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Influence of pressure and mixing with air on the equilibrium composition of laser-induced plasma on ODS steel

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Abstract: Laser Induced Plasma (LIP) is transient, the part of the time evolution of its emission used for the Laser Induced Breakdown Spectroscopy (LIBS) technique corresponds to plasma pressures that are comparable to atmospheric, where a certain degree of mixing of the ablated material and air must be taken into account. The aim of this work was to discuss the influence of pressure change and air mixing of laser ablated material on the equilibrium composition of plasma obtained on an ODS steel target, which is a material of great interest for nuclear technology. Special care is devoted to the influence of pressure and mixing on the equilibrium relationship between temperature T and electron number density N_e, which is important for plasma diagnostics. The obtained results show that the concept of local thermodynamic equilibrium (LTE) can be used for LIBS plasma diagnostics and optimization of spectrochemical analysis parameters. The conclusion refers to the expected interval of plasma pressure during its evolution (10-0.1 bar) and the expected degree of mixing of the ablated material and air, in the temperature range 5,000-15,000 K, relevant for the LIBS technique.

Keywords: equilibrium plasma composition; LIBS; ODS steel; pressure, air mixing.

INTRODUCTION

In order to describe the gas plasma state in detail, it is necessary to know the population distribution of electronic levels of various atoms and ions, as well as the distribution of kinetic energy of various particles, including electrons. In the general case, to fulfill this task, it is necessary to carefully take into account all elementary processes in the plasma, together with their reaction rates and cross sections for collisions. If the plasma fulfills the conditions of Local Thermodynamic Equilibrium (LTE), it can be completely described much easier, by applying appropriate equilibrium distributions, without the need to treat the

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kinetics of elementary processes. Of particular interest is the knowledge of plasma composition at different temperatures. Under the conditions of validity of LTE, the equilibrium composition of the plasma as a function of temperature can be calculated by solving a system of equations consisting of the Saha equations, the mass conservation law and the electroneutrality equation. This approach is successfully applied to describe the equilibrium composition of laser-induced plasma (LIP), for the LIBS technique (Laser-Induced Breakdown Spectroscopy).[1,2] In somewhat colder plasmas, where the existence of molecules cannot be ignored, a more optimal approach to calculating the equilibrium composition is by minimizing the total free energy of the particles, taking into account the appropriate mass and charge conservation relations.[3,4] LIBS is a modern technique of elemental chemical analysis, which uses a plasma induced on the analyzed sample as a source of excitation. Plasma is induced by a high power density laser pulse. Usually, nanosecond laser pulses are used for LIBS, but lasers with shorter pulses can also be used. The interaction of a laser pulse and a solid sample leads to its ablation and the formation of a hot and dense plasma that spreads usually in air, at atmospheric pressure.[5] For analytical applications, a part of the time evolution of the plasma between 1 and 10 μs is usually used, when the plasma expansion slows down almost completely, and the pressure becomes close to the ambient pressure. Careful experimental measurements and theoretical modeling of laser-induced plasma expansion have shown that during the time evolution of the plasma, near the end of its expansion, the pressure can even be lower than ambient.[6,7] Cristoforetti *et al.* showed that the LTE concept can be applied to LIP, in the stage of evolution used for LIBS technique.[8]

Oxide dispersion strengthened steels (ODS) are promising materials in nuclear technology, due to their favorable properties when exposed to high temperatures and intense radioactive radiation.[9] Different types of ODS steel are considered for potential application both in fission technology, for fuel cladding, and in nuclear fusion reactors, where they should play the role of first wall material (known also as plasma face material, PFM). Due to the possibility of remote analysis, the LIBS technique is of particular interest for application in nuclear technology.

The aim of this work was to clarify in more detail the influence of pressure on the equilibrium composition and the relationship between temperature and electron number density during the time evolution of laser-induced plasma. Since it is difficult to monitor the temporal evolution of the plasma pressure during its expansion with plasma diagnostic methods, it is usually assumed that the plasma pressure in the time interval used for LIBS, is close to atmospheric pressure. Given that the acquisition of spectra for LIBS is performed within time interval of 1-10 μs after the laser pulse with an ICCD detector (or from 1 μs to 10 ms or more with a CCD camera), it is of interest to take into account pressures higher than atmospheric, as well as slightly lower values.[6,7] Also, the influence of the mixing of the ablated material and the surrounding air should be taken into account. The equilibrium composition was calculated using the program described in our recent publication,[10] where the influence of the ionization potential lowering (IPL, known also as continuum lowering) was taken into account. ODS steel was chosen to illustrate the effect of pressure and mixing with air, because it contains mostly metals but also non-metal carbon, which will significantly affect the effect of mixing with air.

METHOD

The equilibrium composition of the plasma is calculated by using the previously developed program, which is described in details recently and will only be briefly outlined here.[10] First, local thermal equilibrium is assumed to be established on a given temperature T for the initial atomic number densities of all elements that constitute plasma. Each plasma component is assumed to enter subsequent ionization processes and thus multiple ionization stages are reached. For every atomic species i, the ionization-recombination equilibrium between atoms in ionization stage j and j+1 is described by Saha equation:

$$S_{ij} = \frac{N_{i,j+1}N_e}{N_{ij}} = 2\left(\frac{2\pi m \rightleftarrows \rightleftarrows \rightleftarrows k_B T}{h^2}\right)^{3/2} \frac{Z_{i,j+1}(T)}{Z_{ij}(T)} e^{-\frac{E_{ij}-\Delta E}{k_B T}} \tag{1}$$

where S_{ij} is the Saha coefficient, N_{ij} and $N_{i,j+1}$ are the number densities of species i in j and j+1 ionization stage, respectively, N_e is the electron number density, m is the mass of an electron, h is the Plank's constant, k_B is the Boltzmann constant and E_{ij} is the corresponding ionization energy, while ΔE is ionization potential lowering. $Z_{ij}(T)$ and $Z_{i,j+1}(T)$ are the electronic partition functions of species i in j and j+1 ionization stages, respectively. These partition functions are calculated from the database of statistical weights and energies adopted from NIST.[11] The partition function of an atom or ion is defined as:

$$Z_{ij}(T) = \sum_{k=1}^{n} g_k e^{-\frac{E_k}{k_B T}} \tag{2}$$

where g_k and E_k are, respectively, the statistical weight and energy of electronic level k (relative to the ground level energy), while n is the highest occurring level.

Based on the plasma quasi-neutrality conditions, the partial pressure of free electrons p_e will be:

$$p_e = \sum_{i=1}^{N} \sum_{j=1}^{M} (j-1) p_{ij} \tag{3}$$

where N is the number of elements that constitute plasma, M is the maximal number of ionization stages while p_{ij} is the partial pressure of atomic species i, in ionization stage j (j=1 for neutral atom, j=2 for single charged ion etc.). Also the Dalton's law of partial pressures applies, so the total pressure P is:

$$P = p_e + \sum_{i=1}^{N} \sum_{j=1}^{M} p_{ij} \tag{4}$$

All Saha equations are mutually dependent through the p_e (or N_e) value, and after N_e is obtained the program calculates all N_{ij} values.[12] Based on this set of N_e and N_{ij} values, the ionization potential lowering is determined by Stewart-Pyatt model,[13,14] via equation:

$$\Delta E = \frac{3}{2}\frac{je^2}{4\pi\varepsilon_0\lambda_D}\frac{\left[\left(\frac{r_{\mathrm{IS}}}{\lambda_D}\right)^3+1\right]^{2/3}-1}{\left(\frac{r_{\mathrm{IS}}}{\lambda_D}\right)^3} \tag{5}$$

r_{IS} being the ion-sphere radius:

$$r_{\mathrm{IS}} = \left(\frac{3j}{4\pi N_e}\right)^{1/3} \tag{6}$$

while λ_{D} is the Debye length, defined as:

$$\lambda_D = \sqrt{\frac{\varepsilon_0 k_B T}{e^2\left(N_e+\sum_i\sum_j (j-1)^2 N_{ij}\right)}} \tag{7}$$

ε_0 being vacuum permittivity and e is the elementary charge.

After that, the iterative procedure follows and all Saha equilibriums now need to be recalculated (this time with IPL included) in order to obtain a new, more accurate set of N_e and N_{ij} values. This again leads us to a new and more accurate value of ΔE. Pressure reduction due to ionization potential lowering, ΔP is also applied:[15]

$$\Delta P = \frac{e^2\left(N_e+\sum_i\sum_j j(j-1)N_{ij}\right)}{24\pi\varepsilon_0\lambda_D} \tag{4}$$

Iterations are further performed until the difference between two adjacent iterations of N_e value is lower than 10^5 m^{-3}. Reduction of the partition functions can occur due to the lowering of the ionization potential, if certain high-lying electronic states remain above the new ionization potential. In present calculations, the reduction of partition functions due to IPL is taken into account in order to obtain more reliable results. Validation of the used program was performed by comparison with the literature data, and can be found in our recent publication.[10]

RESULTS AND DISCUSSION

Effect of pressure on plasma composition

The equilibrium composition of the plasma in the range of temperatures relevant for the LIBS technique, from 5.000 to 15.000 K, is given in Figures 1 and 2, for pressures of 0.1 and 5 bar*, respectively. The pressure of the LIP ranges between those two values in the final phase of its time evolution, when the spectral emission is registered for elemental analysis and characterization of the plasma. The equilibrium composition of the plasma corresponds to the starting composition typical for ODS steel, Table I. Since ODS steel contains large ammount of Fe, we have assumed that selective evaporation does not occur during the ablation, as shown earlier by Pershin *et al.*[16] For that starting composition and temperature range, the presence of molecules in plasma is negligible. The concentration of single charged ions becomes higher than the concentration of the corresponding atom in the temperature interval from 8,000 to 9,700 K for metals depending on their first ionization energy (from 6.77 eV** for Cr to 7.90 eV for Fe), for plasma

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* 1 bar = 10^5 Pa

** 1 eV = $1.602\cdot10^{-19}$ J

at atmospheric pressure. As a non-metal, carbon has a significantly higher first ionization energy (11.26 eV), so its ion and atomic mole fractions become equal at higher temperature of 13,500 K. With the increase of plasma pressure, the proportion of ions decreases, and thus it equals the fraction of atoms at higher temperatures. Conversely, at pressures lower than atmospheric, the degree of ionization increases. For example, the temperature at which the atom and single charged ion mole fractions are the same for Ti, shifts from approximately 6,290 K at a pressure of 0.1 bar, over 7,548 K at 1 bar, to 8,750 K at 5 bar. The significant shift of the ionization-recombination equilibrium with pressure indicates that this fact must also be taken into account during the spectroscopy of LIP, especially if the emission is integrated over a long period of time. Even in time-resolved measurements, using an ICCD camera, where the integration time can go well below 1 μs, the decision of whether it is better to use an atomic or an ion line for spectrochemical analysis also depends on the pressure evolution during the camera exposure. The concentrations of doubly charged ions are significant at higher temperatures, at low pressure (Figure 1), while they become negligible as the pressure increases.

TABLE I. Composition of the ODS sample with first ionization energies of its constituents

element	mole fraction	E_{ion} (eV)
C	0.00303	11.26
Cr	0.12700	6.77
W	0.00584	7.87
Ni	0.00028	7.64
Ti	0.00152	6.83
Fe	0.86233	7.90

The effect of pressure on the ionization-recombination equilibrium of the analyte is actually related to the dependence of the electron number density on pressure, which is why this dependence should be discussed in more detail. In Figure 3, the mole fraction of electrons as a function of temperature is presented, for the ODS steel plasma, at different pressures. As can be seen, for a given temperature, the mole fraction of electrons strongly depends on pressure: with a decrease in pressure (followed by the decrease in the number densities of all particles), the mole fraction of electrons increases. If the mole fractions of electrons for the corresponding pressures are translated into electron number densities N_e, the dependence as shown in Figure 4. is obtained. The increase in electron number density with pressure is not linear, it increases more slowly at higher pressure. The reason for this behavior lies in the fact that the increase in electron number density favors recombination.

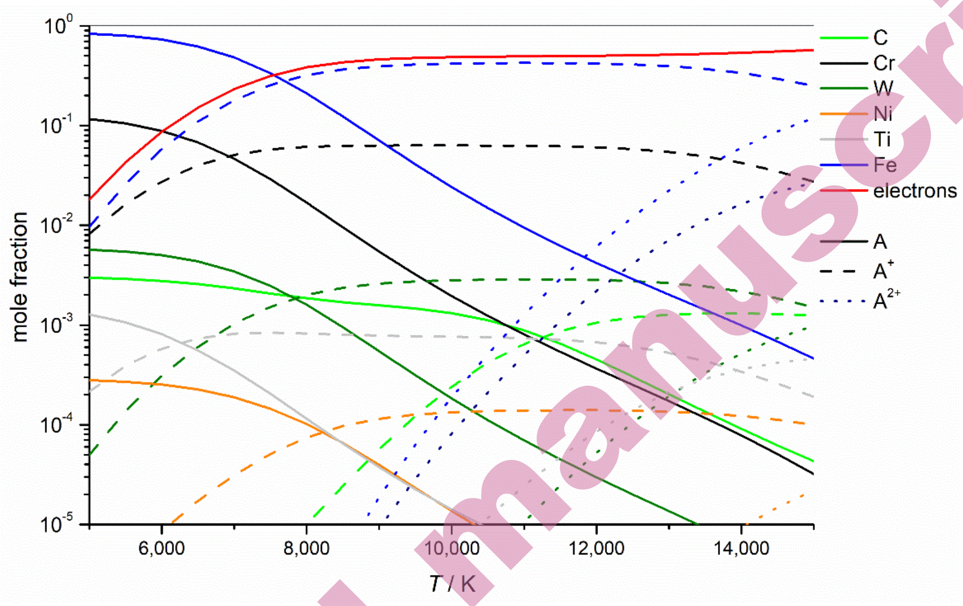

Fig. 1. Equilibrium plasma composition as a function of temperature, which corresponds to the ablation of ODS steel at pressure of 0.1 bar.

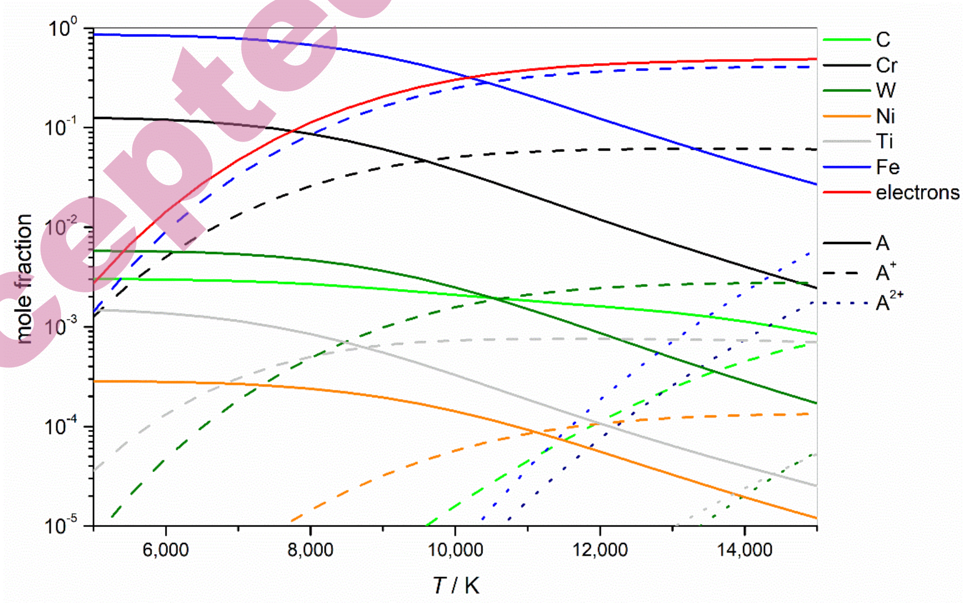

Fig. 2. Equilibrium plasma composition as a function of temperature, which corresponds to the ablation of ODS steel at pressure of 5 bar.

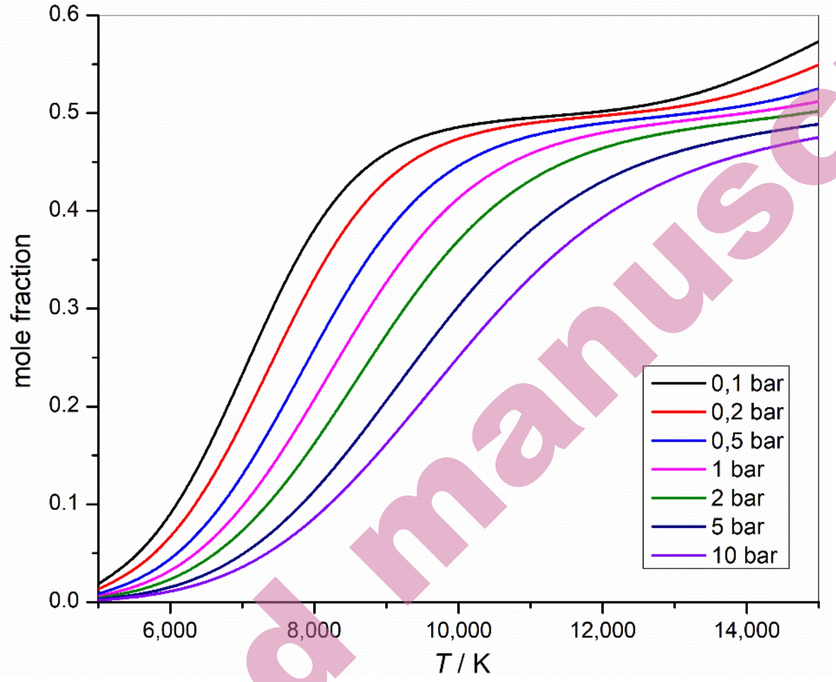

Fig. 3. Mole fraction of electrons in ODS plasma at various pressures.

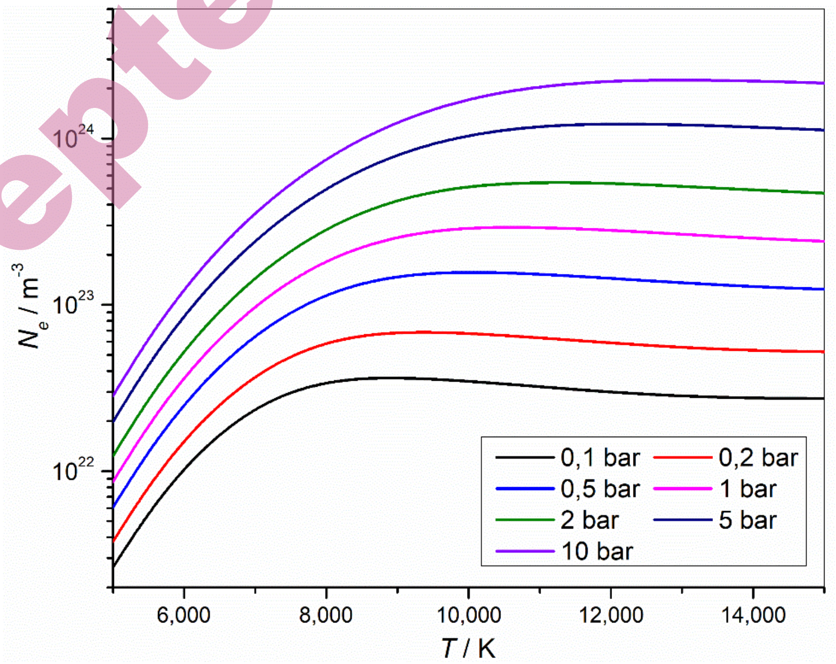

Fig. 4. Number density of electrons in ODS plasma at various pressures.

Figure 4. is very interesting from the plasma diagnostics point of view. If the plasma is in LTE (for a given plasma composition and pressure) each temperature corresponds to exactly one value of N_e, considering the lower temperature region in which (at a given pressure) N_e increases. For higher temperatures, where N_e reaches maximum, starts to fall and changes very slowly, the approach cannot be applied. It follows that the pressure could be determined using Figure 4, based on the previously measured values for N_e and temperature. Or *vice versa*: if the pressure value is known, based on the previously determined N_e, the temperature of plasma can be determined. For pressures (up to 10 bar, including the development phase of plasma plume with a pressure lower than the ambient) and temperatures relevant for the LIBS technique (5,000-15,000 K), the dependence curves of $N_e(T)$ are sufficiently different that from them one can, at least roughly, estimate the pressure in plasma that emitted spectral lines, based on which T and N_e were determined.

In plasmas where the pressure changes with time, such are LIPs, there is no simple diagnostic method to measure the pressure, especially not by non-invasive diagnostic methods of optical emission spectroscopy. Therefore, any possibility of even a rough estimation of the pressure value is of interest. The aforementioned possibility of applying Figure 4. to determine T or N_e, if one of those two values is previously determined by some diagnostic method, is also interesting for plasma diagnostics, especially in cases when N_e is determined first (from the Stark broadening of spectral lines, for example) and then the temperature is estimated using the curves from Figure 4. This is because the determination of temperature by emission methods (Boltzmann plot, emission line-intensity-ratio method) is somewhat unreliable, due to large gradients of plasma parameters, especially if the spectral measurements are not time-resolved.

The effect of mixing with air on plasma composition

The transient nature of LIP results in several additional problems for applications in optical spectroscopy. In addition to the unknown plasma pressure during the time interval of the spectrum acquisition, the plasma composition is also unknown. It is undeniable that, while the plasma pressures during its expansion are significantly higher than the ambient pressure, there is practically no mixing of the ablated material with particles from the surrounding atmosphere. In other words, the composition of the plasma corresponds to the composition of the ablated material, taking into account, of course, the ionization processes. However, knowing the typical time interval of the time evolution of LIP and the expected pressures, the mixing of the ablated material with the surrounding air must also be taken into account. Therefore, the influence of mixing air and ablated material must also be considered in the context of the results and discussion in the previous chapter.

When it comes to LIP plasma that is formed in air at atmospheric pressure, considering that the drop in pressure is accompanied by a drop in temperature, i.e. the extinction of optical emission, it is reasonable to assume that in the stage of plasma evolution when the LIBS signal is registered, complete mixing of the air and ablated material (50 mol % sample + 50 mol % air) is not achieved. Therefore, we took into consideration the following degrees of mixing: 5, 10 and 20 mole percent of air, in order to evaluate the influence of mixing on the degree of dissociation of analytes and the connection of N_e (T) in the LIP plasma. Figures 5. and 6. show the distribution of the equilibrium plasma composition as a function of temperature, at a pressure of 1 bar, without mixing with air and for a mixture with 20 vol % of air and 80 vol % of ablated material (with the composition given in Table I).

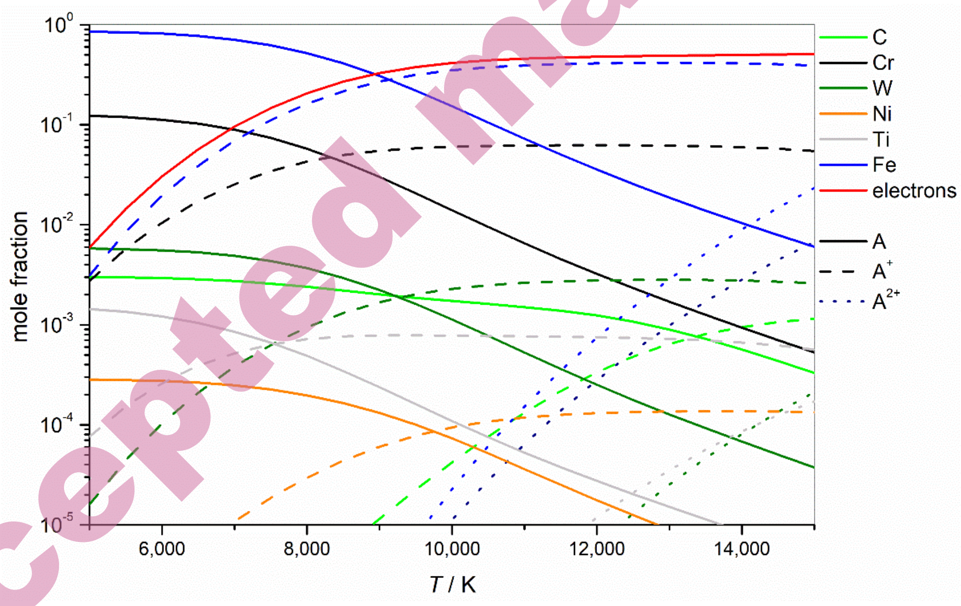

Fig 5. Equilibrium plasma composition as a function of temperature at 1 bar, which corresponds to the ablation of pure ODS steel.

The introduction of 20 molar percentages of heavier ionizing elements (the first ionization energy for N is 14.53 eV, for O 13.62 eV and for Ar 15.76 eV) in relation to the metals present (6.67 eV to 7.90 eV), favours a slightly higher metal ionization, so that the temperatures on which the mole fractions of atoms and ions are equalized, move to lower values: 100 K for C, up to 122 K for Ti (see Table II), with the shift being smaller for higher ionization energies. At pressures lower than atmospheric this effect is somewhat smaller, while at higher pressures, it is greater. However, one should not lose sight of the fact that an even more significant proportion of air in the plasma can only occur when the plasma pressure approaches the surrounding pressure. It can therefore be concluded that possible mixing of ablated material and air does not significantly affect the choice of optimal lines (atomic or ionic) for the LIBS technique.

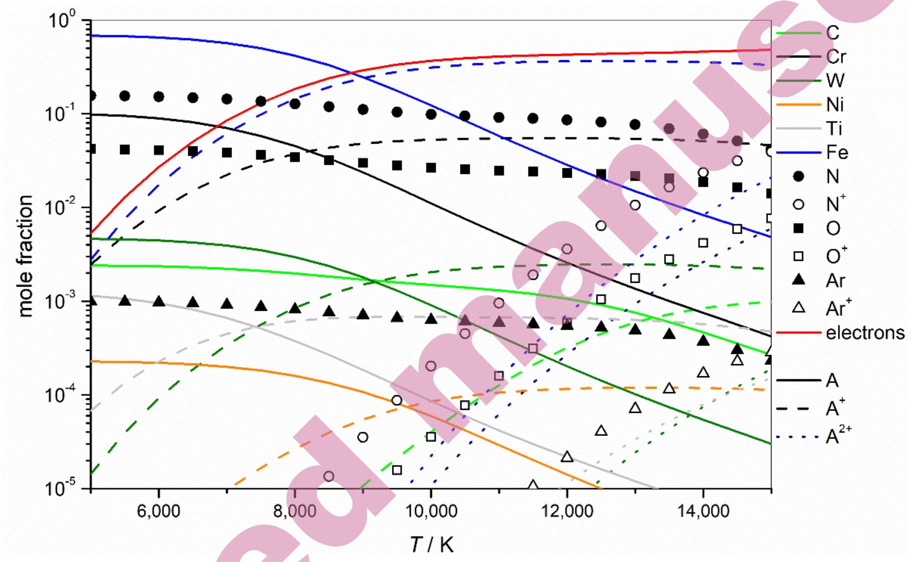

Fig 6. Equilibrium plasma composition as a function of temperature at 1 bar, which corresponds to the ablation of 80 vol % ODS steel mixed with 20 vol % air.

TABLE II. Temperatures at which mole fractions of atoms and ions of all ODS constituents are equalized, for pure ablated ODS and for mixing with 20 vol % of air, at pressure of 1 bar. The differences between these two temperatures are given in ΔT column

element	T / K		ΔT / K
	ODS	ODS + 20 vol % air	
C	13,375	13,275	100
Cr	8,327	8,209	118
W	9,281	9,177	104
Ni	9,747	9,644	103
Ti	7,548	7,426	122
Fe	9,130	9,025	105

The effect of mixing the ablated material with air on the ionization-recombination balance is illustrated in Figure 7. Due to the overall decrease in the portion of metals, their degree of ionization is lower, and so is the mole fraction of electrons. However, the decrease in the mole fraction of electrons is less than the decrease in the mole fraction of the ablated material due to mixing with air: the introduction of heavier ionizing elements from the atmosphere favors the metal ionization. For plasma diagnostics, the influence of mixing with air on the number density of electrons is very important. With increasing temperature and number density of electrons, the influence of mixing with air increases, until at around 9,000 K it reaches approximately 5%, compared to a mixture with 20% air, i.e. 8% at 10,000 K. This means that curves on Fig 7. can be used to determine T if N_e is known, and *vice versa* for the atomic lines of metals, because those in the part of the time evolution of the LIP plasma used for analysis usually have emission maxima at temperatures below 8,000 K. On the other hand, the lines of metal ions have emission maxima at temperatures higher than 10,000 K. If the temperature is measured experimentally, the electron number density can be determined by applying curves from Fig 7. in the entire temperature interval, therefore also for the ionic lines of metals, with an accuracy better than 10%.

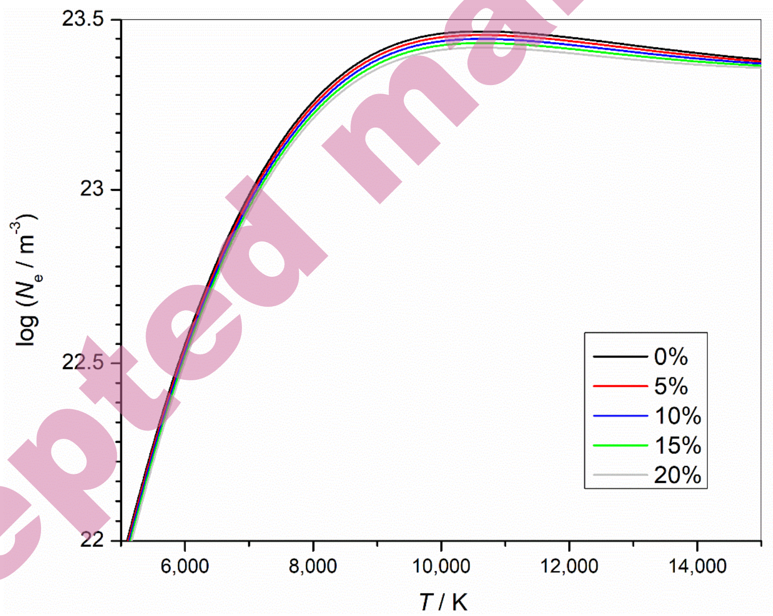

Fig 7. Logarithm of the electron number density as a function of temperature in ODS plasma with various portions of air indicated (in vol %).

CONCLUSION

With increasing pressure, the equilibrium composition of the plasma shifts towards recombination, i.e. the degree of ionization decreases. Depending on the first ionization energy of the observed element, the temperature at which the mole fractions of atoms and ions are equalized shifts to higher values: this shift is significant, for metals it can be up to 1,500 K when the pressure changes by an order of magnitude. With an increase in pressure, the electron number density for a given temperature also increases, with the increase in N_e being slower than the increase in pressure, because an increase in pressure favours recombination. The dependence of N_e on T at different pressures is sensitive enough that, based on previously measured T and N_e, one can at least roughly estimate the pressure. In other words, by monitoring the time evolution of N_e and T, the time evolution of the plasma pressure can be roughly estimated. This fact is particularly important considering the lack of suitable emission spectroscopic methods for plasma pressure determination.

Considering the fact that the emission of LIP plasma is recorded at pressures close to atmospheric, the influence of the mixing of the ablated material with the surrounding air on the ionization-recombination balance of the analyte elements, as well as on the relationship between N_e and T, cannot be completely ignored. Air into the plasma increases the degree of ionization, so that the temperatures at which the mole fractions of atoms and ions are equalized move to higher values to a certain extent. The effect of mixing with air on the dependence of N_e (T) can be neglected up to 9,000 K, even for a mixture with 20% air, which is the upper limit of mixing relevant for LIBS. In the interval of 9,000-15,000 K, the LTE concept can still be used to determine N_e from the previously measured temperature.

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ИЗВОД

УТИЦАЈ ПРИТИСКА И МЕШАЊА СА ВАЗДУХОМ НА РАВНОТЕЖНИ САСТАВ ЛАСЕРСКИ ИНДУКОВАНЕ ПЛАЗМЕ НА ОДС ЧЕЛИКУ

МИРОСЛАВ РИСТИЋ, НИКОЛА КРСТЕВСКИ, МИЛИЦА МАРКОВИЋ, АЛЕКСАНДРА ШАЈИЋ И МИРОСЛАВ КУЗМАНОВИЋ

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Ласерски индукована плазма је транзијентна, део временске еволуције њене емисије који се користи за технику спектроскопије ласерски индуковане плазме (ЛИБС) одговара притисцима плазме који су упоредиви са атмосферским, при чему се мора узети у обзир и известан степен мешања аблираног материјала и ваздуха. Циљ овог рада је да продискутује утицај промене притиска и мешања са ваздухом ласерски аблираног материјала на равнотежни састав плазме добијене на мети од ОДС челика, материјала који је од великог интереса за нуклеарну технологију. Посебна пажња је посвећена утицају притиска и мешања на зависност између температуре T и концентрације електрона N_e, која је од великог значаја за дијагностику плазме. Добијени резултати показују да се концепт локалне термодинамичке равнотеже може користити за дијагностику ЛИБС плазме и оптимизацију параметара спектрохемијске анализе. Закључак се односи на очекиван интервал притиска плазме током њене еволуције (10-0,1 бар) и очекивани степен мешања аблираног материјала и ваздуха, у опсегу температура 5.000-15.000 K, релевантном за ЛИБС технику.

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