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Subject: Monthly Highlights for March, 1970, Chemical Engineering Department

Sodium-Sulfur Battery

Milled, 0.2% Li_2O samples, containing SiO_2 from 0.005 to 0.08% have been sintered, although the 0.08% samples sintered to a lower density than the other silica-containing materials. Preliminary results reported last month indicated a decrease in strength with increase in silica content after static testing in sodium at 300°C after four weeks. More extensive results indicate that no significant loss in strength is observed in samples containing up to 0.02% SiO_2 after six weeks in sodium, and samples containing 0.04% SiO_2 were unaffected after four weeks in sodium. The remaining samples are still in test, with the test duration limited to six weeks.

Samples containing 0.005 to 0.06% SiO_2 were subjected to the standard, 10-minute, sodium nitrate-mercury electrolysis test and no detrimental effect of the silica content was observed over this range of silica. Samples containing up to 0.2% SiO_2 will be tested as they are prepared.

A milling study designed to improve homogeneity of the 0.2% Li_2O material was completed. By milling four hours before and after the reaction step with a high-purity alumina mill and grinding media, a uniform, fine-grained microstructure was produced. When tested in the 10-minute electrolysis

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test, the material was consistently stronger with a smaller scatter of the data than those samples processed without milling.

A milling study designed to refine grain size as well as improve homogeneity of the 0.7% Li_2O material was continued. The sintered product had a low density, and chemical analysis indicated an unexpected loss of Na_2O during sintering. This material is being resintered and will be tested mechanically and electrically.

Degradation of the ceramic at high current densities up to 1.5 A/cm^2 has been attributed to the discharge of liquid sodium at rapid rates in the microcracks present at the ceramic surface. The pressure created by the rapid discharge of liquid sodium in a confined volume may be great enough to fracture the ceramic and extend the microcracks. If this is true, then discharging of solid sodium should cause cracking and degradation at much lower current densities. To test this hypothesis sodium was discharged into mercury at a 0.2% Li_2O ceramic surface at temperatures above 120°C and below room temperature, the melting point of sodium at a low current density, 10 mA/cm^2 . At room temperature the ceramic exhibited extensive degradation, whereas at 120°C no degradation was observed. This result supports the hypothesis since solid sodium would be expected to exert greater pressures in microcracks than would liquid sodium, thus causing microcrack extension and degradation enhancement.

Nexsil coatings containing 70% graphite on 446 stainless steel specimens have successfully passed a six-month test in molten Na_2S_2 at 300°C at 200 mA/cm^2 .

Corrosion tests on niobium show that it is corroded rapidly by the sodium polysulfide melt.

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Bumper Task Force

Scientific Research Staff personnel, in cooperation with Monroe Process Control and Quality Control, have determined the operating conditions in the white brass plating process responsible for producing the majority of warranty failures. These failures involving zinc-rich bands are associated with current variations and interruptions occurring during machine stoppage. Appropriate steps are to be taken during future breakdown to avoid the production of these failure-prone bumpers.

Attention is now being focused on the overall plating process. An investigation has begun with Monroe Process Control to determine in some detail the nature of the excessive polarization present in a large number of the plating cells. Cell voltages ranging from 7.5 to 15 volts were recorded. The current variation among the anode baskets within a cell was large. These conditions indicate inadequate bath composition stability or control, poor current distribution at the racked bumpers and inefficient power consumption. A program will be initiated, with laboratory research support, to determine to what extent various factors such as bath additives, complexing ions, agitation, etc., are responsible for the difficulties. A study is also underway to determine the influence of agitation, cathodic current density and other factors on the composition and phase structure of the deposit. These data will be especially valuable in relating these parameters to the anodic behavior of the brasses deposited.

Technical assistance is being given MDC in their evaluation of a brass bath adjusted to a higher copper-to-zinc ratio. Of special interest at this time is the investigation of factors that will assure uniform anode corrosion and the study of the anodic reactivities of deposits obtained from the bath.

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Fuel Tanks

An investigation is being undertaken at the request of Dearborn Engine Plant and Chassis Materials Engineering to determine the cause for the early rusting of a large number of fuel tank interiors prior to car assembly. The corrosion proceeds through galvanic cell action between cathodic terne coating and anodic steel exposed at pore sites. This condition is present due to moisture condensation and acid flux residue in tank interiors. Steps have been taken to combat the corrosion through a better wash procedure and the use of corrosion inhibitors. However, for a more positive solution to the problem the objective is to determine whether there is a marked variation in the quality of the terne coating as indicated by the corrosion failures. The investigation indicates that the terne coating on the stamping sheets supplied by Armco, a major supplier, are substantially more porous than the coatings on sheets obtained from U.S. Steel, Republic or Wheeling. Armco has recently modified the hot-dip process by substituting an air-jet finishing technique for the more conventional grease-pot roller step for coating thickness control. The results to date indicate that high porosity is in part related to insufficient coating weight. Substantial differences in coating thickness for both sides of the sheets have been found. Revisions to the specifications are indicated since they refer to only a total coating weight—not weight per side. The burnishing or brushing operation used by Armco on the coatings (0.1 to 0.2 mil average thickness) in an attempt to reduce the porosity count may, at times, aggravate the situation.

The investigation is continuing and test procedures for improving quality control inspection will be recommended.

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Air Pollution from Brake Lining Asbestos

In order to assess the possible contribution of brake lining wear debris to environmental pollution, an estimate was made of the amount of asbestos which is worn away during braking. Data obtained from Ford Test Track Brake Life tests were used. The result—about 100 g of asbestos per car per year—indicates that approximately 11,000 tons per year of brake lining asbestos would be worn away in the U.S. (about 1 million tons of asbestos per year are consumed in the U.S. for all applications).

A report by J. R. Lynch of the Public Health Service indicates that less than 1% of the brake lining wear debris generated during normal braking is fibrous asbestos—the remainder of the asbestos is decomposed into non-fibrous dust particles. However, a question has been raised by I. J. Selikoff, an expert in the field, regarding the adequacy of the electron microscope magnification used to observe the fibers. Dr. Lynch's samples are being checked in Dr. Selikoff's laboratory.

Asbestos has been implicated as a carcinogen, and it is suspected that this carcinogenic effect is associated with the fibrous nature of asbestos. The chronic effect of asbestos decomposition products is not known; however, some work has been reported in which asbestos decomposition products produced an acute toxic effect when injected into the peritoneal cavities of mice.

Because of the possible hazard associated with inhalation or ingestion of asbestos, a study was made of personnel exposure to asbestos dust during compounding, sample preparation, and friction machine testing of brake lining formulations in our laboratory. The evaluation was conducted by Mr. H. B. Lick, Industrial Hygiene Department. Air samples taken during all operations were below the threshold limit value for asbestos dust recommended by the American

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Conference on Governmental and Industrial Hygienists, as measured by a light field count technique. However, it is likely that the smallest fibers would not be observed by this technique. As an added precaution, we have obtained approved dust masks for use during these operations.

In view of the questions which remain regarding the fiber content and respirability of wear dust, and the possible hazard from even the decomposed asbestos, we are giving thought to what we might do to help to answer these questions.

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FRICTION MATERIALS AND SYSTEMS

The objective of this project is to develop and evaluate friction materials and to establish effective test methods and standards. This includes investigations of heat transfer, vibration, and other phenomena which affect the application of friction materials. This program is pursued cooperatively with the Chemical Engineering Department. Progress is reported jointly.

1. New Materials

Friction test (FAST) results have shown that additives such as NaF, Na_3AlF_6 (cryolite), LF, KF, or BaSO_4 play a dominant role in altering the characteristics of the interface between the friction material and the mating surface. Thus, for a given brake lining formulation, the FAST friction level for a particular additive was the same on copper, steel and cast iron surfaces. In many cases this friction level was different for different lining additives. The test samples had essentially the same weight loss on the steel and cast iron surfaces but a much greater wear on the copper surface. The copper rubbing surfaces became much rougher with use than those of cast iron or mild steel. The roughness change with use of each metal surface was affected to varying degrees by the different additives. Studies are in progress to determine if the wettability of the mating surfaces by the additives at their melting point may be important. The studies of the wetting of the copper surface show that KF, NaF, Na_3AlF_6 and PbF_2 wet the copper surface in approximately the same order as the additives improve the friction and wear characteristics of the friction material. Tests are in progress to study the wetting of the additives on the cast iron and steel surfaces.

To obtain a better understanding of the interface phenomenon, friction and wear characteristics of samples of friction material without additive

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were measured in the main sample arm of the FAST machine while samples containing the additive under study were held in an auxiliary arm and processed along the wear track. The results of this dual arm approach indicate that the additive applied to the wear track in this way does influence the interface characteristics for the main arm sample, probably either by adhesion to the mating surface or by transfer through the wear debris that has entered the interface. Studies are in progress to ascertain the underlying mechanism.

Attempts to develop a secondary lining with the desired low wear and low friction coefficient are continuing. As suggested by results from dual-arm FAST tests, linings were prepared with a higher resin-to-asbestos ratio than the linings previously tested. During the break-in period on the Dynamometer, cracks were again observed in the lining. This was probably due to poor kneading of the mix during the cold pressing operation. Preliminary experiments indicate that this difficulty could be overcome by a preforming and repressing operation. Once this processing problem has been resolved new mixes with additives such as MoS_2 , coal and graphite will be evaluated in order to obtain the optimum secondary lining to match the superior drum primary and disc linings that have been found and reported. Dual arm FAST tests will be made to help ascertain the compatibility of the secondary and primary linings before full brake tests are performed.

A patent application has been filed on our alkaline fluoride filled friction materials.

2. Test Methods

A small laboratory shear test fixture has been designed and built which should provide valuable information on the structural strength of linings. This device is somewhat unique in that it uses small (.2 x .3 x .6 inch) samples, thereby permitting samples to be prepared with the shear plane at various angles to the asbestos fibers. Preliminary tests have shown good stability of results from several consistent lining formulations. A lining which

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provided inconsistent results also was found to have large variation of asbestos fiber orientations.

Further shear strength studies will be performed to determine which processing and compounding parameters are significant with respect to reproducibility of results, strength level, and orientation sensitivity.

Air Pollution from Brake Lining Asbestos

In order to assess the possible contribution of brake lining wear debris to environmental pollution, an estimate was made of the amount of asbestos which is worn away during braking. The estimate was based on lining volume worn from various types of brakes using Ford Proving Ground lining life experience. The result --- about 100 g of asbestos per car per year --- indicates that approximately 11,000 tons per year of brake lining asbestos would be worn away in the United States (about 1 million tons of asbestos per year are consumed in the U.S. for all applications). Asbestos has been implicated as a carcinogen, and it is suspected that this carcinogenic effect is associated with the fibrous nature of asbestos. Some work reported in the literature indicates that less than 1% of the brake lining wear debris was fibrous asbestos, whereas roughly half of the original lining was asbestos. The remainder of the asbestos is decomposed into non-fibrous dust particles. However, there is some controversy on this point. In view of the questions which remain regarding the fiber content and respirability of wear dust emitted to the atmosphere, some preliminary measurements are being made of wear dust sampled from a moving air stream downstream of an operating brake dynamometer.

3. Support to Divisions

POC and FAST tests were run on samples from 10 batches each of two brake lining formulations submitted by Ford of Germany. The objective was threefold: to demonstrate the applicability of these quality control tests to formulations used in Germany, to provide benchmarks for comparison with test

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results which may be generated in Germany, and to provide some information about the linings.

The combined results from the chemical and mechanical tests indicated that one of the linings, Textar V-1431FF, was quite consistent from batch-to-batch, even though it has somewhat greater morphological inhomogeneity within a piece than is usually found for U.S. production linings. The other lining, Pagid 300/62, was higher in wear rate than the Textar lining and most U.S. disc brake linings. Both FCC and FAST results indicated that the Pagid lining is fairly inconsistent, which may lead to a quality assurance problem. Both linings generated many times more cast iron wear than typical U.S. disc brake linings.

Also, in response to their request, some suggestions were given on the application of gas chromatography for the quality control of a particular brake fluid used in Germany.

4. Wet Clutches

In order to provide the basis for improved quality control and design of wet clutches, a theoretical model has been developed which simulates the engagement of a pair of un-grooved wet-type clutches. This is based on a study of squeeze films for porous annular disks. The model provides estimates of fiber thickness, viscous shear torque, oil film temperature, and energy dissipation during the engagement. The method and results have been described in two technical reports.

Laboratory apparatus has been developed to measure this squeeze film thickness for two sizes of non-rotating clutch plates. Informal meetings were held with Transmission and Chassis Division personnel who concluded that this new approach warrants further effort. Future studies will be directed to improving the present model and to correlating the theoretical results with Laboratory measurement of oil film thickness vs. time characteristics of production clutch facings.

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5. Analytical Studies

Characteristic values for the deflections and stresses in brake drums have been obtained for most passenger drum designs which are currently in production. The structural behavior obtained is based upon uniform lining pressure distributions for both the leading and trailing shoes. Some study has been made of the effect of the ring which is attached to the drum at the labyrinth seal. The stiffening effect of this ring is so predominant that the analysis of this portion of the drum structure warrants further study.

Consideration of the problem of incorporating brake shoe deflections into the calculation of lining pressures indicates that the analytical method will have to be revised somewhat.

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