

Tuesday Evening Poster Sessions, November 14, 2006

Vacuum Technology

Room 3rd Floor Lobby - Session VT-TuP

Vacuum Technology Poster Session

VT-TuP1 Vacuum Pump Oil Testing to Minimize Oil Waste at the National Synchrotron Light Source*, C.L. Foerster, E.-P. Hu, E. Haas, Brookhaven National Laboratory

An oil-testing project was established three and a half years ago to determine if synthetic vacuum pump oil could be used effectively to reduce some of oil waste produced during normal operation of storage rings and beam lines in the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory (BNL). More than two hundred oil-sealed rotary vane pumps are currently used at the NSLS facility, such that a longer oil change interval would greatly reduce maintenance costs as well as oil waste. Prior to this project the mechanical vacuum pump oil waste was approximately 75 gallons per year. Two basic types of vacuum pump oils, mineral and synthetic, are being tested for a direct comparison. Three two-stage mechanical pumps were set up and run simultaneously. Convectron gauges, cold cathode gauges, and isolation valves were connected to a central vacuum chamber with a common inlet pressure control and an RGA sampling valve. To simulate long-term mechanical pump operation, the system gas load was constantly adjusted at an inlet pressure of 500 mTorr using an air bleed valve. This inlet pressure was suggested by the suppliers of vacuum pump oil to expedite the oil viscosity change, acid buildup, and pump-wear debris production under minimal risk of oil backstreaming in the test pump. After three and a half years of continuous running there have been no significant changes in either of the oil types. Detailed test data for the resulting oil properties, oil degradation, visual comparison, vacuum conditions, and pump characteristics will be presented for evaluation of the pump oils used at NSLS and for estimation of the resulting oil waste reduction. @FootnoteText@*This research is supported by the U.S. DOE under the contract DE-AC02-98CH10886.

VT-TuP2 A Diagnostic Technique for Particulate Deposits in the Pipes within the CVD System, J.Y. Yun, Korea Research Institute of Standards and Science, S. Korea; J.H. Lee, D.K. Moon, Konkuk University, Korea; S.W. Kang, Korea Research Institute of Standards and Science, South Korea; D.-J. Seong, Y.H. Shin, Korea Research Institute of Standards and Science

In order to investigate the pipe clogging due to the particulate deposits in the CVD system, the various tests were conducted using the ultrasonic sensor and the vibration sensor system. At first, the pipe was examined with the ultrasonic sensor where the peak amplitude was observed to decrease as the particulate deposits increased. This is only applicable to the early stage where the particulate deposits are absorbing the ultrasonic energy. However, this trend would only last for a short period of time. The ultrasonic diagnostics would not be effective anymore when such samples are placed in a vacuum for two weeks. In this case, no particulate deposits were identified through the re-examination. This is attributed to particulate deposits drying off from the wall after some time, hindering particulate deposits from absorbing the ultrasonic energy and reflecting the ultrasonic wave back instead. As a result, this method will fail because it will not be able to differentiate the measurement from the reference sample. On the other hand, the vibration diagnostics system was able to show distinct differences depending on the amount of particulate deposits. The amount of particulate deposits could be detected by closely observing that the clean pipe achieves a high-level frequency with the impulse given by the vibration generator, while this frequency greatly reduces as the particulate deposits inside the pipes increase. It is believed that the vibration method can be applied to examine the pipes regardless of the conditions whether or not the particulate deposits are attached to the inner wall of pipes. This research also suggests that this method could be further investigated for effective application in monitoring the semi-conductor production line.

VT-TuP4 Specification Study of the Two Different Types Quadrupole Mass Spectrometer in Different Configurations and Operating Parameters, S.S. Hong, Korea Research Institute of Standards and Science (KRISS), Rep. of Korea; I. Tanvir, Pakistan Vacuum Society; S.W. Kang, Korea Research Institute of Standards and Science (KRISS), South Korea; Y.H. Shin, Korea Research Institute of Standards and Science (KRISS)

Quadrupole mass spectrometers (QMS) are used in many vacuum systems for leak checking and general monitoring of background gases. The QMS performs mainly three functions: it ionizes a gas, it separates the resulting molecular ions as a function of the mass-to-charge ratios and it detects the

ions. @FootnoteText@ Partial pressures as measurement of concentration of individual molecule species have been increasing interest in many semiconductor and display manufacturing applications. The Korean Research Institute of Standard and Science (KRISS) has newly developed a QMS calibration system for the investigation of all the QMSs performances. It consist of a vacuum chamber which is divided by orifice conductance, ultra-high pumping system, vacuum gauging instruments, and gas mixture chambers. And the system is also equipped with bake-out heater. Extractor ionization gauges and spinning rotor gauges have been used as accurate total pressure measurements. QMS sensitivity is the measurement response as a ratio of the change in spectrum peak height to corresponding change in total pressure due the change in partial pressure of a particular species. We will present the sensitivities dependences of the different types of two QMSs according to the operating parameter such as ion source, ion energy and ion detector. We will also present the ion current in combination some ionization energies to investigate behaviors of the either instrument's configuration, peak resolution and peak maximum behaviours, the response of number of cycle and etc. @FootnoteText@ @Footnote 1@ D. J. Mitchell, OJ. Vac. Sci. Technol. A 3 (3) May/June, 527 (1993).

VT-TuP6 A Two-Stage Flow Divider System for the Test and Calibration of Vacuum Gauges, H. Yoshida, K. Arai, H. Akimichi, M. Hirata, AIST, Japan

Rapid generation of a stable vacuum pressure is necessary to evaluate characteristics of vacuum gauges. A two-stage flow divider system was developed to generate the stable vacuum pressure in high and ultra-high vacuum. This system mainly consisted of three parts, chamber 1, 2 and 3, which were connected in series using capillary A and capillary B. Chamber 2 for the gauge test was evacuated by a turbo molecular pump thorough orifice with the conductance of about 80 l/s. Chamber 1 is also evacuated by a turbo molecular pump, of which effective pumping speed is about 10 l/s, to reduce the gas flow rate from chamber 0 to chamber 2. The pressure P@sub 2@ in chamber 2 could be generated precisely by adjusting the pressure P@sub 0@ in chamber 0 using a pressure controller. P@sub 2@ was determined by P@sub 0@, the conductance of capillary A and B, and the effective pumping speed in chamber 1 and chamber 2. As P@sub 0@ was changed from 3.0x10@super 2@ Pa to 1.6x10@super 5@ Pa, P@sub 2@ was generated from 2.4x10@super -7@ Pa to 5.7x10@super -3@ Pa. P@sub 2@ reached stable within a few seconds and the fluctuation for 2.5 minutes was from 0.01% to 0.2%, which mainly depended on that of P@sub 0@. In addition, it was confirmed that the gas flow through capillary B was molecular flow because the pressure ratio of P@sub 2@ to P@sub 1@ was constant. Therefore, P@sub 2@ in lower pressure could be quantitatively determined from P@sub 1@ by using the P@sub 2@/P@sub 1@ ratio. This system is useful to evaluate characteristics of vacuum gauges. For example, it was found that the indication of ionization gauge was unstable for 600 s from the pressure setting.

VT-TuP7 ROR(t) Method in Vacuum Diagnostic, J. Zhou, H. Gao, D. Paul, Applied Materials Inc.

Rate of rise (ROR) in pressure is a key indicator used to diagnose vacuum issues in semiconductor industry. Normally, the final value of measurement will be compared with a preset pass/fail specification. A more useful approach is to evaluate the dynamic moving-average, and the value of this method is presented in this paper. In comparison to the go/no-go data provided by the final vacuum reading, the graph of vacuum over time can provide information on vacuum chamber performance: for example, if it had a leak or an outgassing problem. Thus, this approach minimizes downtime by avoiding unnecessary retests and parts replacement in troubleshooting. In this work, data was collected from more than 100 high vacuum chambers, and three basic types of ROR(t) curves were identified. Also, a controlled-leak experiment was performed to verify the correlation between the ROR(t) curves and chamber status in leak or outgassing. When a controlled leak was added to a leak-tight baseline chamber, the leak not only increased the chamber pressure, but also altered the shape and smoothness of the ROR(t) curve. When the leak was removed, the curve returned to the original shape and smoothness after a certain delay time. The ROR(t) curve signature reflected an evolution process of all gases loaded within the test chamber, which was sensed by the transducer (ion gauge). In a vacuum leak condition, the diffusion process during ROR will experience interference from the normally point-source leak, and this indicator may be especially useful in high volume manufacturing. The possibility of an automatic vacuum diagnostic employing the ROR(t) method will also be discussed.

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VT-TuP8 Viscosity Measurement of Ozone-oxygen Gas Mixture with a Quartz-Friction Sensor, A. Kurokawa, Y. Kobayashi, AIST, Japan

We would report on the viscosity of ozone-oxygen gas mixture. To measure the viscosity we developed a quartz-friction sensor method which was based on the principle that the resonance impedance of the vibrating crystal oscillator depends on the viscosity of the surrounding gas. This is an essential technique because the highly concentrated ozone gas is very reactive and easy to decompose by light absorption and heating. With the following procedure we evaluated the viscosity of the gas. The crystal oscillator was vibrated at its characteristic frequency which was around 32 kHz. The oscillator was set in the flow of ozone-oxygen gas mixture, and its impedance was monitored. During the measurement the gas pressure was measured by a capacitance manometer to cancel the pressure dependence of the impedance of the oscillator. We used the approximation that the variation of the impedance related to the viscosity of the gas. The impedance variation was defined with the impedance origin obtained at the vacuum pressure. The approximation curve was derived by the fitting to viscosity-known gases such as argon, nitrogen, oxygen, and consequently the measurement of impedance variation could give the viscosity. The highly-concentrated ozone gas was supplied from the ozone-gas generator in which 5 vol% ozone gas generated by electric discharge was condensed by selective absorption on cooled silica gel. All of the gas piping for ozone-gas had been electrochemically polished and well stabilized to minimize the decomposition of the ozone gas on the wall surface. Finally we derived the viscosity of the mixed gas with 20 vol% ozone in the 10 kPa to 100 kPa range of the gas pressure and that with 70 vol% in the 1 kPa to 10 kPa range.

VT-TuP9 Comparisons of Vacuum Standards Among NMIJ, NIST, IMGCC and KRISS in Medium Vacuum, H. Akimichi, M. Hirata, NMIJ AIST, Japan; P.J. Abbott, National Institute of Standards and Technology; M. Bergoglio, A. Calcatelli, IMGCC, Italy; S.S. Hong, KRISS, Korea, Rep. of Korea

Comparison of vacuum standards was carried out among the National Metrology Institute of Japan (NMIJ), the National Institute of Standards and Technology (NIST), the Istituto di Metrologia "G. Colonnetti" (IMGCC) and the Korea Research Institute of Standards and Science (KRISS) from March 2004 to September 2005. Pressure range was from 0.01 to 1 Pa. Test gas was nitrogen. Spinning rotor gauges were used as transfer standard. After the calibration of the gauge by a static expansion system at NMIJ, rotors of the gauge were removed from their thimbles and packed in clean aluminum foil or clean dust free paper. They were transported to another NMI by hand and calibrated by their primary system, a dynamic system at NIST, a dynamic system and a static system at IMGCC and a dynamic system at KRISS. Rotors were driven by electro units and coil heads at each NMIs. After the calibration, rotors were packed again and brought back to NMIJ by hand for re-calibration. Comparisons were done among the effective accommodation coefficient of rotors obtained by calibration at each NMI. While relatively large differences in the coefficient up to 2% (depending rotors) were observed compared with uncertainty at each NMIs, the difference was mainly caused by the change in the coefficient of the rotor during their packing and/or transportation. Mean value of coefficients of 2-6 rotors showed good agreement within 0.5% reasonably estimated from uncertainty of each NMIs. This result shows that the primary standards of NMIs seem to be equivalent to each other. It is important to clarify the way to carry the gauge without change in its accommodation coefficient.

VT-TuP10 Influences of Handling Procedures on the Accommodation Coefficient of a Spinning Rotor Gauge, M. Hirata, E. Komatsu, H. Akimichi, NMIJ AIST, Japan

A spinning rotor gauge is widely used as a reference standard and as a transfer standard. Stability of the effective accommodation coefficient of the rotor in the gauge is very important for these applications. The value of the coefficient is very stable in operation mode. Though the gauge was turned switch to on and off several times, the relative value of two gauges was constant within 0.05 % - 0.1 % corresponding to the limit by drift and fluctuation of their offset. But significant change in the value is some times observed by transportation. For example, change in the value up to 2% was observed during the comparison of vacuum standard among NMIJ, NIST, IMGCC and KRISS. In this study, we examined the influence of handling procedures on the value. In the case that the sensing head was mounted and demounted from the thimble, the value changes about 0.3 % - 0.5 % occasionally. By the washing of the rotor by alcohol and water, the value changes about 2 % - 3 %. Scrubbing the rotor inside the thimble by moving a magnet externally caused about 1 % change in it. Putting the rotor out of and into the thimble also caused about 1 % change in it some times. Even if the rotor was handled with spring-loaded device, in which the rotor is

moved and fixed by a spring, used for the comparison of vacuum standards in Europe countries, it changes also about 0.3 % - 0.5 % occasionally. These results show that it is depend on the surface condition of the rotor and scrubbing the rotor inside the thimble causes also unexpected change in it. Finally stability within 0.1% in it was obtained by the transportation of the rotor together with the thimble and the sensing head.

VT-TuP11 Measurement of the Photon Stimulated Desorption for the Aluminum Chambers with Ti-Zr-V NEG-coating, G.Y. Hsiung, C.Y. Yang, C.M. Cheng, C.L. Chen, C.K. Chan, NSRRC, Taiwan; J.R. Chen, NSRRC and NTHU, Taiwan

Several NEG-coated aluminum chambers have been considered for the injection section of the 1.5 GeV Taiwan Light Source (TLS) for improving the effective pumping speed. The performances of the NEG chambers including the pumping capabilities, the outgassing behaviors, the electron yield, the dusts, the reliabilities after activations and the long term photon exposures by synchrotron radiation, etc. will be inspected prior to the installation. The tests for the NEG chambers are performed at the 19B1 white light beam line of TLS, 2.14 keV of the critical photon energy, for the photon exposure experiments. A residual gas analyzer has been installed for measuring the outgassing rate and the yield of the photon stimulated desorption (PSD) for the chambers coated with the NEG film of Ti-Zr-V at 0.5 ~ 1 micron in thickness. Activations of the NEG film at both 180 °C for 24 hours and 200 °C for 2 hours have been made for the test. The behaviors of the outgas including the hydrogen, methane, krypton, and carbon monoxide, desorbed during the activations and PSD exposures are interesting throughout the experiments. The measured yields of PSD are lower than 1.0E-4 molecules/photon and reduced to 100 times lower after the exposure with > 100 Ah of the accumulated beam dose. The residual gases are mostly the hydrogen rather than methane or others. More measurements on the desorption of methane and krypton, the photoelectron yield, the dust, for the Al NEG-chambers are inspected and the results will be discussed.

VT-TuP12 Viscosity of Hydrogen-Methane Mixed Gas, Y. Kobayashi, A. Kurokawa, National Institute of Advanced Industrial Science and Technology, Japan; M. Hirata, Shibaura Institute of Technology, Japan

In order to reduce CO₂ emission, EU started in May, 2004, an experimental approach named "Naturalhy Project" to transport hydrogen by mixing with existing high pressure natural gas pipelines. Naturalhy means a mixture of hydrogen and natural gas. Properties of Hydrogen-Methane mixed gas should be investigated much more because Hydrogen-Methane mixed gas is predicted to be an energy carrier to replace methane in the near future. Thus, our result would be useful for those who are developing pumps, compressors, or Mass Flow Controllers (MFC) for the next generation energy systems. This paper is considered to be the first report on the viscosity measurement of Hydrogen-Methane mixed gas. To measure the viscosity, the authors conducted two methods for comparison. The first method is the ordinary way that measures pressure drop in laminar pipe flow. The authors prepared a chemically polished, ultra clean and smooth tube and carefully measured pressure drop between upstream and downstream using a capacitance manometer. The second method measures drag acting on the quartz friction gauge in a gas atmosphere. The quartz friction gauge is sensitive to pressure, temperature and viscosity. To achieve proper conditions, the experiments are held in ultra clean tubes that have reached a constant and uniform temperature in a bath. The impedance, which correlates with drag acting on the quartz friction gauge, is measured for each gas. The authors computed viscosity of the gas from this drag by solving the Navier-Stokes equation. This study concluded as follows: (1) The authors give viscosity of Hydrogen-Methane mixed gas for system designers. (2) The authors verify the use of the quartz friction gauge as a viscosity measurement system.

VT-TuP13 Outgassing Rates of Stainless Steel Pipes Treated with 400°C Air Bake, X. Liu, K.W. Smolenski, Y. Li, C.K. Sinclair, Cornell University

Creating an extreme-high vacuum environment is essential to the operational lifetime of the photo-cathode in the high voltage DC electron gun of the Cornell Energy Recovery Linac project. The key is to achieve an ultra-low hydrogen outgassing rate (OR) of the chamber material. Studies have reported dramatic decreases in hydrogen OR for stainless steels (SST) after a 400°C air bake. In this paper, we report the results of 400°C air bakeout of 304L SST tubes. The tested SST tubes are 6" in diameter and 40" long, with 8" ConFlat flanges welded to both ends. The tubes were cut from long tubes of the same production lot to assure consistent material properties and history. All bakeouts were carried out in a specially built oven to provide uniform temperature. The SST ORs were measured by two

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methods. A spinning rotor gauge was used for a rate-of-rise method by valving out the tube from active pumps and gauges. For a throughput measurement, the tube was connected to a measurement chamber (MC) through a small orifice. The MC is pumped by an ion pump through a second orifice, to give a known pumping speed, and is equipped with a cold cathode ion gauge and a residual gas analyzer. The two methods agree very well. For each sample SST tube, ORs were measured after 150°C and 250°C vacuum bakeout both before and after a 400°C air bake. We have observed as much as a factor of three of reduction in the SST ORs, with a lowest measured OR of 3×10^{-13} Torr l s/cm². This reduction in SST OR is significant, though much smaller than other reported values. Evidence also shows that bakeout history has a strong effect; higher bakeout temperature may result in higher outgassing rate.

VT-TuP14 The Small Quadrupole Mass Spectrometer for the Pressure Range Over 0.1Pa, Y. Matsumoto, ULVAC Inc. Japan, Japan; K. Yamamuro, ULVAC Inc. Japan; N. Takahashi, ULVAC Inc. Japan, Japan; N. Mizutani, T. Nakajima, ULVAC Inc. Japan

The quadrupole mass spectrometer (QMS) is usually used for the partial pressure measurement and the residual gas analysis of vacuum systems. However, the usual QMS, which quadrupole mass filter electrode length is about 100mm, is not available for partial pressure measurements over 10^{-2} Pa such as sputtering process etc., because of the sensitivity drop with pressure increase. This is caused by the loss of ions that arrive at an ion detector by ion-to-molecules scattering. The differential pumping system with a small orifice for gas introduction is usually used in such processes in order to maintain the pressure in the QMS less than its maximum operation pressure. But this system is relatively large and expensive. Another way to use the QMS in high pressure is down sizing of it. This presentation reports the development of the small QMS and its basic characteristics. The length and the field radius of the mass filter are 25mm and 1.8mm, respectively. The mass filter is operated with radio frequency of 7MHz. The filament and the grid of the ionizer is made of Y₂O₃ coated Ir wire and photo-etched Mo mesh, respectively. The ionizer includes an ion collector for the total pressure measurement. The ion detector for the partial pressure measurement is a Faraday cup type or a small secondary electron multiplier (SEM). The electron acceleration potential for ionization and electron emission current are 40V and 0.4mA, respectively. The sensitivity of this QMS for nitrogen was about 1×10^{-7} A/Pa with a Faraday cup type ion detector. The linear relation between ion current at mass-to-charge ratio of 28 and nitrogen pressure was obtained up to 0.1Pa. Proper correction of the sensitivity would make it possible to extend the operating pressure to 1Pa. The mass resolution, which is determined as peak width at 10% of the peak height, was less than 1 for all mass range from 1 to 50.

VT-TuP15 New Sublimation Reactor for Epitaxial Growth of II-VI Films, L.J. Rascon, L.C. Romo, S. Rogers, S. Quinones, J. McClure, University of Texas at El Paso; D. Zubia, University of Texas at El Paso, US

Close Spaced Sublimation (CSS) is a well established technique for deposition of polycrystalline CdTe thin films for solar cell applications. However, due to the very high growth rates at usual operating conditions, the technique has not been used for high quality films for advanced electronic applications. Our group has recently discovered operating conditions that yield smooth epitaxial CdTe thin films. This presentation will discuss a new CSS reactor designed at University of Texas El Paso which permits deposition of up to three different materials in layers as thin as a few tens of angstroms. Layers can be repeated as often as needed and with varying thicknesses if desired to build up a final device structure. The new reactor is able to deposit unary, binary and ternary semiconductor alloys and was conceived as a low cost alternative to high quality epitaxial crystal deposition techniques such as the costly molecular beam epitaxy (MBE). This poster presents the conceptual and physical design as well as the construction and operation of the new CSS reactor. The system performs depositions on a vertical arrangement in a vacuum of 5×10^{-1} Torr. Furthermore, it employs two servomotors. The first, to control the substrate to source distance, and the second, to position the substrate directly on top of one of the three sources. Hydrogen, helium and oxygen can flow during deposition to create different environments. A PID-loop controlled throttle valve manages the pressure to any set point regardless of the gas inflow. Moreover, the temperature of the three sources and the substrate are independently administered by three button heaters and a flat plate heater, respectively. Finally the complete deposition process is controlled by a computer program made in LabVIEW. Preliminary results of epitaxial CdTe films grown in the CSS reactor using very small temperature gradients will be presented.

VT-TuP16 A Study of New-Type System for Vacuum Gauges Calibration, Y.-W. Lin, C.-P. Lin, C.N. Hsiao, Instrument Technology Research Center, National Applied Research Laboratories, Taiwan

The purpose of this article is to analyse the stability of new-type system for vacuum gauges calibration. Design and operating procedures of the system mainly uses direct comparative method to calibrate vacuum gauges ranging from 10^{-3} to 10^{-6} Torr. The review of system is depended on capacitance gauge, ionization gauge and spinning rotor viscosity gauge, and estimated uncertainty is calculated with different pressure. Observational data show that chamber evacuates down to 10^{-8} Torr, the uncertainty of system varies from 0.6% at the high vacuum, to 1×10^{-5} Torr at midrange, and 1×10^{-2} Torr at the rough vacuum. From these pieces of result, the system can effectively use for calibration of vacuum gauges besides ultra-high vacuum.

VT-TuP17 Design of the EBIS Vacuum System, M. Mapes, Brookhaven National Lab, usa; E. Beebe, A. Pikin, J. Alessi, J. Ritter, L. Smart, Brookhaven National Lab

At Brookhaven National Laboratory the Electron Beam Ion Source (EBIS) is presently being designed. The EBIS will be a new heavy ion pre-injector for the Relativistic Heavy Ion Collider (RHIC). The new pre-injector has the potential for significant future intensity increases and can produce heavy ion beams of all species including uranium. The background pressure in the ionization region of the EBIS should be low enough that it does not produce a significant number of ions from background gas. The pressure in the regions of the electron gun and electron collector can be higher than in the ionization region provided there is efficient vacuum separation between the sections. For injection the ions must be accelerated to 100KV by pulsing the EBIS platform. All associated equipment including the vacuum equipment on the platform will be at a 100KV potential. The vacuum system design and the vacuum controls for the EBIS platform and transport system will be discussed as well as the interface with the Booster Ring which has a pressure 10-11 Torr. Work performed under the auspicious of U.S. Department of Energy.

VT-TuP18 Vacuum System of ISAC RFQ at TRIUMF, I. Sekachev, D. Yosifov, TRIUMF, Canadian National Laboratory

RFQ (Radio Frequency Quadrupole) accelerator of ISAC (Isotope Separator and Accelerator) at Triumf has been in full operation since 1999. The RFQ accelerator is a split ring 4-rod structure, 8m long with a bore radius of 7.5 mm operating at 35.36 MHz placed into diagonally split rectangular vacuum tank with dimensions of 1 x 1 x 8 m. The tank's main seal consists of two circumferential Viton O-rings with differential pumping of the space between them. There are six 1000 L/s turbo pumps operating continuously to keep the vacuum in the tank at about 5.0×10^{-7} Torr, measured by two hot filament ion gauges. The gauges are also used for interlocks. This paper describes the details of the vacuum system of RFQ and its operation.

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