutral spectral lines of nitrogen and oxygen. Table 6 summarizes the obtained results. From the obtained results it is clear that the values of electron temperature determined without *W III* spectral lines are significantly smaller then temperatures determined from *W I-III* spectral lines and from *N* and *O*. This confirms that using doubly ionized spectral lines of *W* improves the precision of electron temperature determination.

The results of these experiments are also presented in publication *"Use of the near vacuum UV spectral range for the analysis of W-based materials for fusion applications using LIBS"* in Physica Scrita [2]

Gate delay (ns)	T (eV)				n_e (cm^3)
	W I-III	N I, O I	W I-II	W I, W III	
100	1.22	1.21	1.02	1.32	2.21 x 10^{17}
500	0.87	0.85	0.73	0.99	1.06 x 10^{16}

Table 6: The electron temperature determined using Saha- Boltzmann plot for *W I-W III* spectral lines, Boltzmann plot for *N I* and *O I* spectral lines, Saha-Boltzmann plot for *W I-II* spectral lines and Saha-Boltzmann plot for *W I, W III* spectral lines and electron density calculated from Stark broadening of $H_\alpha$spectral line, *W*, sample measured under the low pressure (*1330 Pa*) for different gate delays (*100 ns* and *500 ns*).

The second part of the analysis of tungsten-based alloys represents the measurement of the analysed layers of *AlW* and *BeW* (*Al/Be 67 %, W- 33 %*) without or with deuterium deposition applied on the molybdenum substrate. The aim of the analysis these samples was to verify the possibility of using *Al* as a proxy of *Be* for the fusion experiments. For this purpose, ablation rate of the *AlW, AlWD, BeW* and *BeWD* samples were identified. The results are listed in Table 7.

Ablation rate (nm/shot)	AlW	BeW
without D	45 ± 8	39 ± 10
with D	330 ± 80	275 ± 35

Table 7: Ablation rates for *AlW* and *BeW* samples with and without *D* doping.

From these results is clear that the ablation rate increases for *D* doping layers by an order of magnitude. It means that the coating with *D* doping is easier to ablate. Also the evolution of the intensity of the spectral lines was observed from shot to shot and depth profile analysis was done. In comparison the depth profile of *AlWD* and *BeWD* samples it seems that *D* content in *AlWD* sample is significantly less than the content of *D* in *BeWD* sample. Because the layers of *AlWD* and *BeWD* were produced under the similar deposition condition these differences indicates the different retention properties of *Al* and *Be*. This comparison also shows that the using of *Al* as a proxy of *Be* during the fuel retention experiments is not suitable, but it can be used as a substitution in ablation experiments which is proved by the similar values of ablation rates. The results of these experiments are also presented in submitted article *"Determination of deuterium depth profiles in fusion-relevant wall materials by nanosecond LIBS"* [3].

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AFD04 Suchoňová, Mária [UKOMFKEFd] (25%) - Krištof, Jaroslav [UKOMFKEF] (25%) - Anguš, Michal [UKOMFKEFd] (25%) - Veis, Pavel [UKOMFKEF] (25%): Rotational temperatures in hydrogen and hydrogen-argon dc discharge
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AFD05 Gavrilovic, M. R. (5%) - Pribula, Marek [UKOMFKEFd] (30%) - Suchoňová, Mária [UKOMFKEFd] (30%) - Veis, Pavel [UKOMFKEF] (30%) - Jovicevic, S. (5%): Diagnostics of laser induced lead plasma
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In: ESCAMPIG XXIII : Europhysics Conference [elektronický zdroj]. - [Mulhouse] : European Physical Society, 2016. - S. 377-378 [USB kľúč]. - ISBN 979-10-96389-02-5
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BFA Abstrakty odborných prác zo zahraničných podujatí (konferencie, ...)

BFA01 Pribula, Marek [UKOMFKEFd] (20%) - Krištof, Jaroslav [UKOMFKEF] (16%) - Suchoňová, Mária [UKOMFKEFd] (16%) - Maťko, Igor (16%) - Švec, Peter (16%) - Veis, Pavel [UKOMFKEF] (16%): Analyses of Fe-B-Si amorphous alloys by CF LIBS using VUV-NIR spectra
Lit. 5 zázn.
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[EMSLIBS 2015 : Laser-Induced Breakdown Spectroscopy : Euro-Mediterranean Symposium. 8th, Linz, 14.-20.9.2015]

BFA02 Suchoňová, Mária [UKOMFKEFd] (20%) - Krištof, Jaroslav [UKOMFKEF] (20%) - Kociánová, Mária [UKOMFKEFd] (20%) - Pribula, Marek [UKOMFKEFd] (20%) - Veis, Pavel [UKOMFKEF] (20%): LIBS analysis of tin and tin alloys using vacuum UV-NIR spectral range
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[EMSLIBS 2015 : Laser-Induced Breakdown Spectroscopy : Euro-Mediterranean Symposium. 8th, Linz, 14.-20.9.2015]

GHG Práce zverejnené spôsobom umožňujúcim hromadný prístup

GHG01 Suchoňová, Mária [UKOMFKEFd] (25%) - Anguš, Michal [UKOMFKEFd] (25%) - Krištof, Jaroslav [UKOMFKEF] (25%) - Veis, Pavel [UKOMFKEF] (25%): Study of rotational spectra of Lyman and Fulcher-alpha system in hydrogen discharge
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GHG02 Pribula, Marek [UKOMFKEFd] (25%) - Suchoňová, Mária [UKOMFKEFd] (25%) - Krištof, Jaroslav [UKOMFKEF] (25%) - Veis, Pavel [UKOMFKEF] (25%): Study of H and D atoms in fusion relevant wall materials
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GHG03 Suchoňová, Mária [UKOMFKEFd] (25%) - Pribula, Marek [UKOMFKEFd] (25%) - Martišovitš, Viktor [UKOMFKAFZMs] (25%) - Veis, Pavel [UKOMFKEF] (25%): Study of RF discharge enhancement for LIBS analyses of fusion-relevant wall materials

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Štatistika kategórií (Záznamov spolu: 16):
ADC Vedecké práce v zahraničných karentovaných časopisoch (2)
AED Vedecké práce v domácich recenzovaných vedeckých zborníkoch, monografiách (1)
AFC Publikované príspevky na zahraničných vedeckých konferenciách (3)
AFD Publikované príspevky na domácich vedeckých konferenciách (5)
BFA Abstrakty odborných prác zo zahraničných podujatí (konferencie, ...) (2)
GHG Práce zverejnené spôsobom umožňujúcim hromadný prístup (3)

Štatistika ohlasov (1):
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Zoznam prijatých vedeckých prác

ADC Vedecké práce v zahraničných karentovaných časopisoch

Suchoňová, Mária [UKOMFKEFd] - Veis, Pavel [UKOMFKEF] - Karhunen, Juusso - Paris, Peeter - Pribula, Marek [UKOMFKEFd] - Piip, Kaarel - Laan, Matti - Porosnicu, Corneliu - Lungu, Cristian - Hakola, Antti: Determination of deuterium depth profiles in fusion-relevant wall materials by nanosecond LIBS
In: Nuclear Materials and Energy, NME_2016_294