Study of Adhesion by the High-Speed Rotor Technique†

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ABSTRACT

If films of a material are uniformly deposited on the peripheries of cylindrical rotors having different radii and the rotor speeds are increased until the material is thrown off, both the tensile strength and the adhesion to the rotor surface of the material may be determined. If the material is deposited on a rotor in circumferencially disconnected patches, the absolute value of the adhesion can be obtained directly. The techniques of spinning magnetically suspended rotors in a vacuum is described and its application to the measurement of the adhesion of electrodeposited and evaporated metals on rotor surfaces is discussed.

The absolute measurement of the adhesion of two substances is often complicated by the presence of unavoidable stress concentrations introduced by the method of measurement. These stress concentrations may give rise to tearing or they may introduce shearing and other forces of a nature that make the data difficult to interpret. One way of reducing these difficulties to a minimum is to apply the forces at the interface between the substances by centrifugal methods. In this way, the stresses are produced by body forces and consequently not only can be made uniform but also may be determined with precision. The principle of the centrifugal method is very old; however, the method has not been used extensively in the past because of the relatively high centrifugal fields required for most measurements of practical interest. Suppose that a layer of a substance of thickness h is uniformly deposited on the periphery of a solid cylinder of radius R. Then:

$$4\pi^2N^2R^2d\left(1 + \frac{h}{R} + \frac{h^2}{3R^2}\right) = T^1 + A\frac{R}{h} \qquad (1)$$

or when h is small in comparison to R

$$4\pi^2N^2R^2d = T + A\frac{R}{h} \qquad (2)$$

where N is the number of revolutions per sec of the cylinder, d the density of the coating, T^1 an average tension and A the adhesion. (Adhesive force per unit area). The maximum value of the tension is roughly $\frac{(R + h)^2}{R^2}T$ where T is the tension near the interface between the coating and the rotor. It will be noted that the centrifugal force on the coating or film is balanced by the hoop stress and by the adhesion of the film to the rotor. Also, it will be observed that if the adhesion can be made negligibly small, the tensile strength of the coating material T can be determined. Use has been made of this method[1, 2, 3] for studying the tensile strengths of thin electrodeposited and thin evaporated films on steel rotors as a function of film thickness h, and it has been found that for the very thin films, the tensile strength increases many fold. Also, the adhesion A can be measured[1, 2, 3] directly if the material is deposited on the rotor in circumferentially disconnected patches which eliminates the hoop stresses and makes T^1 vanish in equation 1. This gives for the adhesion $A = 4\pi^2N^2R\left(h + \frac{h^2}{R} + \frac{h^3}{3R^2}\right)$. At first sight it would seem that the patches might loosen around the edges and hence would rip off and complicate the measurements. However, in practice, the deposit near the edges is usually made slightly thinner by the masking process, and observations show that the edges are the last part to fly off. In many cases, $A\frac{R}{h}$ turns out to be considerably larger than T and it is not necessary to use patches, but it usually is desirable to make the adhesion measurements on the patches first. For example, if elastic theory is assumed and if $A\frac{R}{h}$ is somewhat larger than the tensile strength T then, from the work of Morton[4] and Turner,[5] it may be shown that the adhesive bond may be broken when:

$$4\pi^2N^2R^2d_2\left(1 + \frac{h}{R} + \frac{h^2}{3R}\right)\left[1 - \frac{d_1}{d_2}\,\frac{1\text{-}p}{4}\,\frac{E_2}{E_1}\right] = \frac{R}{h}A \qquad (3)$$

where d_1 and d_2 are the densities of the steel rotor and of the coating respectively, p, is Poissions ratio for the steel, E_1 and E_2 are the Youngs modulii for the steel, and the coating, respectively. For thin silver films, this reduces to $\frac{R}{h}A \sim (0.95)\, 4\pi^2N^2R^2d_2$. Both experiment and theory show that the bursting strength of rotors of a given shape made of the same material is directly proportional to the square of the peripheral speed i.e. $4\pi^2N^2R^2$. Consequently, from equation 1 it will be observed that for measuring large values of A, the rotor radius should be as small as possible. Clearly h also should be as large as possible.

Interest in the adhesion problem at the University of Virginia began in 1930 during a series of experiments with high-speed, air-driven air-supported rotors.[6] In these experiments, part of the periphery of the rotor was painted white in order to measure the rotor speed by strobiscopic light. It was found that the paint flew off when the centrifugal field became the order of a million times that of gravity. This, of course, suggested that the method might be of value in determining adhesion. However, the rotor spun in air and the abrasive effect of the air and the shock waves produced in the air somewhat complicated the results. Later, rotors were designed with inner cylindrical cores on which the samples were attached, surrounded by air tight shields that prevented the abrasion. However, this weakened the rotor and thus restricted the measurements to comparatively thick coatings of the adhesive material. The development of the vacuum type ultracentrifuge[6, 7] in 1935 provided a simple means of spinning rotors in a high vacuum at speeds which were limited only by the strength of the rotor material. This instrument was used to make a few qualitative measurements of the adhesion to the rotor of some dried paints and upon some low vapor pressure oil films. However,

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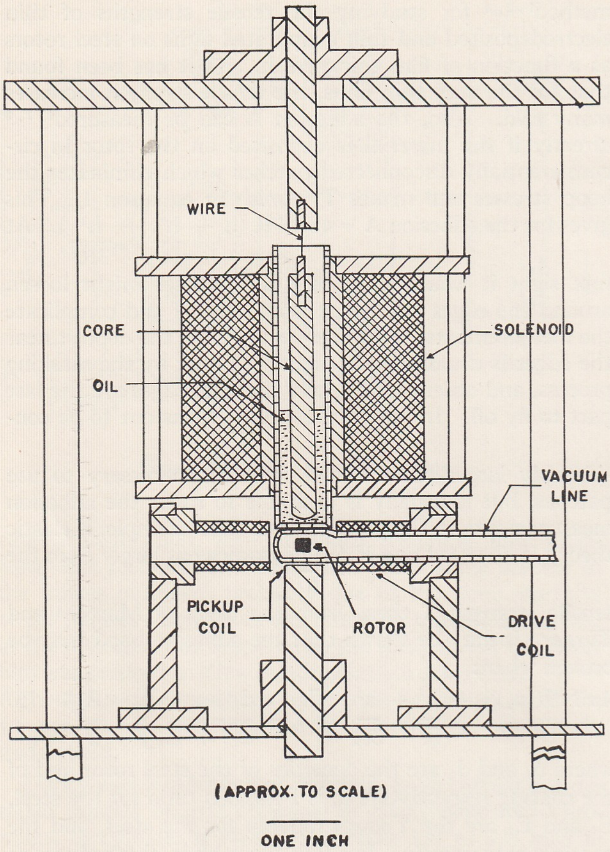

Figure 1—Schematic diagram of Magnetic Suspension

again the apparatus proved not to be ideal for the purpose because the method was better suited to spinning large rotors rather than small ones, so the centrifugal fields available were limited. The data were never published. The method of spinning magnetically suspended rotors in a high vacuum developed in the laboratory[8, 9] at Virginia beginning in 1937, fortunately is free of the restrictions in the methods mentioned above. In the first place, the magnetically suspended centrifuge rotor spins in a high vacuum which eliminates all gaseous erosion of the sample and in the second place, small rotors may be spun which produce extremely high centrifugal fields. Rotors from 0.008 cm to 30 cm in diameter have been spun. The rotor speeds attained are limited only by the strength of the rotor. For example, a 0.022 cm rotor was spun to over 10^6 rps[10] which produced centrifugal fields of the order of 10^9 times that of gravity.

The method is shown schematically in Figure 1. The rotor which is made of ferromagnetic material, such as steel or nickel, is freely suspended (without touching anything) inside of a glass vacuum chamber by the axial magnetic field of the solenoid. The solenoid is situated above the vacuum chamber and produces a magnetic field which is symmetrical with respect to a vertical axis and is diverging downward. The vertical force on the rotor is approximately equal to $M\frac{dH}{dz}$ where M is the magnetic moment of the suspended rotor and $\frac{dH}{dz}$ is the vertical gradient of the magnetic field. The rotor is maintained at a definite vertical position by an automatic regulation of the electric current in the solenoid while its horizontal position, which is on the axis, is determined by the shape of the axial magnetic field. Several different methods have been used for automatically regulating the current in the solenoid.[9, 11] Figure 2 shows a circuit which is easily adjustable and stable. A small pick-up coil L_1 mounted below the vacuum chamber serves as a sensing element of the servo circuit which regulates the current through the solenoid in such a manner as to maintain the rotor at the desired height. The rotor automatically seeks the strongest part of the magnetic field which is on the axis. On the other hand, if it is disturbed, it will oscillate around the axis so that means should be provided for damping this type of motion. In Figure 1, the steel core of the solenoid is hung by a fine wire as a pendulum in a dash pot of oil. If the rotor moves, the core will follow the horizontal motion and damp it out. For rotors, less than a millimeter in diameter, the pick-up coil is sometimes placed above the rotor either inside or on top of the vacuum chamber, and a small damping needle is mounted in a tube of liquid just below the vacuum chamber. When properly adjusted, no oscillations or movements of the rotor, either horizontal or vertical are observed with a microscope focused on fine scratches on the rotor.

The support solenoid consists of approximately 20,000 turns of No. 28 enameled copper wire wound on a 1.1 cm o.d. plastic tube. Its direct current resistance is of the order of 1000 ohms and its inductance about 30 henries. The pick up coil consists of from 5 to 12 turns of No. 34 copper wire bunch wound with an inside diameter of from 1.2 cm to 0.5 cm (these sizes depend upon the diameter of the rotor). The pick-up coil is in the grid circuit of a partially neutralized tuned-grid, tuned-plate oscillator which operates at from 5 to 8 megacycles/sec. A variation in the position of the rotor, with respect to the pick-up coil, changes the Q of the oscillator circuit and hence the amplitude of the oscillations. The output of the oscillator is applied to the grid of a 6J5 which serves as an infinite impedance detector stage. The d-c signal appearing across the cathode resistor is proportional to the amplitude of the oscillations and, therefore, is an electrical measure of the rotor height (Figure 1) above the pick-up coil. This d-c signal is amplified and used to regulate the grid voltage, and thus the plate current of the power amplifier, which is the current through the support solenoid. In order to prevent vertical oscillations of the rotor, a phase lead, together with effective damping or "antihunt," is introduced into the amplifier stage.

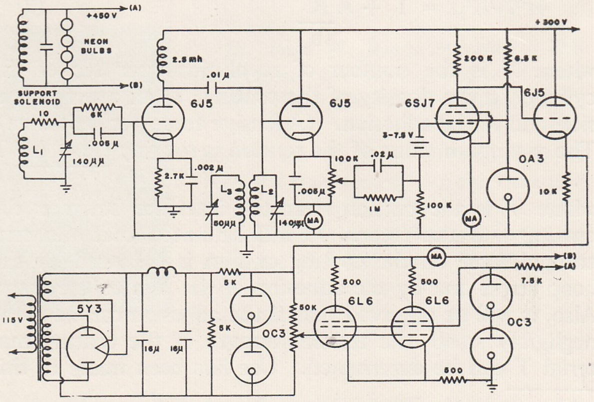

Figure 2—Magnetic support circuit

The rotors used were solid cylinders of a high strength ferromagnetic material. The ratios of their lengths to radii were always slightly less than $\sqrt{3}$ in order to insure stability. Most of them used so far, were made of 0.95/1.10 carbon tool steel which was heated to 1450° F and quenched in water. They were tempered by heating to 425° F and again quenched in water. They were then polished with jeweler's rouge. Next they were carefully cleaned in acetone, then in warm water containing a commercial detergent followed by a thorough rinsing in distilled water. After this, they were ready for the process of electrodeposition.

The rotors are spun inside of the vacuum chamber by a rotating magnetic field in a manner similar to that of the armature of an induction or of a synchronous motor. If the rotors are less than a centimeter in diameter and if a small temperature rise during the acceleration period can be tolerated, the induction motor type of drive is usually preferred because of its simplicity; however, if the final rotor speed or the final operating temperature must be extremely constant, the synchronous drive should be used. In both methods the rotating magnetic field is produced by alternating current in two pairs of drive coils mounted outside the vacuum chamber as shown in Figure 3. Any standard drive circuit may be used for the induction motor drive. Figure 4 shows a circuit which has been used for most of the experiments on the adhesion and tensile strength of metals. It will be noted that the output of a fixed frequency oscillator is applied to a 90° phase splitting bridge. Each phase is separately amplified and fed to a pair of the drive coils. The drive coils are connected in parallel with the proper capacitors to produce resonance at the oscillator frequency. Because of eddy currents induced in the rotor, a torque is produced which accelerates it to the desired rotor speed. These eddy currents heat the rotor; but, if the acceleration rate is not too rapid, the temperature rise is not excessive for metal adhesion experiments. If desired, the rotor temperature may be measured by its thermal radiation or by the change in its permeability. The first method is more precise; but, if only very rough measurements are required, the latter method of determining the temperature is comparatively simple if the permeability of the rotor material varies rapidly with temperature. It can be shown that the force which supports the magnetically suspended rotor is $M \frac{dH}{dz}$, which in turn is proportional to I^2 where I is the current in the solenoid. If M now varies with change in permeability, the temperature may be determined by measuring the current in the support solenoid necessary to maintain the rotor at a given vertical position. Obviously, calibration curves of I vs T must first be made with each rotor. Friction on the rotor due to the magnetic suspension is negligibly small, but the gaseous friction is large except when the pressure is low. By maintaining the gas pressure in the vacuum chamber surrounding the rotor at a pressure below 10^{-6} mm of Hg the gaseous friction on the rotor also becomes extremely small and no observable heating of the rotor results from this. As a result, 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. The speed of the rotor is measured by reflecting light off of the rotor into a photomultiplier tube. The output of the photomultiplier is periodic with a repetition rate equal to the speed of the rotor. This signal is amplified and compared with that from a variable-frequency oscillator by means of a cathode ray oscilloscope. The rotor speed is determined from the resulting Lissajous pattern on the oscilloscope screen. When metal flies off the rotor i.e. when the centrifugal force breaks the adhesion to the metal, the rotor is slightly disturbed, and produces changes in the Lissajous patterns which are easily observed. Also, the rotor disturbance varies the Q of the pick-up circuit which in turn shifts the frequency of the support circuit. This frequency is radiated and may be monitored by a radio frequency receiver in its vicinity. With the receiver beat-frequency oscillator in operation, the receiver is tuned to give a low audio frequency note in the loud speaker. This note changes pitch when the material is thrown off the rotor. Consequently, this serves as a simple device for determining when the adhesion is broken.

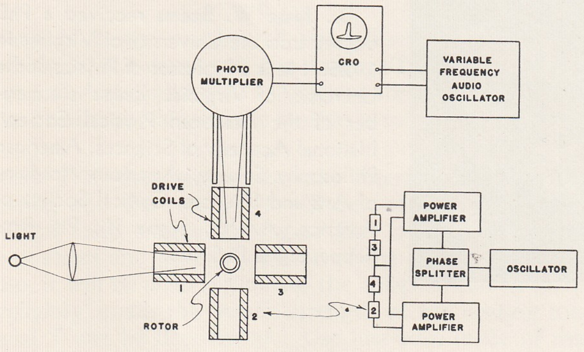

Figure 3—Schematic diagram of rotor drive and of speed measuring device

Since the primary purpose of this paper is to describe the high-speed rotor technique for measuring adhesion, no attempt will be made to review the results obtained by various workers with the method. However, it might be of interest briefly, to indicate some of the results obtained in the laboratory which illustrate the use of the technique.

In connection with the measurement of the tensile strengths of thin films of metals, as a function of thickness, by the high-speed rotor technique, it was essential to reduce the adhesion of the electrodeposited film on the rotor to a negligible value.[3] In order to do this, it was necessary to determine the adhesion for each electrodeposition and film loosening process as a function of film thickness. In this way, it was found for example, that in the case of the deposition of silver on a steel rotor, standard methods could be used provided certain very thin coatings were put on the rotor before electrodeposition and the films were thermally cycled afterwards. The rotors were first cleaned as previously described, dipped in a solution of human albumin, and electroplated in a standard cyanide bath without "striking" or etching.[3] They were then cycled several times between about 370° and 80° K. More recently, extremely thin films deposited from silicone mixtures has been found effective in reducing adhesion. In the development of the above process, measurements were made on the adhesion as a function of the film thickness when the albumin silicone and thermal cycling were omitted. The silver was electrodeposited in patches on rotors from 0.55 cm to 0.02 cm in diameter and spun off in a high vacuum. The film thicknesses were determined from the amount of charge transferred per unit area after

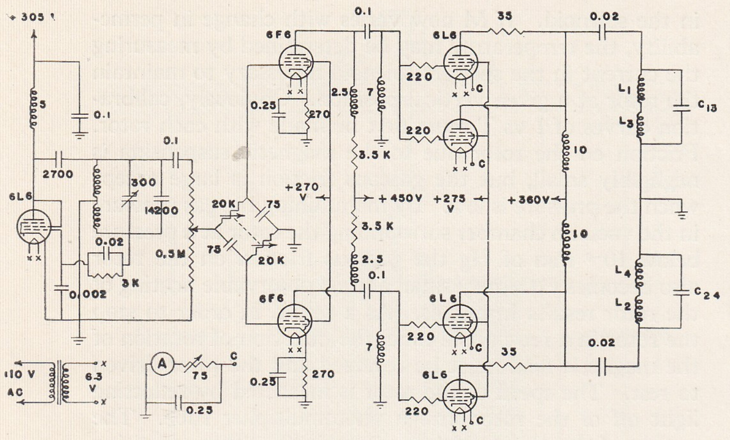

Figure 4—Circuit for driving rotor

careful calibration with standard weighing and interferometer techniques. While there was some scatter in the values obtained, probably due to the nonuniformity of the adhesive bond over the interface, a marked increase in the adhesion was found when the thickness of the silver film was reduced to a few hundred lattice diameters.[1, 2, 3, 10] Although some hypothesis have been advanced, as far as we are aware, this increase in adhesion is not clearly understood.

Dancy and Kuhlthau[13] at Virginia, have carried out detailed studies on the adhesion of electrodeposited chromium to cylindrical steel rotors from 1/8 to 3/8 inch in diameter. They were particularly interested in the effects of the many variables inherent in the electrodeposition process upon the adhesion of the chromium plate. All of their samples were prepared in the following manner. Carefully ground heat treated straight rods of 4140 steel were washed in carbon tetrachloride and given an alkali treatment according to ASTM specification B-177-49 and rinsed in water. The surface of each rod was then prepared according to the procedure under study and immediately immersed in the plating bath on the axis of a cylindrical anode. The plating bath consisted of a mixture of 250 g/l of CrO_3 and 2.5 g/l of H_2SO_4. The plating current was an amp/in^2 and temperature 55° C. Upon removal from the bath each rod was cut into short cylinders which served as rotors. Axial slots were next ground in the rotor in such a way that the chromium deposit was clearly cut into eight axial bands which eliminated the hoop stress. In these instances, no appreciable difference was found in the adhesion on the slotted and unslotted rotors.

Their result showed a marked effect on the adhesion of the length of use of the plating bath. In fact, the adhesion fell to less than half value in a few electrodepositions. To assure maximum adhesion, it was necessary to use a fresh plating solution for each deposition. The effect of dipping the cleaned rotor in various contaminants before the electrodeposition also was investigated. Human albumin, contrary to the results found with copper and silver, seemed to have little or no effect, while such contaminants as ortholeum and SAE No. 30 motor oil gave large reductions in the adhesion. In the case of ortholeum 162 the adhesion could be controlled by proper use of the contaminant i.e. with an immersion of 30 seconds in a 3 cm^3/l solution of ortholeum/62, the effective adhesion of a 0.01 inch chromium plate was roughly 16400 lb/in^2, whereas with a like immersion time in a solution of 10 cm^3/l ortholeum 162 the average adhesion dropped to less than 400 lb/in^2. They also found that a reverse etch of 15 seconds almost completely removed the effect of all of the contaminants tried so far. Micrographs of the rotor surface after the tests showed that when ortholeum is used as a contaminant, the separation takes place at the steel-chromium interface, and the polishing scratches are visible on the rotor surface. On the other hand, when the same contaminant is used, but followed by a 15 second reverse etch, the separation takes place in the chromium plate and the steel surface is not visible in the micrograph.

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