Production and Use of High Centrifugal Fields

J. W. Beams

Department of Physics, University of Virginia, Charlottesville

HE action of a gravitational field and a centrifugal field on matter, for most purposes at least, may be considered to be essentially equivalent (1). Since the gravitational fields in regions near the earth are much smaller than the centrifugal fields that readily may be produced, it is common practice to express the magnitude of a centrifugal field in terms of the standard acceleration of gravity at the earth's surface. The purpose of this paper (2) is to describe a few typical experiments in which high centrifugal fields are used, together with some of the techniques employed.

In general, problems that require the use of high centrifugal fields fall into two principal categories. In the first type the centrifugal potential, which is proportional to $4\pi^2 N^2 r^2$, must be large; in the second type the centrifugal field, or $4\pi^2 N^2 r$, must be as intense as possible. N is the number of revolutions per second, and r is the radius of the rotor. As is shown later, with the present techniques the mechanical strength of the rotor is the factor that sets an upper limit to both the centrifugal potential and the intensity of the centrifugal field that can be produced with a given rotor. Both theory and experiment show that, for rotors of a given shape, the maximum stresses produced in the rotors are proportional to $4\pi^2 N^2 r^2$ or the square of the peripheral speed. Consequently, to produce a maximum centrifugal potential, the rotor must be carefully designed to reduce stress concentrations to a minimum.

The choice of rotor material depends primarily upon the load that the rotor must carry. If there is no load or if it is very light, the rotor material should have as large strength-to-density ratio as possible. If the load is not light, then the design of the rotor and selection of rotor material are more complicated. In general, tough materials are preferable to brittle ones if the strengths and densities are equivalent. Since both the centrifugal potential and the "bursting strength" depend directly upon the square of the peripheral speed of the rotor, the maximum centrifugal potential is independent of the radius of the rotor. On the other hand, since the centrifugal field is given by $4\pi^2 N^2 r$, to obtain a maximum field intensity, the radius of the rotor should be made as small as possible.

Although the maximum rotor speed is at present limited only by the rotor strength, in most experiments several other problems must be solved. In the first place, as a rotor is accelerated, it may pass through critical vibration frequencies which, unless they are damped, will destroy the rotor and the bearings. If the rotor is mounted in rigid bearings it must, of course, be carefully balanced. In many practical cases

where the balancing cannot be done with enough precision the rotors are mounted on flexible shafts in a manner similar to the De Laval turbine (3) or in air bearings so that they can seek their own axis of rotation. In ordinary journal or ball bearings the frictional drag increases very rapidly with the speed of the bearing surface (4), so the radius of the bearing for high-speed rotors must be made very small, or the friction will become excessive. This frictional drag not only consumes a large amount of energy but introduces gyroscopic, heating, and other effects that are usually undesirable. Another difficulty encountered is the excessive heating of the rotor by the air friction on its surface. This is especially troublesome in sedimentation experiments where the rotor temperature must be held constant.

For rotors several centimeters in diameter the aforementioned problems have been solved successfully in several ways. Svedberg and his collaborators (5)solved the problems in their ultracentrifuge with an oil turbine drive and special oil-lubricated and -cooled bearings. The carefully balanced rotor is spun in hydrogen at reduced pressure. The hydrogen not only reduces the gaseous friction on the rotor but, because of its high thermal conductivity, conducts the heat that is generated to the walls of the chamber. Pickels and I (6-8) solved the foregoing problems in the vacuumtype ultracentrifuge by spinning the rotor in a vacuum. The rotor is mounted on the end of a long, very small diameter, flexible shaft that passes through a vacuum gland in the top of the chamber and is connected either to a small-diameter, air-supported, airdriven turbine or to an electric motor drive situated above the vacuum chamber. In this apparatus no appreciable heat is generated in the rotor, so not only does it remain at constant temperature when the chamber walls are properly thermostated, but the power required to drive it is very small. This vacuum-type ultracentrifuge has been improved by many different workers and for some time has been commercially available. Space does not permit the mention here of other methods that have been devised for spinning high-speed rotors, and reference must be made to more detailed reviews (5, 8, 9).

For several years a method of magnetically suspending and electromagnetically driving a rotor in a vacuum has been under development at the University of Virginia (10) which automatically solves the aforementioned difficulties (11-15). Also, rotors with diameters from 10^{-2} to more than 25 cm, and with weights from 5×10^{-5} to 2.5×10^4 g have been used. The general method is illustrated by Fig. 1, which is a schematic diagram of an apparatus used for measuring the tensile strengths of metals (16) as well as the tensile strengths of metal films as a function of their thickness (17, 18). The steel rotor is freely suspended inside a glass vacuum chamber by the axially symmetric magnetic field of the solenoid situated above the vacuum chamber. Figure 2 is a photograph of a $1\frac{1}{2}$ in. steel sphere freely suspended with the vacuum chamber and drive coils removed. The vertical force on such a rotor is roughly equal to M(dH/dx), where M is the magnetic moment of the suspended rotor and dH/dxis the vertical gradient of the magnetic field. In order to maintain the rotor suspended freely at a definite height, means must be provided for regulating the current in the solenoid. This may be accomplished in



Fig. 1. Schematic diagram of magnetic suspension.

several ways (14, 15). In Fig. 1 a small pickup coil mounted on a plastic support below the glass vacuum chamber serves as the sensing element of a servocircuit that regulates the current through the solenoid in such a way that the rotor is held at the desired vertical position. The rotor automatically seeks the strongest part of the field, which is on the axis of the solenoid. However, if it is disturbed, it will oscillate around the axis. To prevent this the core of the salenoid is supported by a fine wire and thus hangs as a pendulum in a dash-pot of oil. If the rotor is disturbed, the core follows the horizontal motion of the rotor and damps it out. When the apparatus is properly adjusted, no movement of the rotor can be observed with a 50-power microscope focused on the rotor surface. For example, for use with rotors from 0.1 to 0.2 in. in diameter, a cylindrical steel rod core 3.5 in. long, 0.2 in. in diameter, suspended by a 0.25-in. length of 0.017-in. piano wire is satisfactory. The core is immersed to a depth of 1.2 in. in S.A.E. No. 10 motor oil contained in a flat-bottom glass tube.



Fig. 2. Magnetically supported 11/2-in. steel sphere.

The support solenoid L_1 (Fig. 3) consists of 20,500 turns of A.W.G. No. 30 enameled copper magnet wire wound on a 1.1-in. o.d. plastic tube. The outside diameter of the solenoid is 3.12 in. and its length is 2.63 in. Its direct-current resistance is roughly 1200 ohms, and its inductance is about 30 hy, which gives it a time constant of about 0.025 sec. The "pickup" coil L_2 consists of 12 turns of A.W.G. No. 34 copper wire bunchwound with an inside diameter of 0.4 in. The support circuit is shown in Fig. 3. The pickup coil, L_2 , is in the grid circuit of a partially neutralized, tuned-grid, tuned-plate oscillator that operates at a frequency between 5 and 8 Mcy/sec. A change in the vertical position of the rotor changes the Q value of the oscillator circuit and, therefore, the amplitude of the oscillations. The output of the oscillator is applied to the grid of a 6J5 tube in an "infinite impedance" detector stage (cathode follower), which does not load the oscillator. The d-c voltage appearing across the cathode resistor is an electric measure of the rotor height above the pickup coil. A portion of this voltage is picked off the cathode resistor and constitutes the control or "error" signal. In parallel with the cathode resistor is a resistance-capacitance differentiating circuit. The potential difference across the resistance of this network is proportional to the time rate of change of rotor height. The "error" and the



derivative or damping signals are separatively amplified, mixed, and applied to the grid of a second cathode follower stage. The combined signal is next applied to the grids of the 6L6 output stage and, hence, regulates the current through the support solenoid, L_1 . It will be observed that separate means are provided for independently adjusting both the error and the derivative or damping signals, so that it is a simple matter to find the proper relative and absolute values to provide stable support for the rotor.

The rotor is spun by a rotating magnetic field in a manner similar to that of the armature of an induction motor or of a synchronous motor. If the rotor is not too large and a small amount of temperature rise of the rotor (order of 5° C) can be tolerated, the induction motor drive is preferred because of its simplicity; but, if the rotor temperature and rotor operating speed must remain constant, then it should be both accelerated and operated as a synchronous motor. When accelerated as a synchronous motor, the frequency of the rotating magnetic field is automatically increased at the same rate as the acceleration of the rotor. When at operating speed, the frequency of the rotating magnetic field is maintained constant by a crystal-controlled circuit. The rotating magnetic field is produced by alternating current in two pairs of drive coils outside the vacuum chamber, as shown in Fig. 4. Standard drive circuits are used to produce the alternating current in these coils (14, 19).



Fig. 4. Schematic diagram of drive circuit and method of measuring rotor speed.

Figure 4 shows a schematic diagram of a drive system and a method of measuring the rotor speed. If the rotor is to be accelerated in a way similar to that of the armature of an induction motor, the output of a fixed-frequency oscillator is fed to a 90-deg phase splitting bridge. Each phase is separately amplified and applied to one pair of drive coils, as shown. The drive coils are in parallel with capacitors of the proper magnitude to give resonance at the desired drive frequency. Because of the high electric resistance of the rotor, the torque is comparatively large, even when the rotor is at rest or turning slowly. During the acceleration period the rotor is heated by the eddy currents induced by the rotating magnetic field; but, with the acceleration rates used in practice, the temperature rise is usually only a few degrees. The rotor temperature may be measured by its thermal radiation.

Since the supporting magnetic field is axially symmetric, the rotor friction caused by the magnetic suspension itself should be exceedingly small. This has been found to be the case (14, 20). The observed rotor friction can be entirely accounted for by the gaseous friction on the spinning rotor. By maintaining the gas pressure surrounding the rotor below about 10^{-6} mm-Hg, this gaseous friction is very small; so that, in order to stop the rotor in a reasonable time, the direction of rotation of the magnetic field must be reversed and the rotor driven to rest. For a spherical rotor of radius r, density d, and absolute temperature T, surrounded by a gas at low pressure of molecular weight M and pressure p, it can be shown that approximately

$$\log_{\theta} \frac{N}{N_0} = \frac{-5p}{rd} \left(\frac{M}{2\pi RT}\right)^{1/2} (t-t_0),$$

where N_0 is the number of revolutions per second at time t_0 , and N is the number of revolutions per second at time t. As a matter of fact, by measuring the deceleration of the rotor while it is coasting freely, the apparatus serves as an excellent absolute low-gaspressure gage. With the gas pressure below 10^{-6} mm-Hg the friction is so small that the rotor will accelerate as an induction motor to the speed corresponding to the frequency of the oscillator and then "lock in" and spin as a synchronous motor. In practice, when it is desired to spin the rotor at extremely constant speed and a small temperature rise can be tolerated during the acceleration period, as in the case of a rotation mirror, the rotor is accelerated as an induction motor to just above the running speed. The oscillator is then connected to a crystal control which holds it constant to the order of 1 part in 10⁸, and the rotor speed settles down and "locks in." In a few minutes all observable "hunting" vanishes, and the rotor speed is at least as constant as the frequency of the oscillator (19). The rotor speed is determined by reflecting or scattering light off the rotor into a photomultiplier tube, as shown in Fig. 4. A mark placed on the rotor causes the photomultiplier output to be periodic with a repetition rate equal to the speed of the rotor. This signal is amplified and compared with that from a calibrated variable frequency oscillator on a cathoderay oscilloscope screen. For the most precise determination of rotor speed, the output of the photomultiplier is compared with the broadcast signal of station WWV of the National Bureau of Standards.

By spinning a rotor until it explodes, the maximum strength of the rotor material can be computed if the rotor is properly designed. For spherical rotors the maximum stresses are at the center and, therefore, in a region not containing a surface. Table 1 gives the results obtained with a series of carefully selected steel ball bearings just before they exploded (16). They are the maximum values obtained. It will be observed that all the rotors exploded at approximately the same peripheral speed, which is in accord with theory, and also that the highest centrifugal acceleration, which

Table 1. Bursting speeds of spherical steel rotors.

| Rotor diam. (mm) | Rotor speed (10 ³ rev/min) | ${ m Peri-} \ { m pheral} \ { m speed} \ (10^{3} \ { m cm/sec})$ | Centrifugal acceleration $(10^{e}g)$ | Maxi- mum ealcu- lated stress (10 ³ lb/in. ²) |
|------------------------|--|--|--------------------------------------|--|
| 3.97 | 4,420 | 96 | 47.1 | 410 |
| 2.38 | 7,410 | 92.5 | 72 | 385 |
| 1.59 | 12,660 | 105 | 143 | 498 |
| 0.795 | 23,160 | 96.5 | 240 | 420 |
| 0.521 | 37,980 | 104 | 428 | 488 |
| 0.398 | 48,000 | 100 | 515 | 454 |

was just greater than a half-billion times gravity, was obtained with the smallest rotor. The calculated maximum stresses that occur at the center were calculated by elastic theory (21) and were not corrected for plastic flow.

If a thin film of material is uniformly deposited on the complete cylindrical surface of a rotor such as that shown in Fig. 1, it can be shown (17) that $4\pi^2 N^2 r^2 d = T + Ar/h$, where N is the rotor speed in revolutions per second, r is the rotor radius, d is the density of the deposited film, h is the film thickness, Tis the tensile strength of the deposited film, and A is the adhesion (adhesive force per unit area). Since the second term on the right contains r/h, while the first does not, by using rotors of different radii both the tensile strength and adhesion can be obtained. Also, by depositing the films on the rotor surface in patches, the "hoop strength" vanishes, and the adhesion is obtained directly. However, in order to obtain the tensile strength of the films, small rotors were used in order to make r/h as small as possible; and a process of thermal cycling was applied to the rotor containing the deposited film in such a way that the adhesion was reduced to a negligible value. A series of measurements made on electrodeposited silver films as a function of their thicknesses showed that for film thicknesses greater than about 3×10^{-5} in. the tensile strengths were approximately the same as found in bulk silver and independent of the thickness. However, between about 3×10^{-5} and 10^{-5} in. the tensile strength of the films increases manyfold (17, 18). This result is probably to be expected from the dislocation theory of metals. The experiments on the adhesion of silver films to metals are less clear, but there is an indication that the adhesion begins to increase at thickness of roughly half that where the tensile strength increases.

If the rotor of Fig. 1 is made of high-strength ferromagnetic material with properly ground flat mirror surfaces, it becomes an excellent high constant-speed rotating mirror. A rotor with six flat mirror surfaces each 0.25 in. wide has been operated at 20,000 rev/sec for long periods of time (19). The rotor speed was controlled by a thermostated crystal, and no variation in speed could be measured. The speed was determined to about 1 part in 10^7 . The accuracy was limited by the estimated reliability of the signal received from WWV. This rotating mirror is almost ideal for studying phenomena that radiate light in short intervals of time. Also, it should be well suited for velocityof-light measurements as soon as suitable light paths can be measured with accuracy comparable to that of the rotor speed.

The fact that the tensile strength of silver films increases rapidly when the thickness is decreased below 3×10^{-5} in. at room temperature naturally raised the question of whether or not this effect will occur at very low temperatures and, if so, at greater or smaller thicknesses. Figure 5 is a schematic diagram of an apparatus at present being used to study the tensile strength of films and of metal crystals at liquid nitrogen and liquid helium temperatures. The rotor is freely suspended in a glass vacuum chamber surrounded by two concentric Dewar flasks. The inner flask contains liquid helium and the outer liquid nitrogen. The support solenoid surrounds the outer flask. The horizontal damping of the rotor is provided by a small, steel needle attached to a small cylinder of plastic foam. The support circuit is essentially the same as that previously described. With some modification of the method, the rotors also may be suspended and spun up to temperatures where the rotor material loses its ferromagnetic properties.

Perhaps the most important use of high-speed rotors is in centrifuges for separating material and for the determination of molecular weights. Of these two centrifuge problems, the measurement of molecular weights is perhaps the more exacting on the appara-



Fig. 5. Method of magnetically supporting rotors at low temperatures.



Fig. 6. Magnetically supported ultracentrifuge.

tus. Therefore, a brief description of the magnetically supported ultracentrifuge (22) is given. There are two principal methods of determining the molecular weight of a substance by centifuging (5). In the first, or rate-of-sedimentation, method the rate of settling of the substance in the centrifugal field is measured. In the second, or equilibrium, method the centrifuging is continued until the sedimentation is balanced by back diffusion, and the concentration of the substance is measured at various radial distances. The first method requires a relatively high centrifugal field and a centrifuging time of a few hours, whereas the second employs a relatively less intense centrifugal field but a centrifuging time of several days or weeks. In the past the first, or rate-of-sedimentation, method has been used more widely where the molecular weights are large enough to give an appreciable rate of sedimentation because of the difficulty of maintaining the speed and temperature of the centrifuge rotor extremely constant for long periods of time. On the other hand, the theory used in the second, or equilibrium, method is much more reliable because it is based directly upon thermodynamics rather than upon Stokes' law, as in the case of the rate-of-sedimentation method. Furthermore, with the same rotor speed the equilibrium method may be used for much smaller molecular weights.

Since magnetically suspended rotors in a high vacuum have a very small frictional resistance and are freely suspended, their temperature may be accurately maintained constant by thermostating the vacuum chamber walls. Also the rotor speed can be accurately controlled. Consequently the magnetically suspended rotor should make an excellent ultracentrifuge.

Figure 6 is a diagram of the ultracentrifuge and Fig. 7 shows the ultracentrifuge rotor with the vacuum chamber removed. The rotor R is freely suspended inside the brass vacuum chamber V by the solenoid Fsituated above the chamber. It is accelerated to operating speed by the air turbine T below the chamber. The turbine is connected to the rotor by the small diameter shaft S that passes through the vacuum-tight oil glands G_1 and G_2 . The shaft S fits into a slot in the rotor; and, when the rotor reaches the desired operating speed, it is pulled out. This disconnects the rotor from the drive and allows it to coast freely during the period of observation of the sedimentation. The sedimentation is observed optically by passing a beam of light through window W_1 , the centrifuge cell K_1 , the right-angle prism P, and out through window W_2 .

The rotor R shown in Figs. 6 and 7 is made of Duralumin with a steel core and weighs about 10 lb. More recent rotors are about the same shape and made of alloy steel. They weigh about 30 lb, and the distance from the axis of rotation to the center of the centrifuge cell K_1 is 6.5 cm. The support solenoid Fconsists of 28,000 turns of No. 22 enameled copper magnet wire and has a resistance of about 1200 ohms. The operating current in the solenoid is between 150 and 350 ma. The cylindrical steel core C of the solenoid $(1\frac{15}{16}$ in. in diameter and 12 in. long) is suspended by a small wire B, as shown in Fig. 6. The lower end of C hangs in a dash-pot of No. 40 motor oil which damps any swinging motion. This damps any horizontal motion of the rotor R in a way similar to that in



Fig. 7. Magnetically supported ultracentrifuge rotor with vacuum chamber removed,

the apparatus of Fig. 1. Incidentally the core C can, of course, be permanently magnetized if desired. Sometimes this has been found to be advantageous. The support circuit is similar to that shown in Fig. 2, and reference should be made to previous papers for details (22, 23).

With the air pressure the order of 10⁻⁶ mm-Hg and the 30-lb rotor coasting freely at 300 rev/sec, it required approximately 3 days to decelerate 1 rev/sec. This very slow rotor deceleration allows the rotor speed to be measured by comparing it with the broadcast signal from station WWV to 1 part in 1 million. The temperature of the walls of the chamber and. hence, that of the rotor is held constant to better than 1 part in 10,000 for as long as desired. With this equilibrium method the molecular weight M_e is given by the relationship (5).

$$M_e = \frac{2RT \log_e (C_1/C_2)}{(1 - dV) 4\pi^2 N^2 (r_1^2 - r_2^2)}$$

where C_1 and C_2 are the concentrations at the radii r_1 and r_2 , respectively, T is the absolute temperature, V is the partial specific volume, d is the density, and Nis the number of revolutions per second. This formula is for a dilute ideal incompressible solution. With Nmeasured to 1 part in 10^6 and T to 1 part in 10^4 , the values of C_1 and C_2 are the least precisely known values in the relationship. The partial specific volume V and density d are measured in separate experiments, so they need not be considered here. In view of this, a great deal of effort has been devoted to improving the precision with which the concentration in the centrifuge cell can be measured. First, it was necessary to develop a centrifuge cell (24) in which the optical distortions were balanced out; and, second, an interferometer method was developed for determining the refractive indexes of the solutions while the rotor was spinning (25). From these refractive index measurements the values of C_1 and C_2 may be much more precisely determined, but they are still the least accurately known of the quantities measured while the rotor is spinning.

In the true equilibrium experiments the rotor speed is held constant during the process of centrifuging. It will be noted that, although "hunting" of the rotor is entirely absent in the afore-mentioned centrifuge, the rotor speed is very slowly decreasing (0.3 rev/sec day). Archibald (26) has shown that, if

$$\left. \frac{1}{N} \cdot \frac{dN}{dt} \right/ 4\pi^2 N^2 S << 1,$$

where S is the sedimentation constant, then the equilibrium condition will remain once it is established in the centrifuge cell. These conditions are satisfied in practice by the foregoing equilibrium centrifuge. As a matter of fact, the equilibrium condition is established in the centrifuge cell in much less time with the rotor speed very slowly decreasing than if the speed were held constant, which is a considerable advantage. In practice this centrifuge may be used for molecular weight measurements over the range from 10² to 10⁷ molecular weight units with excellent precision. For

example, in the case of sucrose, which is in the most difficult range, the molecular weight values obtained are accurate to three significant figures.

The air-turbine drive shown in Figs. 6 and 7 was adopted because the rotor could be rapidly accelerated to operating speed without heating. However, there are many experiments in which it would be better to have an electric drive for the centrifuge with which the operating speed could be made extremely constant. The problem is difficult, because the temperature of the rotor should not change as a result of the drive during the acceleration period. Such a drive has been under development at the University of Virginia for some time (20). In one method (27) small permanent magnets are mounted in the lower part of the rotor at about one-third of the rotor radius with their axes parallel to the axis of rotation and are driven by a rotating magnetic field below the rotor with a frequency equal to the rotor speed but slowly increasing. The rotor thus accelerates as a synchronous motor and is not heated. When the rotor reaches the operating speed, the frequency of rotation of the magnetic field is held constant. It is believed that the ultracentrifuges here described will be useful, not only for the measurement of molecular weights, but also for the measurement of molecular weight distributions. and that they may be used not only as equilibrium centrifuges but also for rate-of-sedimentation measurements.

Space does not permit the further review of many more uses of the magnetically supported, high-speed rotors; but it is believed that their use should be seriously considered whenever high or constant rotor speed is required or the bearing friction must be low. Finally, for some cases where the control circuits are not feasible, the diamagnetic properties of superconductors may be used for supporting spinning magnets. When magnetic fields cannot be used electrostatic suspensions are probably feasible.

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Scintillation Spectrometer with Improved Response

P. R. Bell

Oak Ridge National Laboratory, Oak Ridge, Tennessee

HE physical electronics group at the Oak Ridge National Laboratory has constructed an improved scintillation spectrometer that is one step closer to the ideal gamma-ray spectrometer, namely, one that would give one single peak in its response for a monoenergetic gamma ray and would have nearly 100-percent efficiency.

The usual scintillation counter produces a pulse distribution for a monoenergetic gamma ray that includes, in addition to a peak representing the full energy of the gamma ray, a continuous distribution of pulses from zero size to a well-defined upper limit lower than the principal peak. This continuous distribution is caused by gamma-ray quanta that interact with the phosphor by the Compton process wherein an electron and a scattered gamma-ray photon share the energy of the original quantum. The scattered photon may escape from the phosphor; if it does, only the smaller pulse caused by the electron is recorded. A similar escape of energy often follows a pair-production process, wherein the created electron pair shares the energy left over in the original quantum above the amount (1.02 Mev) required to produce the electron and positron of the pair. After both members of the pair are stopped in the phosphor, the positron combines with some nearby electron to produce by annihilation two photons of 0.511-Mev gamma ray. One or both of these photons might escape from the phosphor without absorption.

The response of an ordinary scintillation counter to gamma rays is shown in Figs. 1a and 2a. The phosphor in the ordinary counter was a right circular cylinder of sodium iodide activated with thallium, $1\frac{1}{2}$ in. in diameter and 1 in. high. Figure 1a shows the response to the radiations from Zn65 which gives a gamma ray of 1.114 Mev and a very weak positron emission (resulting in a small amount of annihilation radiation). The peak at 640 pulse-height divisions is produced by complete absorption of the 1.114-Mev gamma ray. The peak at 300 pulse-height divisions is produced by the 0.511-Mev annihilation photons from





Fig. 1. Response of large and small crystals to the gamma rays of Znº5.