Studies of thorium and ytterbium ion trap loading from laser ablation for gravity monitoring with nuclear clocks

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Compact and robust ion traps for thorium are enabling technology for the next generation of atomic clocks based on a low-lying nuclear transition in thorium-229 atoms. We aim to study a laser ablation loading process for single thorium ions in a radio-frequency electromagnetic trap. We use a linear Paul trap loaded by laser ablation from metal targets. Detection of ions is based on a modified mass spectrometer and a channeltron with single-ion sensitivity. We successfully detected Yb+, Th+, and Th2+ ions from plasma plumes, studied their yield evolution, and compared the loading to a quadrupole ion trap. The thorium ablation yield shows a strong depletion, suggesting that we have ablated oxide layers from the surface and the ions were a result of the plasma plume evolution and collisions. Our results are in good agreement with similar experiments for other elements and their oxides.

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I. INTRODUCTION

Gravity can be monitored with atomic clocks through a technique known as chronometric levelling [1]. It is a well known consequence of general relativity that clocks sitting in different gravitational potentials have different ticking rates. In recent years, rapid progress in atomic clock networks make this kind of gravity sensor feasible [2]. Clock networks can provide a new way for ground-based gravimetry at the Earth’s surface, allowing us to monitor minute drifts in the gravity anomalies. Recently, such networks have been demonstrated with two state-of-the-art optical atomic clocks compared via optical fibre link [3–6], including demonstrations of a transportable, optical atomic clock [7]. The best of lab-based optical clocks are capable of detecting down to 1 cm differences between clock altitudes, which is already beyond geodetic limit [8]. The optical fibre links are no longer a limiting factor for such networks, as a stability of frequency transfer down to $10^{-20}$ has been demonstrated [9]. Even optical links hundreds or thousands of kilometres long are able to perform well enough for requirements of chronometric levelling with millimetre precision [9, 10]. The portability of optical clocks and maintaining their performance out-of-the-lab is one of the main obstacles we face in the development of a true gravity observing clock network. To reach the level of stability and accuracy down to $10^{-18}$, the best available clocks must be maintained at cryogenictemperatures [11], and portable optical clocks still lack the accuracy and stability of laboratory-based clocks [12], but advances in the field are being made rapidly [13].

A nuclear clock is an alternative approach pursued to overcome the challenges of the best present optical clocks. Thorium-229 is the isotope with a lowest-energy nuclear transition that could serve for clocking instead of an electronic transition. The transition has been predicted, and its existence has been debated for a long time before recent final experimental confirmation [14–16]. A clock based on a nuclear clocking instead of an electronic transition. The transition has been predicted, and its existence has been debated for a long time before recent final experimental confirmation [14–16]. A clock based on a nuclear transition in a single $^{229}$Th ion housed in an electromagnetic trap is expected to allow frequency measurements of unprecedented accuracy, and stability better than $10^{-18}$ [17]. That is at least an order of magnitude better than the best optical clocks in use now. Moreover, the single-ion clock has great potential to be highly miniaturised [15].

The low-lying nuclear isomer transition provides us with a clock transition that is uncoupled from most of the environmental effects that plague existing atomic clocks. In addition to the spectacular expected fractional accuracy of $10^{-19}$, the $^{229}$Th$^{3+}$ system is attractive from a number of technical perspectives [19]. Design and simulation work has shown that a trapped Th$^{3+}$ ion could be developed in a compact and relatively low-cost package, making field deployment in a dispersed network achievable. In addition, it has been shown that the Th$^{3+}$ is an excellent system for precision measurement of the drift in the fine structure constant [20]. Efforts to directly detect the nuclear isomer transition are underway, and several groups have joined this effort recently [15]. To this date, a few groups worldwide have demonstrated thorium trapping in ion traps [21–27].

Laser ablation of solids is a technique widely used in material processing and deposition where precise and effective material removal from the surface is crucial [28]. This technique can also be employed for delivering ions to an RF trap by vaporization and ionization of material from the metal surface [29]. Short laser pulses in the nanosecond range are the most effective in terms of the level of ionization of the ablated material [30]. Nanosecond laser ablation offers temporal control over trap loading and is a particularly attractive method for experiments with micro traps [31, 32]. The trap loading of ions into RF traps was demonstrated for Ca [33], Sr [34], and Th atoms [35, 36].

Systematic studies of the ablation process and trap loading are difficult because of changes in the metal surface caused by the removal of the material from the spot. After repeated pulses, a crater in the metal surface appears and the ablation yield diminishes. To describe this process quantitatively we performed a series of measurements where the number of ions from the same spot was monitored over long times. Our findings about the evolution of the yield from the laser ablation are complementary to previous findings, and we present a more thorough examination of pulse yield depletion.

The nuclear clock is not the only interesting application of thorium ion traps. Single ions can be used as very sensitive probes of the electric field, as was recently demonstrated for ytterbium ions [37]. A potential application of such single ion-based force sensors is the manipulation and investigation of dipole moments in bio-molecules [38]. The UV light used in the ytterbium ion force sensor can be harmful to bio-molecules, and thus might not be very useful. In contrast, Th$^{3+}$ ion has atomic transitions used for cooling and imaging in the red and infrared parts of the electromagnetic spectrum [21], making it very attractive as a potential force sensor for large bio-molecules.
Figure 1. A rendering of the experimental setup. The ion trap is placed inside the central vacuum chamber. The port on the left of the chamber leads to the RGA based ion detection system. The axis of the ion trap is aligned with the axis of the RGA’s mass filter rods. The port on the right-hand side connects to the vacuum control and detection system. Top and bottom 4.5-inch ports and front smaller port are closed with windows for optical access. The rear smaller port serves for electrical connection to the ion trap.

II. METHODS AND EXPERIMENTAL SETUP

The experimental set-up consists of an ion trap with an ion detector and a laser system for ablation. The ion trap, the ablation targets, and the ion detection system are under ultra-high vacuum, pumped by turbomolecular and ion pumps. Fig. 1 shows the rendering of the experimental setup. A spherical cube vacuum chamber (Kimball Physics) holds the ion trap and laser ablation targets. The parts are mounted by groove grabbers to the internal grooves of the vacuum ports. The optical access to the ion trap is provided through the top and bottom 4.5 inch diameter ports and one smaller side port. The viewports are equipped with anti-reflection coated glass windows. The vacuum pumps, gauges, and detection system are connected by three 2.75 inch ports. The remaining fourth port allows additional optical access for laser cooling and probing of ions in the trap.

A. Ion trap

Our ion trap is a standard radio-frequency (RF) linear Paul trap [39] modified from the one formerly used for experiments with Yb ions [40, 41]. The rod diameters, spacing, and other crucial dimensions are listed in table I. The RF field is provided from a digital signal generator (RIGOL DG4162), amplified by 30 dB, and transmitted to the trap via a helical resonator for impedance matching. The trap and helical resonator are resonant at a frequency around 6.2 MHz, allowing us to confine different ionic states of thorium and ytterbium by tuning the amplitude of the drive RF signal and the DC bias applied between adjacent rods.
Quadrupole rods radii $r_0$ 4.508 mm
Quadrupole rods length $l_0$ 51.5 mm
Rods and end cap wire radius $r_w$ 787.5 µm
End cap distance from centre $z_0$ 20 mm
End cap radius $r_z$ 7 mm

Table I. Quadrupole radius refers to the distance from the geometric centre of the square formed by the quadrupoles (trap axis) to a quadrupole rod. Quadrupole length is the length of the rods. Wire radius is half the wire gauge used for the quadrupole and end caps. End cap distance from the centre is half the distance between the two end caps. End cap radius is the distance from the perimeter of the circular end cap nodes to their centre.

B. Laser system for ablation of metal targets

The laser ablation as a method for trap loading is most commonly applied to elements like thorium with very high evaporation temperature. Our laser plasma ablation setup is similar to the one described in [35]. The ablating laser source is a nanosecond pulsed nitrogen laser (Stanford Research Systems NL100). It produces a 3.5 ns pulse with an energy of 170 µJ. The wavelength is 337.1 nm. The repetition rate of the laser can be tuned between 1 - 20 Hz. The output beam was focused to a spot of 350 µm diameter. The laser beam enters via the top window of the chamber and is focused on the targets that are to be ablated. Taking into account the absorption coefficient of thorium, our pulsed laser generates a maximum fluence of about 0.35 J/cm².

C. Thorium and ytterbium targets

A series of preliminary experiments were tried to electrodeposit thorium on a rectangular (2 cm) stainless steel plate (Kimpball Physics eV parts). The electrodeposition was carried out using 0.02M thorium nitrate water solution. We built a deposition cell with a platinum electrode and the ammonia hydroxide for pH control adapting the method for depositing uranium described in [42]. According to weighing the plate before and after the process we deposited 0.05 g of material on the plate. A laser ablation signal from a target prepared in this way was observed visually (lasting a few pulses) and used for tests of our detection system at that time. The thorium signature was observed. However, the plate size was not compatible with our ion trap, it was not possible to insert the plate between the trap rods.

We then changed to a pure thorium-232 metal wire target. All the results presented here are collected with that target. A piece of thorium wire approximately 1 mm long was spot-welded to the surface of an aluminium strut inserted throughout the ceramic holder. The thorium 0.22 mm wire is naturally monoisotopic thorium with 99.5% purity (The Goodfellow supplier). Similarly, a piece of ytterbium wire with natural abundance is placed by the side of the Th target.

D. Ion detection and plasma diagnostics

We developed a simple non-optical detection system which was sensitive enough to detect a single ion signal. We utilized a commercial residual gas analyzer (RGA) to serve as the ions filter and detector (Extorr XT300M). The ions were detected by the channeltron with the electron multiplier. The mass filter from the RGA has been used to guide the ion ejected from our ion trap towards the channeltron. The RGA allows filtering ions up to 300 amu. To make the RGA serve as a detector we removed the grid and filament at the RGA’s entrance. Normally, these elements ionize the gas to be analyzed, but in our case, we wanted to detect already ionized atoms. When the grid and the filament were removed, the ions from our trap were able to enter freely and to be guided by the mass filter of the RGA. We also added a switching mechanism for the RF field to RGA rods. In this way, we can switch off the filtering in RGA completely and filter charge states only with our Paul trap. The scheme in Fig. 2 illustrates the relative positions of the ion trap and detection system elements.

We observed that the number of ions guided by the quadrupole mass filter which reached the detector varied periodically. One explanation of this behaviour might be the phase difference of trap drive fre-
Figure 2. A schematic illustrating the relative positions of the laser plasma creation, the ion trap, and RGA based ions detector with the channeltron. The ions created in the ablation process travel along the path marked towards the detector.

Figure 3. Example of the ion current detected in the channeltron as a function of time (green lines). The repetition rate of the ablation laser is 1Hz, the ion trap driving frequency 6.24 MHz, ion signal data points were averaged over 500ms. The blue line is a fit with periodic signal $\sin^2(2\pi f t + \phi)$ with frequency $f = 31.58$ mHz corresponding to the beat frequency between ablation and trap frequency.

III. RESULTS

In our experimental setup, we observed laser ablation from metallic targets of Yb and Th. The two ablation plumes could be visually easily distinguished - blue fluorescence from the plasma plume of Yb and white from Th. The Ytterbium was much brighter to the eye and lasted longer, while the thorium ablation was much fainter and short-lived. This was consistent with the expected higher threshold for laser ablation of thorium. To develop a thorough understanding of these observations we present here a detailed quantitative analysis.
A. Trapping and detection of Yb+, Th+ and Th2+ states

The laser ablation plume contains both neutral and ionized atoms. The ratio of the constituents of the plume depends on the energy density of the ablation laser. The first check of our ablation setup was to test it on a ytterbium target without the ion trap. The produced plasma plume was first analyzed by the unmodified RGA. The products of ablation were ionized by the RGA filament and were accelerated by the entrance grid voltages. This way we reproduced the expected natural isotope abundance of ytterbium. The graph in figure 4 shows the result of this measurement with Yb atoms ionized at the RGA entrance.

We also confirmed the signal from all isotopes of ytterbium without the high voltage and filament at the entrance of the RGA, we only measured those ions produced by the ablation. This measurement was repeated with the ion trap driving field on and the RGA guiding rods off, so the only ions reaching the detector were filtered by the ion trap. The method was tested on both ytterbium and thorium metal targets.

The rods of our ion trap served as a mass filter before the ions entered the detector. We detected a clear signal from Yb\(^{1+}\), Th\(^{1+}\) and Th\(^{2+}\) ions. These results are presented in figure 5. Both graphs are a result of the scan of the RGA around mass to charge ratio during the laser ablation of metal targets. The ions created in this process were filtered by our ion trap first and then detected. We have only confirmed a signal for singly and double ionised thorium. We were ablating ions at 20 Hz repetition while scanning the detector. Each point represented as a vertical line is an integration over 50 ms.

The first assumption about the threshold for the laser plasma creation is the energy fluence required for vaporization from the metal surface. This threshold for thorium is \(0.99 \text{ J/cm}^2\) [44]. It is based on a purely thermal model of laser ablation. Other models predict that the volume of the material from the metal surface scales linearly with pulse energy [45]. We might expect that the fluence required for the ionization of metal atoms would be even higher; some energy from the pulse must be absorbed by the atoms to release electrons. To estimate the threshold for ionization, the energy density in the pulse must be compared with the ionization energy. We estimate our peak fluence to be only a fraction of the vaporization threshold, less than \(0.7 \text{ J/cm}^2\). Regardless of the nominally sub-threshold fluence level, we observed the ablation of thorium targets. That may explain the fact that what we ablate is not the thorium metal, but rather the layer of oxides on the surface. The thorium ions could be created during the plasma plume evolution afterwards. This agrees with the reports of other groups, however, was not
Figure 5. A signal confirming successful creation and RF-trap loading of Yb1+, Th1+ and Th2+ ions using our laser ablation method and detection scheme. The plots show ion current in the electron multiplier during the \( m/z \) scan of the RGA detector (horizontal axes).

concluded earlier. In our setup, we obtained lower laser intensity than reported in [35], but still measured successful creation of singly and doubly ionised thorium. Successful trap loading of triply ionised thorium has been reported in [23] but with a more powerful laser source and intensities reaching 6 GW/cm\(^2\).

B. Stability plots for thorium charge states

The exploration of the parameter space of our ion trap confirms the scaling between charge states of thorium. Using the method of data acquisition shown in figure 2, we scanned the RF voltage amplitude \( V_{RF} \) and DC bias voltage applied to the trap electrodes \( U_{DC} \). The radial motion of the ions on the quadruple trap is governed the Mathieu equation [39] and we can extract two parameters called \( a \) and \( q \) that are proportional to \( U_{DC} \) and \( V_{RF} \) and other constant trap parameters. The trap parameters were scanned in 20 V steps for both RF and DC fields, while the detector parameters were fixed. For each data point, we measured the ion current in the detector tuned to the charge state of interest. The results are presented in figure 6. The amplitude \( V_{RF} \) is inferred from the amplitude of the signal generator, accounting for the amplifier and independently measured losses in the helical resonator.

The results show that, while there may be a systematic shift in the overall scale due to incomplete quantitative characterisation of the trap, the stability regions do scale as expected when comparing the behaviour of different \( m/z \) ratios. Of particular importance is the scaling between Th\(^{2+}\) and Th\(^{1+}\) stability regions, which appear to follow the expected factor of two in the RF and DC voltages. Despite a rigorous search, we were unable to detect any signal from Th\(^{3+}\). Some signal in the \( m/z \) region corresponding to triply ionised thorium was detected, but this signal showed no scaling behaviour corresponding to the expected stability region of Th\(^{3+}\), indicating that it must have been produced by some background signal whose origin we were unable to isolate.

C. Laser ablation yield evolution

The yield of the ablation pulse changes over the number of pulses in the same spot on the target surface. The ablation decays are shown in Fig. 7 for the ytterbium (a) and thorium (b) targets. The ablation laser runs at 1 Hz rate with an integration time of 500 ms in our detector. The blue points represent the data after binning and averaging, while the red line is the fitted curve of the exponential decay. We observed a \( 1/e \) decay after more than 1500 pulses in the same spot. The experimental runs with higher repetition rates and shorter exposures were noisier but showed similar behaviour. The data were binned and averaged with a bin size of 25 points for Yb and 5 points for Th.
Figure 6. The contour plots of the ion current as a function of the parameters $a$ and $q$ of the trap for Th$^{1+}$ and Th$^{2+}$ charge states. The horizontal axes represent the $q$ parameter proportional to the amplitude of the driving voltage of the ion trap at 6.2 MHz. The vertical axes represent $a$-parameter linked to the DC bias voltage applied to the trap electrodes. The color bar indicates the ion current at the detector (arbitrary log scale), a measure of the number of ions that travelled through the ion trap. The predicted boundaries of stability regions from Mathieu equations are indicated by white lines.

Figure 7. Time dependence of the ytterbium (a) and thorium (b) ions yield. The laser is moved to an undamaged location on the ablation target, and a sequence of pulses is repeated on the same spot. The observation of a significant decrease in the ablation yield is contrary to previous studies which have suggested that decay process is not significant and for solid metal targets ablation yield remains constant [30]. Some results of ablation yield evolution for Sr, Yb and their oxides were also reported in [35]. However, they recorded the yield evolution for Yb up to 500 pulses and did not observe significant decay. In contrast to these reports, we have observed laser ablation from thorium and ytterbium for much longer. Laser ablation from a Yb target was investigated up to a few thousand pulses. The significant drop in the yield amount was observed after 1500 pulses (reduction by $1/e$), which is consistent with previous studies; the authors could have missed a yield drop when observing only for 500 pulses. However, the exponential decay for thorium target was evident much more quickly ($1/e$ decay after 150 pulses). This might seem surprising unless we consider that the ablation only comes from the oxide layer on the metal surface. That explains why it disappears so quickly, and the decay is in good agreement with other papers describing the yield evolution for metals and metal oxides. This also explains why we see the thorium ion...
signal, even though the ablation laser operates below the expected laser ablation threshold for thorium metal.

IV. CONCLUSIONS

We have proposed a gravity sensing method with a network of nuclear clocks based on thorium isomer transition and discussed experimental efforts toward its realization. The first version of the ion trap was designed and built along with the vacuum enclosure and detection system. At the initial stage of research, we investigated methods of delivering thorium ions into the trap. We conducted a series of laser ablation experiments to determine a reliable and efficient way to deliver ions from metal targets. We showed that even a simple and fairly weak ablation laser can lead to thorium ionisation lasting long enough to be effectively injected to an RF trap. Our results are in good agreement with previously reported trap loading with thorium, but the extended studies of yield from laser ablation suggest that only the oxide layers contribute to the Th ion production by UV nitrogen laser pulses. The findings are important for optimal loading of miniaturized thorium traps, which are expected to be the crucial component of future atomic clocks. Moreover, our trap setup and detection system have the potential for studies of co-trapped large bio-molecules.

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DISCLOSURES

The authors declare no conflicts of interest.

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