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VELOCITY AND ELECTRONIC STATE DISTRIBUTIONS OF SPUTTERED Fe
ATOMS BY LASER-INDUCED FLUORESCENCE SPECTROSCOPY.*

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ABSTRACT

Velocity distributions and relative populations in the fine-structure levels of the a^5D_J ground state of Fe atoms, produced by sputtering with 3 keV argon ions, have been investigated by Doppler shifted laser induced fluorescence. The laser system employs a single-mode, scanning ring dye laser, amplified by a sequence of three excimer-pumped flowing-dye cells. Frequency doubling in a KD*P crystal was used to produce high energy ($>.5$ mJ) pulses of narrowband tunable UV output near 300 nm. Laser power influence on effective velocity bandwidth was investigated. Favorable light-collection geometry minimized distortion of the velocity spectra from apparatus-averaging effects. In impurity flux diagnostic applications in fusion devices, substantial spatial averaging may occur. In the latter case, the narrow velocity bandwidth (70 m/s, transform limit) of the present laser system is particularly useful.

I. INTRODUCTION

Laser-induced fluorescence spectroscopy (LFS) has recently been applied in a number of experiments as a diagnostic technique for metallic impurity atoms in the edge region of fusion devices. Measurement of both atomic densities¹⁻³ and velocity distributions⁴⁻⁸ are of interest, the latter quantities being necessary for the determination of impurity flux toward the plasma as well as for information regarding the mechanism responsible for release of the detected atoms. In almost all cases reported¹⁻⁶ pulsed laser systems were employed which had repetition rates limited to the regime below ~20 Hz. Minimum bandwidth⁵⁻⁷ was ~2 GHz, too coarse for resolving thermal velocity distributions. Continuous wave, single-mode lasers exhibit ultra high resolution (bandwidth $\leq$ 20 MHz) and have been used in numerous laboratory experiments to measure velocity distributions by Doppler-shifted LFS. Drawbacks for fusion diagnostic applications are the lack of saturating power and the restriction to a few metallic atoms with strong visible transitions⁸.

The present work describes high power, pulsed laser system which extends the repetition rate to the 100-200 Hz regime while maintaining a bandwidth near the Fourier transform limit of a few tenths of 1 GHz. This resolution is ideal for probing thermal distributions (typically 1-2 GHz FWHM) and can be broadened by the use of higher laser intensity, in order to excite a greater fraction of the atoms, eg. for studying sputtered velocity distributions. Access to the ultraviolet (UV) regions is via high efficiency, angle tuned second harmonic generation.

As an initial test of this system, velocity distributions were measured for Fe atoms sputtered in all fine-structure components ($J=0-4$) of the a^5D_3 ground state multiplet, using 3 keV normal incidence bombardment of

polycrystalline iron (99.99%) by argon ions. Relative populations in the 0-4 fine-structure components were determined, and confirmed by measurements with broadband laser excitation. Comparison with previous laboratory LFS studies on Fe atoms, and implications for fusion diagnostics are discussed.

II. EXPERIMENTAL

The laser system, shown schematically in Fig. 1, consists of a narrowband cw oscillator followed by a high-power pulsed amplifier chain. A detailed description of this system is available,⁹ so this account will center on key features. The oscillator stage consists of a commercial cw ring dye laser (Spectra Physics 380A),¹⁰ pumped by an argon ion laser (SP 171-19). Normally, the ring laser is operated as a single-mode device with ~20 MHz linewidth, electronically scannable over a 30 GHz range in 100 msec. Broadband operation is also possible, by removal of the optical diode and thick etalon, giving a 50 GHz envelope, fine-tunable via the thin etalon. Such operation is potentially useful for absolute density measurements. In comparison with previously described systems employing standing-wave single mode oscillator stages,¹¹⁻¹⁶ the present system benefits from the relatively higher power (100 mW vs 10 mW regime) available as narrowband input to the first amplifier stage, permitting a high contrast ratio (of amplified input signal to amplified spontaneous emission) to be maintained in that cell and the subsequent amplifier chain.

Flowing dye cells with individual circulation systems were transversely pumped by the output of an excimer laser (Lumonics, 861-T) operating at 308 nm with a nominal pulse length of 8 ns, and pulse energy of 60 mJ when operated for maximum power (8W) at 130 Hz. Pickoff mirrors were used to optimize the power distribution to the amplifier cells 1-3 at 5, 30, and 65%, respectively, of the excimer laser output. For the present work, Rhodamine B in methanol

was used at $4.6 \times 10^{-4} \text{M}$ in cell 1, $3.8 \times 10^{-4} \text{M}$ in cells 2 and 3, while a standard Rhodamine 6G mixture was used in the ring. Substantial enhancement of contrast ratio, to values routinely greater than 100 at a gain of 10^3 to 10^4 in cell 1, was achieved by peaking the gain curve^{17,18} at 604 nm via the addition of suforhodamine B at $1.8 \times 10^{-5} \text{M}$ concentration.

Visible narrowband output energy from the amplifier chain was about 6 mJ/pulse, with fresh dye and excimer gas. Frequency doubling in 95% deuterated KD^*P with type I phase matching gave up to 0.6 mJ/pulse of narrowband UV output. Broadband light is less efficiently doubled, leading to UV contrast ratios of several hundred. The Fourier transform limit for the laser pulse of duration $\tau = 8 \text{ ns}$ implies a frequency spread $\Delta\nu = 4 \ln 2 / (\pi\tau) = 110 \text{ MHz}$ in the visible output of the amplifier system, for Gaussian pulse shape. The corresponding velocity width Δv in Doppler-shift experiments is $\Delta v = \lambda \Delta\nu / c = 66 \text{ m/s}$ at the visible wavelength $\lambda = 604 \text{ nm}$. Measurements with scanning etalons in the visible indicate operation within a factor of two of this limit without the use of dispersive devices¹¹⁻¹³ between amplifier stages. The resulting velocity width is still narrow relative to thermal distributions (eg. for Fe atoms at $T=1120\text{K}$, $\Delta v = 960 \text{ m/s}$, FWHM for the optogalvanic hollow cathode signal of Fig. 2 discussed below).

The laboratory experiments were controlled by a micro-computer through a CAMAC interface. A staircase ramp voltage was used to scan the frequency of the ring laser through the 30 GHz range. At each frequency position, the excimer laser is pulsed one or more times. Each pulse is centered in a $1 \mu\text{s}$ window during which the acousto-optic modulator allows the narrowband laser beam into the amplifier system, a procedure employed to minimize the power input to cell 1. The ring laser is continuously monitored by a temperature stabilized etalon of 2.95 GHz free spectral range, providing calibration of the

frequency scale. Many successive sweeps through the ring laser scan range are typically used to average out any gradual drift in experimental parameters such as the laser pulse output energy, which decreases in response to depletion of the excimer gas mixture (nominally 2×10^6 shots to half power) dye photolysis. Essentially constant UV power could be obtained across the 30 GHz scan range without variation of the doubling crystal position.

Analog fluorescence signal from the experiment was detected by a cooled photomultiplier tube (EMI 9659 QB) and digitized within a 100 ns window around the laser pulse. A signal proportional to the integrated UV laser output was recorded by a fast photodiode circuit. Opto-galvanic signal¹⁹⁻²¹ from an iron hollow cathode lamp is centered on the zero-velocity position. A few percent of the UV lower energy directed into the lamp provides a strong response, which is useful for unambiguous alignment of successive Doppler scans. Experiments were performed at laser pulse repetition rates of up to 80 Hz (temporarily limited by computer software). Constant energy per pulse but slightly ~~increasing shot to shot~~ amplitude variation was observed with increasing rate.

The sputtering chamber⁹, used ultra high vacuum components and reached a base pressure of 3×10^{-10} mbar after mild 200°C bakeout. Experiments were performed in a argon background gas at 6×10^{-5} m bar with a commercial ion gun (Varian Associates, Model 981-2043). Ion current at normal incidence to the target was 10-20 μ A at a beam energy of 3 keV, and was focused to a diameter comparable to that of the 3.9 mm dia. polycrystalline iron target, which was sharply defined by a thin molybdenum mask. The UV laser beam was directed normal to the iron disk, opposite to the ion-bombarded side. About 10% of the pulse energy passed through a 1.2 mm diameter central aperture, producing a cylindrical illuminated region with a nearly constant intensity

profile in front of the target. Fluorescence photons were collected by a $f/2.2$ optical system, the region within 5 mm of the surface being excluded by a mask in an intermediate image plane. An interference filter with 10 nm bandpass, centered at 383 nm was used to discriminate against ambient light, while transmitting the lines of the $y^5D \rightarrow a^5F$ multiplet used in detection.

A sample velocity spectrum for Fe sputtered in the ground state is given in Fig. 2. The laser was tuned to produce excitation on the a^5D_4 to $y^5D_4^0$ line at 302.065 nm, with fluorescence detected mainly on the $y^5D_4^0$ to a^5F_5 line at 382.043 nm. The laser was attenuated to an intensity of $\sim 2 \text{ kW/cm}^2$ in the detection zone, producing negligible broadening of the velocity distribution of the sputtered Fe atoms, the top trace in Fig. 2. The data were taken in 20 successive sweeps of the ring laser through its 30 GHz scan range, with 5 shots of the pulsed system per data point on each sweep, the excimer laser running at 40 Hz. The zero frequency marker is taken to be the center of the optogalvanic line with no significant pressure shift being expected²² in the hollow cathode lamp (Instrumentation Laboratory, VISIMAX) which contained a few mbar of neon. A second optogalvanic peak is observable 24.45 GHz (0.815 cm^{-1}) to higher frequency corresponding to the $J = 2$ to $J' = 2$ component of the same multiplet, serving as a convenient calibration of the monitoring etalon. The population of the $J = 2$ state is sufficiently low as to have negligible effect on the ground state velocity measurements. Signal was observed to be proportional to $I^{1/2}$ as the laser intensity, I , in the sputtering chamber was increased up to about $I = 3 \text{ kW/cm}^2$ as would be expected according to the theory of the "hole burning" by a quasi-monochromatic wave in a line exhibiting substantial inhomogeneous broadening.²³ At higher laser intensity some saturation of the signal was observed along with broadening of the sputtered velocity distribution profile.

Similar measurements were conducted for the set of fine structure components $J = 0-4$ of the ground state multiplet. The velocity distributions and the transitions employed for excitation are displayed in Fig. 3. Vertical scaling is proportional to population of the initial state by the sputtering process as determined in calculations described below. Laser intensity from 20 kW/cm^2 for $J = 4,3,2$ to $>300 \text{ kW/cm}^2$ for $J = 1$ and 0 measurement was used in order to achieve larger signals. At the higher power levels some broadening of the measured distributions is expected. For the ground state case the line through the points is that of the Sigmund-Thompson distribution for atom density sputtered into an infinitesimal solid angle and velocity range $(v, v + dv)$

$$g(v)dv \sim v^2(v^2 + v_b^2)^{-(n+1)} dv \quad (1)$$

with $n=2$, and v_b given by $E_b = 1/2 m v_b^2$ for a given atomic mass m with surface binding energy E_b . In Fig. 3, n is fixed equal to 2 and E_b is taken to be the sublimation energy of iron,²⁴ 4.315 eV giving $v_b = 3.86 \text{ km/s}$. Spatial averaging models for the present geometry show less than 2% shift of the distribution peak to lower velocities. Good agreement with the data is seen except near zero velocity. This is thought to be attributable to fluorescence from atoms excited by laser photons scattered back from the ion gun in a large volume visible to the detector. In fact at very high intensities a strong peak on the negative velocity side could be seen. This problem was greatly alleviated subsequent to these measurements by tightly masking the detection zone to the laser illuminated volume.

The principal conclusion from the set of velocity spectra is that no significant shift in velocity distribution is discernible in the various

initial states which have energy levels up to 0.12 eV above the ground state. Thus density and flux determinations are equivalent, for these levels. There is also the implication that the large variation in sputtered population observed cannot be attributed to the velocity dependence of various rates of excitation or de-excitation processes, as the atoms leave the surface.^{27,28}

There have been previous reports of velocity spectra for the ground ($J=4$ only) state of Fe, with 10 KeV Ar^+ sputtering.^{29,30} Agreement with cascade theory, Eq. (1), was found after substantial averaging over contributing directions³¹ was considered. The data of Ref. 30 show a depletion to the high-velocity side of the distribution peak. The cause is uncertain since atomic transit through the observation volume occurs on the microsecond scale in that and the present work, much slower than the $\sim 6\text{ns}$ $\text{Fe } ^5\text{D}^0$ radiative lifetime. A substantial shift to lower velocities was also observed when a stainless steel target was substituted for iron.²⁹

The relative population of the ground state components $J = 0-4$ was determined from the peak heights of the velocity spectra, assuming signal to vary as $I^{1/2}$ for runs at different laser intensity, I . Data were corrected for branching ratios to different states according to the following method. Laser intensity is assumed sufficiently high that atoms are pumped in negligibly short time from initial state a to upper state b , from which they decay into states c visible to the detector, the Einstein coefficients being A_{ba} and A_{bc} respectively. States c are a subset of the total number of radiative decay channels k , with $A_{bk} = A - A_{ba}$, A being the natural decay rate of the upper state, applicable when the laser is turned off. Then, for a laser on-time of t_1 followed by a further collection interval t_2 with the laser off, the ratio B of detectable fluorescence photons to atoms initially in the lower state a

is given by

$$B = \frac{A_{bc}}{A_{bk}} \left[1 - e^{-\left(\frac{\alpha}{1+\alpha}\right) A_{bk} t_1} \right] + \left(\frac{\alpha}{1+\alpha}\right) \frac{A_{bc}}{A} e^{-\left(\frac{\alpha}{1+\alpha}\right) A_{bk} t_1} [1 - e^{-A t_2}] \quad (2)$$

In Eq. (2), $\alpha = g_b/g_a$ the ratio of upper to initial state degeneracies. Einstein coefficients, given in Table I. were taken from Ref. 32 for the $a^5D - y^5D^0 - a^5F$ pump and detection lines except for the 302.065 and 302.050 nm transitions. These were estimated from line intensity ratios from a hollow cathode lamp and are believed accurate to ~20%. The coefficients A_{bc} in Table I include a weighting factor proportional to the relative transmission of the interference filter used in this work. Known transitions from the upper state to higher lying final states³³ were included in the decay rates A_{bk} and A but are a minor contribution. For the narrowband laser $t_1 = 8$ ns, and $t_2 = 50$ ns were used. The resulting branching ratios B_1 of Table I were then used to calculate the populations given in Table II. for narrowband excitation. These fractions include contributions from cascade from any higher lying states initially produced in the sputtering process.

For comparison, measurements of these relative populations were repeated with a Nd: Yag laser system having a measured UV bandwidth of 52 GHz and pulse length $t_1 = 19$ ns. Experiments with this broadband light were conducted under high saturation conditions ($\sim 10^6$ W/cm²) on most of the available excitation lines. The results, analyzed using the branching ratios B_2 of Table I ($t_2 = 50$ ns) are displayed under broadband excitation in Table II. The two sets of population measurements can be roughly characterized by a Boltzmann temperature of 600K (target at 300K) although the highest lying state shows

higher than expected population on this basis. Systematic population measurements have not yet been made with broadband operation of the ring laser. Preliminary data on the $(J=3)/(J=4)$ ratio, however, is in agreement with Table. II. The principal experimental problem in the present configuration is the lack of an exit port for the laser beam leading to high residual light levels from fluorescence produced in the chamber.

The only published population measurements for comparison is the work of Schweer and Bay³⁴ who performed LFS experiments on Fe atoms ejected from stainless steel by 10 KeV argon ions. They reported relative populations of 1.0, 0.43, 0.21, 0.097 and 0.029 in states $J = 4, 3, 2, 1, 0$ respectively, of the ground state multiplet, corresponding to an approximately Boltzmann distribution of 980K (target: 300K). These excited state fractions are somewhat higher than found in the present work but could be attributable to the differing surface environment of the Fe Atom. Such effects have been seen for higher electronic states, for example, in LFS studies of oxygen monolayer coverage³⁵ and in secondary photon emission³⁶.

The laser system described here incorporates several features desirable for in-situ LFS diagnostics for fusion devices. The intrinsic bandwidth is ideal for direct measurement of velocity distributions characteristic of thermal evaporation. A larger effective bandwidth for monitoring sputtering processes is conveniently available by the use of increased laser intensity. An extended surface area may contribute to the impurity atom signal seen in a given detection volume, with a corresponding shift of the most probable measured velocity to lower values.^{9,35} As discussed in detail in Ref. 9, high resolution, suitable for thermal velocity scans is necessary for distinguishing the erosion mechanism in this case. The high UV power (>50 KW) and the availability of a broadband mode (~50 GHz FWHM) allow absolute density

measurements in high saturation, as in previous pulsed systems,¹⁻⁷ but with sampling rate increased to the 100 Hz regime.

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Table I. Branching Fractions for Excimer and Nd: YAG - Pumped
Laser Excitation of Fe Atoms $a^5D_J + y^5D_J^0$.

Laser line			Radiative Rates			Branching Fractions	
J	J	λ (nm)	A_{ba} (10^8 s^{-1})	A_{bc}	A_{bk}	β_1 Excimer	β_2 Nd:YAG
4	4	302.065	0.67	0.680	0.887	0.399	0.531
3	4	305.909	0.18	0.680	1.373	0.371	0.438
4	3	298.357	0.28	0.638	1.420	0.276	0.362
3	3	302.107	0.456	0.638	1.244	0.315	0.413
2	3	304.760	0.284	0.638	1.416	0.331	0.402
3	2	299.443	0.44	0.521	1.078	0.246	0.338
2	2	302.050	0.19	0.521	1.333	0.262	0.329
1	2	303.739	0.32	0.521	1.198	0.314	0.382
2	1	300.095	0.642	0.591	1.042	0.249	0.360
1	1	301.763	0.0682	0.591	1.616	0.266	0.325
0	1	302.584	0.348	0.591	1.336	0.362	0.416
1	0	300.814	1.07	0.606	0.608	0.194	0.318

Table II. Relative population of the fine-structure components of the a^5D_J ground multiplet of Fe I, from sputtering of polycrystalline iron by 3 keV argon ions.

Atomic State		Relative Population	
J	Energy (cm^{-1})	Narrowband Excitation	Broadband Excitation
4	0	1.00	1.00
3	416	0.33	0.27
2	704	0.083	0.10
1	888	0.039	0.044
0	978	0.020	0.028

Figure Captions

- Fig. 1 Layout of the main components of the narrowband, high-power UV laser system.
- Fig. 2 A velocity distribution run as recorded by the data system. Top trace: Fe laser-induced fluorescence signal, proportional to ground state atom density. Middle trace: Optogalvanic signal produced by the scanning laser. Bottom trace: Fixed-wavelength transmission, monitoring the scanning laser frequency. The interval between fringes corresponds to a 1.78 km/s velocity shift.
- Fig. 3. Velocity distributions for Fe atoms sputtered in the $J = 0-4$ levels of the ground multiplet and laser-excited on the transitions indicated. Ordinates were scaled to the relative populations produced by the sputtering process, as calculated in the text. Solid curve: Sigmund-Thompson distribution for $n = 2$ and $E_b = 4.315$ eV (iron sublimation energy) with no spatial averaging.

LABORATORY DATA IRON GROUND STATE

LASER FLUORESCENCE FROM SPUTTERED IRON