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DAVID W. TAYLOR NAVAL SHIP RESEARCH AND DEVELOPMENT CENTER

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ADVANCED MACHINERY LIQUID METAL WETTING,
CLEANING AND MATERIALS
COMPATIBILITY STUDY

by

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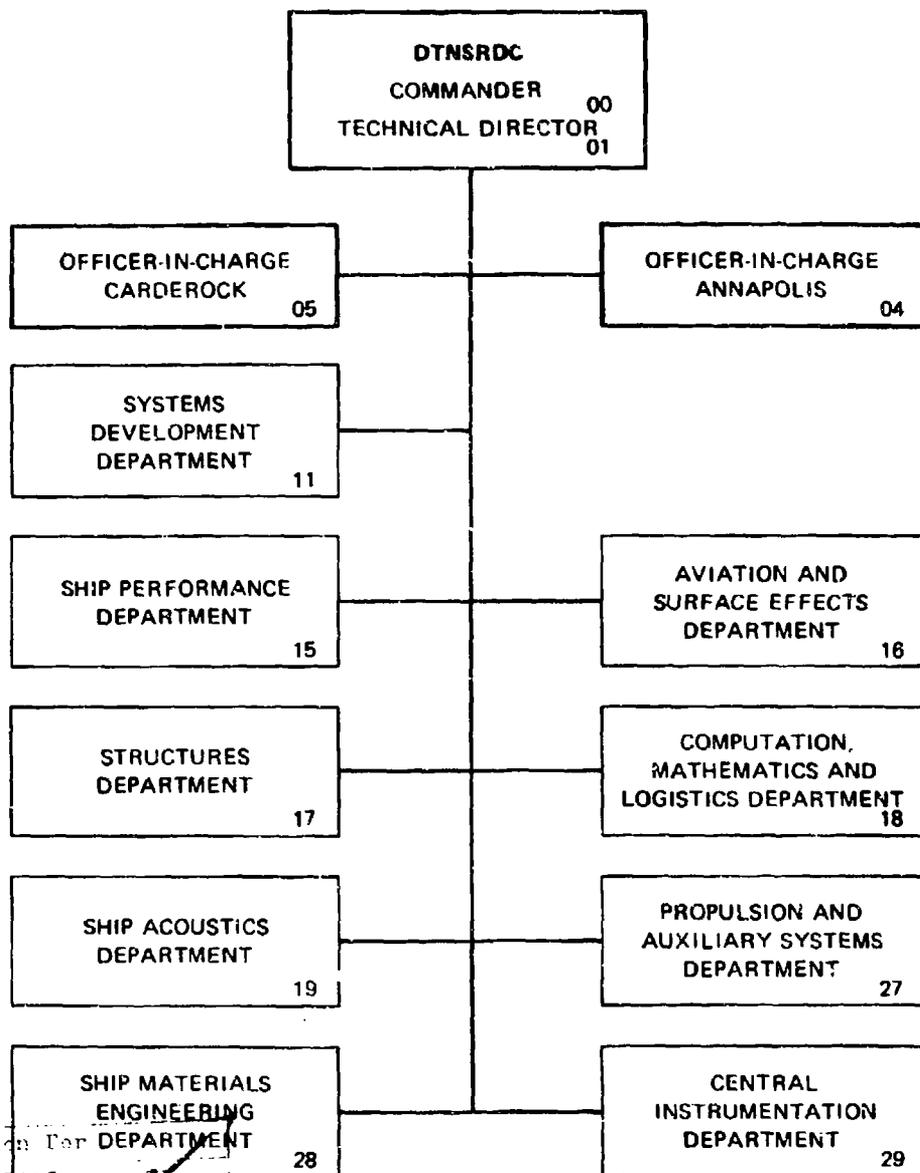
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with collector electrical performance and investigating collector materials/liquid metal compatibility. This study was supported by the David Taylor Naval Ship Research and Development Center (DTNSRDC).

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1.0 ABSTRACT/SUMMARY

Electrical machines which employ high current density liquid metal sliprings (current collectors) for current transfer often utilize alkali metals for the conducting fluids. This study was directed at identifying and solving problems associated with the use of liquid NaK₇₈ (78 w/o potassium, 22 w/o sodium) in prototypic machine current collectors. Areas of investigation included: interfacing of recently developed foam cleaning procedures with collector electrical performance and investigating collector materials/liquid metal compatibility. This study was supported by the David Taylor Naval Ship Research and Development Center (DTNSRDC).

2.0 INTRODUCTION

Homopolar motors and generators that utilize liquid metal slipping current collectors have been developed for naval ship propulsion systems. This program, funded by the David W. Taylor Naval Ship Research and Development Center (DTNSRDC), investigated several problems associated with the use of NaK78 as the conducting fluid in prototypic current collectors. NaK78 was chosen by the Navy as the conductive fluid⁽¹⁾ because of its low melting point, low vapor pressure, acceptable electrical conductance, acceptable hydrodynamic properties, and compatibility with standard current collector structural materials.

Among the problems investigated were: reduction of electrical power losses by minimization of liquid metal/solid metal interface contact resistance through promotion of wetting at current collecting surfaces; testing and evaluation of previously developed foam cleaning procedures on prototypic current collector assemblies; evaluation of current collector operating parameters after foam cleaning; and compatibility of machine structural materials with barium doped NaK78.

During the course of this study, two current collector designs were tested at various collector current densities and rotor tip speeds. A minimal clearance, tongue in groove, braid-type collector assembly obtained from DTNSRDC and a prototypic rectangular tongue in groove collector assembly of in-house design were evaluated at solid/liquid metal interface current densities from 0-3.38 MA/m² and rotor tip speeds from 0-33 m/sec. Electrical tests were performed before and after foam cleaning. Current collecting surfaces of rotors and stators were metallized with tin in an attempt to improve wettability between liquid and solid metals and thus minimize interface electrical power losses. Barium doping of NaK78, the conducting fluid,

was also evaluated as a means for decreasing contact resistance by promoting interface wetting.

Foam cleaning of both current collector prototypes was accomplished utilizing argon driven, alkali stable foam generated from an aqueous mixture of sodium lauryl sulfate. When pumped through the collector assemblies, after electrical performance tests, the foam safely and quickly reacted with and dissolved residual NaK₇₈ retained in the collector internals. This method of cleaning has proven to be a safe and effective method for in-situ cleaning of both in-house and Navy collector prototypes.

Barium doped NaK₇₈ was found to be compatible with a number of typical current collector structural materials at various test temperatures and exposure times. Materials such as copper, metallized copper, fiberglass laminate, rubber, and bearing steel were exposed to 0.5 wt. % barium doped NaK₇₈ at temperatures to 100°C for 250 and 500 hours. Doping of the liquid metal created no compatibility problems with typical collector materials.

3.0 CONCLUSIONS

3.1 Task I - Current Collector Performance and Cleaning Studies

The following conclusions were reached after a series of experiments with both the in-house (Westinghouse) and Navy (DTNSRDC) current collector prototypes:

- Foam cleaning with inert gas driven aqueous foams of 5 w/o sodium lauryl sulfate safely and quickly removed NaK78 and its by-products (oxides and hydroxides) from current collector internals, without requiring machine disassembly. Collector prototypes were cleaned in-situ and returned to service within 72 hours. The use of foam structures permitted highly controlled reaction rates between the aqueous solvent and the liquid metal, thus eliminating the hazards of ignition and explosion of reaction products.
- Foam cleaning was compatible with subsequent collector electrical performance and with collector structural materials. With the Navy (DTNSRDC) collector prototype, no increases in collector electrical losses occurred after foam cleaning. With the in-house collector prototype, electrical losses increased approximately 20%. For both prototypes, post-foam cleaning disassembly, and inspection revealed no damage to collector internals.
- Moist inert gas could be used as a back-up method to foam cleaning. Alcohol cleaning was evaluated as too dangerous for shipboard use.
- Minor removal of tin from metallized collector surfaces occurred during experimental trials where electrical performance tests were followed by foam cleaning, but it was not understood if

removal was due to mechanical wear, dissolution within the liquid metal, or chemical reaction with the foam cleaning solution.

- Turn-around time for the foam cleaning operation was 3 to 4 days. One day was required for securing liquid metal penetrations and air passivating collector internals, one day for inerting, foam cleaning and rinsing, and one day for drying collector internals. This process could be shortened to 2 days.
- The Navy collector prototype, with stator braid inserts, electrically out-performed the in-house collector prototype. Significantly lower electrical losses, at all current densities and rotor tip speeds tested, were attributed to "zero" clearance between rotor and stator, which resulted in minimal electrical losses through the liquid metal annuli and at liquid metal/solid metal interfaces. Rotor and stator current collecting surfaces, including braid inserts, were tin plated to promote improved wetting. Electrical performance was stable throughout the rotor tip speed range tested (11-33 m/sec).
- For the in-house collector prototype, which utilized a tongue in groove design with approximately 1.0 mm clearance between rotor and stator, collector electrical losses were minimized by promoting wetting at the liquid metal/solid metal interfaces. Wetting was promoted by tin plating current collecting surfaces or by doping the liquid metal (NaK₇₈) with approximately 0.5 wt. % barium. Periodic redoping of NaK₇₈ with barium improved electrical performance by replacing barium that was consumed or complexed during machine operation.
- Current collector performance was unaffected by operational time or by machine temperature. Electrical performance did not degenerate during test periods of 4 hours or more. Electrical performance was independent of collector temperature at all temperatures tested (ambient to 70°C).

- Atomization and ejection of liquid metal from labyrinth seals and incomplete filling of annuli with liquid metal were minimized by improved wetting at liquid metal/solid metal interfaces.

3.2 Task II - Machine Materials/Liquid Metal Compatibility Study

- The machine materials tested showed compatibility with NaK in a previous study⁽²⁾, and have exhibited compatibility in barium doped NaK; therefore, the addition of barium to the NaK poses no added problems.

- The fact that the mild steel did not wet would support the fact that chemically, no interaction occurred.

- The cleaning environment was more harsh and resulted in more degradation of the materials than the NaK exposure conditions, particularly in the case of the Buna N Rubber.

- Complete wetting was observed for all of the materials (except the mild steel) exposed to the barium doped NaK. This infers that barium does enhance wetting by NaK.

4.0 RECOMMENDATIONS

4.1 Task I

1. The foam cleaning method developed during this study is recommended for in-situ cleaning of various current collector configurations including tongue in groove and "zero" clearance designs. Liquid metal inventories of 25 cm³ or more per collector were completely removed from in-house and Navy prototypes without damaging collector internals and without deteriorating subsequent electrical performance. The method is therefore recommended for as yet untested collector designs, including those employed on 3000 hp machines.
2. The foam cleaning method is recommended for use on shipboard by semi-skilled personnel. Relatively small quantities of liquid and gas effluents were produced by the process. Thus, for shipboard use, only small effluent reservoirs and limited gas exhaust facilities would be required. The cleaning method is simple and safe enough to be utilized by personnel with little or no training in the handling of corrosive chemicals.
3. It is recommended that a completely self contained and easily transportable foam cleaning system be designed and built which can be utilized either on board or off-board ship to clean collector assemblies of various design and manufacture. A completely portable system can be designed to directly couple to machines at their operating stations. It might include chemical analysis and monitoring instrumentation so that an operator with minimal training could observe and control the entire cleaning process.
4. Additional studies are recommended to determine what causes the removal of electroplated tin from rotor, stator, and braid

surfaces. Long term electrical performance might degenerate with the removal of tin.

4.2 Task II

1. Based on the results found here with 500 hour exposure times and at 100°C temperatures, the materials studied can be recommended for use in a machine utilizing barium as NaK additive.
2. The results of the hardness measurements for the mild steel are inconclusive and should be studied further since an increase in hardness after liquid metal exposure is not an expected result.
3. In any future materials studies it is recommended that cleaning be carried out in an inert environment to virtually eliminate ignition. This would also be more typical of an actual machine cleaning scheme.
4. A better understanding of the hardening which occurred for some materials is a possible area for further investigation. A more in-depth chemical interaction study may explain the hardening results.
5. It should be noted that all studies were performed using typical machine operating temperatures. The possibility of hot spotting and its effects should be considered. A consideration for future study would be the effects on materials exposed in the hot spotting temperature range.

5.0 EXPERIMENTAL PROGRAM

The details of the experimental program for the interfacing of current collector operation with foam cleaning and for the materials compatibility studies are presented in this section.

5.1 Task I - Evaluation of Current Collector Performance with Foam Cleaning

The problem of evaluating current collector performance with in-situ cleaning of collector internals was addressed in an earlier investigation.⁽³⁾ At that time, it was understood that wetting between liquid metal and solid metal substrates would decrease specific electrical contact resistance in liquid metal current collectors and therefore decrease electrical power losses. It was also understood that periodic maintenance of collectors would necessitate that the collector internals be cleaned of liquid metal and its residues. Figure 1 is a flow diagram which defined sequences and interfaces which might occur during normal machine operation. Collector cleaning procedures included the use of in-situ cleaning fluids and aqueous swabbing techniques which could be utilized once the machine had been disassembled. Wetting procedures included various liquid metal additives, as solutionizing rinses, or as metallized collector surfaces, any combination of which might promote wetting at the liquid/solid metal interfaces.

During this investigation the problems associated with wetting, cleaning and their interfacing were significantly simplified. Figure 2 is an updated version of the flow diagram presented in Figure 1. A foam cleaning procedure was developed which effectively replaced the various cleaning procedures referred to in Figure 1. Wetting was obtained by tin metallizing collector surfaces, by additions of barium metal to the

liquid metal, or by installation of metal braid within the stator gap to minimize rotor/stator distance.

Two liquid metal current collector prototypes were evaluated for electrical performance before and after collector annuli were cleaned with an alkali stable foam. The Westinghouse prototype was a tongue in groove design with rotor/stator clearance of ≈ 1.0 mm. The Navy (DTNSRDC) prototype was a tongue in groove design with a braid inserted in the collector grooves to provide a nominal zero (rubbing) clearance between rotor and stator.

Current collector performance was improved by optimizing wetting at the liquid metal/solid metal interface for the in-house prototype and by minimizing rotor/stator clearance for the Navy prototype. Foam cleaning tests were then performed to determine if post-cleaning electrical power losses were significantly different than values obtained before foam cleaning. NaK₇₈ and its reaction products (oxides, hydroxides) were removed from collector internals during machine shut-down by driving a foam generated from an aqueous solution of 5 w/o sodium lauryl sulfate through the annuli with an inert gas.

5.1.1 Minimization of Current Collector Electrical Losses

A review of previously reported efforts to reduce liquid metal current collector losses (3-8) revealed that several approaches could improve wetting. Among the methods investigated were: temperature induced wetting, substrate chemical pre-treatment, ultrasonics, metallization of collector surfaces with easily wetted metals (both consumable and non-consumable), electrically induced wetting, and additives to the liquid metal. This review yielded enough promising methods that testing on current collector prototypes was considered impractical.

A flowcell test apparatus (Figure 3) was designed and constructed into a flowcell loop (Figure 4) to simultaneously evaluate several wetting methods at various experimental conditions including: solid/liquid metal interface current density, liquid metal flowrate,

liquid metal temperature, and running time. The flow cell design permitted detailed measurements of electrical properties, which allowed calculation of specific contact resistance (ϵ_k) at the solid/liquid metal interface (Figure 5). ϵ_k was then evaluated as a function of the various experimental parameters listed above.

Eleven wetting systems were evaluated by holding the liquid metal temperature constant ($\pm 5^\circ\text{C}$) and taking test data at four current densities for each of four liquid metal flowrates. To permit comparison of the wetting systems, interface specific contact resistance was calculated and averaged for each system. The results appear in Table 1.

Table 1
Flowcell Evaluated Wetting Systems

Liquid Metal	Electrode Surface (* - Consumable)	ϵ_k ($10^{-9} \Omega\text{m}^2$)		
		Mean	Std. Dev.	Min.
NaK-Ba	Copper	0.11	0.04	0.03
NaK-Ba	Tin	0.11	0.04	0.03
NaK-Ba	Nickel	0.12	0.05	0.04
NaK	Gold*	0.23	0.02	0.18
NaK	Tin	0.25	0.03	0.19
NaK	Silver*	0.25	0.03	0.18
NaK	Cadmium	0.30	0.03	0.24
NaK	Nickel-Gold*	0.35	0.03	0.29
Nak	Nickel	0.37	0.03	0.32
NaK	Copper	0.38	0.04	0.31
NaK	Zinc	0.44	0.12	0.26

To determine the influence of various test parameters on ϵ_k values, all variables, except the one of interest, were held constant. Using the flowcell test system, interface contact resistance was evaluated as a function of wetting system, interface current density,

liquid metal flowrate, liquid metal temperature, and flowcell running time (Figures 6, 7, 8, 9, and 10).

ϵ_k as a Function of Wetting System

Various metallized electrode pairs were tested for liquid metal-solid metal interface specific contact resistance (ϵ_k). Two liquid metal compositions were used, NaK₇₈ and barium doped NaK₇₈. The following are observations based on test analysis (see Figure 6):

- Eleven wetting systems (see Table 1) were evaluated using the flowcell test assembly, three with NaK-barium as the liquid metal and eight with NaK. They were ranked in ascending value of mean interface specific contact resistance (ϵ_k).
- Barium additions to NaK₇₈ resulted in significant decreases in ϵ_k for the three electrode surface metals tested (Cu, Sn, Ni). Wetting was achieved at an operating temperature of 70°C. Previous test results indicated wetting at room temperature. It appeared that wetting, and the resulting lowering of ϵ_k , would occur over a wide range of normal machine operating and stand-by temperatures. No preference for selecting a current collector surface metal among copper, tin or nickel was evident based strictly upon the results of average ϵ_k measurements.
- Of the electrode metals tested, copper, tin, cadmium, zinc and nickel were not consumed by NaK or NaK-Ba at liquid metal temperatures to 80°C and exposure times to 150 hrs. Although solubilities of these metals were not investigated during this test program, microscopic examination of the flowcell electrodes indicated no etching or metal removal from electrode surfaces.
- Of the electrode metals tested, silver deposited on copper substrate and gold deposited on copper or nickel substrate were characterized as consumable metals when exposed to NaK or NaK-Ba, and their use was of questionable value for rewetting upon machine restart, especially after machine cleaning.

ϵ_k as a Function of Current Density

- For the wetting systems which utilized Nak-Ba as the liquid metal, interface specific contact resistance decreased somewhat with increasing current density (see Figure 7). The decrease may have been due to localized heating of the liquid metal-solid metal interface, rather than a current density related effect. The wetting systems which utilized NaK without barium additive did not indicate any relationship between ϵ_k and interface current density.
- The test data indicated the superiority of wetting systems which utilized NaK with various additives as the liquid metal. Of the three systems tested, NaK with barium additive in contact with copper collector surfaces may be preferred for a machine environment, because no metallization of collector surfaces is required.

ϵ_k as a Function of Liquid Metal Flowrate

- Eleven wetting systems were evaluated for effect of liquid metal flowrate on interface contact resistance (see Figure 8). No clearcut dependence of ϵ_k on flowrate was evident from the test data.
- Graphs of the test data points emphasized the superiority of the wetting systems utilizing barium additive to the liquid metal.

ϵ_k as a Function of Liquid Metal Temperature

- Only two wetting systems were tested for specific interface contact resistance as a function of liquid metal temperature (see Figure 9). Silver and zinc plated metal surfaces were tested with NaK₇₈. No tests were conducted using barium doped NaK₇₈.
- There appeared to be no dependence of ϵ_k on interface temperature, in the range 30°C to 80°C, for wetting systems which, through testing, have demonstrated promising wettability.

ϵ_k as a Function of Running Time

Specific contact resistance did not appear dependent on time duration of current flow through the solid metal/liquid metal interface for tin, silver or cadmium metallized copper, at interface current densities up to of 2.79×10^6 amps per square meter (1800 apsi). The systems tested all used NaK₇₈ as the liquid metal (see Figure 10). For NaK:Zn, ϵ_k decreased significantly, implying that zinc was not initially wetted, but did wet in approximately 30 minutes at 79°C and the stated current density.

Based on analysis of flowcell test results, three wetting methods were selected for further evaluation utilizing in-house and Navy current collector prototypes. The three methods were: 1) NaK₇₈-Ba:Cu (barium doped NaK₇₈ contacting copper collector surfaces), 2) NaK₇₈:Sn (NaK₇₈ contacting tin plated collector surfaces), and 3) NaK₇₈:Cu (NaK₇₈ contacting copper collector surfaces) as a base to evaluate the other two methods. Compatibility with foam cleaning was evaluated by recording, analyzing, and comparing current collector electrical performance before and after foam cleaning.

5.1.2 Foam Cleaning

Homopolar machines for naval propulsion applications that utilize liquid metal sliprings for current collection will require periodic shipboard maintenance. Maintenance may be required for inspection, refurbishment, malfunction, or contamination of the liquid metal (NaK₇₈). In many instances it is too impractical or costly to remove the machine for maintenance. Thus, on-board maintenance requires in-situ cleaning to remove NaK₇₈ from machine internals. A foam cleaning process has been designed and tested for in-situ cleaning of NaK₇₈ from current collector internals.

In designing the cleaning system, several constraints were weighed: (1) the machines will be located on board ship within closed spaces, (2) venting of toxic or explosive fumes and gases must be absolute, (3) safety and reliability of the cleaning method must be

demonstrated, (4) an applied shipboard cleanup method should be performable by personnel who are only moderately trained, (5) the cleaning method must be compatible with subsequent current collector operation, (6) the cleanup scheme must be compatible with machine structural and component materials, and (7) the internal machine configurations are such that alkali metal may not easily drain from collectors and could collect in pockets.

Previous to this study, a detailed investigation⁽³⁾ was undertaken to identify and experimentally evaluate cleaning methods and solutions which would satisfy the previously defined constraints. These included (1) moist inert gas, (2) tertiary butanol, (3) aqueous swabbing, and (4) sodium lauryl sulfate foam.

The moist helium method has been utilized extensively by other investigators⁽⁹⁻¹³⁾ to remove alkali metals and their associated compounds (oxides, hydroxides) from complex geometries, including those containing small cracks and crevices. Previous in-house studies concluded that moist inert gas reaction and removal of NaK₇₈ was reliable at low moisture concentrations (sat. at 25°C) but required excessively long cleaning times. At high moisture concentrations (sat. and at >50°C) vigorous reactions occurred with localized hot spotting and ignition. If used with caution, and if long cleaning times (several days) were not detrimental to planned machine use, this cleaning method could be utilized for in-situ removal of NaK₇₈ and its associated compounds. No experiments were conducted to determine the compatibility of the moist inert gas cleaning method with subsequent current collector electrical performance.

T-butanol, alcohol, and other organic fluids have been studied extensively^(9, 12, 13) as alkali metal cleaning fluids. NaK₅₆ was removed from homopolar machines at the Arnold Engineering Station, Tullahoma, Tennessee utilizing an ethyl alcohol rinse, followed by a rinse of 75% alcohol/25% water, with a follow-up steam cleaning to remove any residual reaction products. Some in-house experiments⁽³⁾ studied the interaction of t-butanol with NaK₇₈, but were discontinued

because of potential alcohol fires resulting from ignition. The use of alcohol as the NaK cleaning fluid was not recommended for shipboard use.

Aqueous swabbing has been utilized at the Westinghouse Research Laboratories to clean up NaK from the current collector region and machine internals of an experimental 3000 hp SEGMAG homopolar generator. This method was described as follows: (1) the machine was isolated, through valves, from the NaK supply loops and ambient air was allowed to fill the machine. (2) The machine was then physically opened and residual, oxidized NaK was mechanically removed from the internal components. (3) Water dampened swabs were then employed to react and remove the remaining small amounts of NaK. Although hydrogen was generated by this process, only small amounts were involved, thus explosive environments were not a problem. This method did require the system to be initially opened to the ambient environment, thus exposing personnel to oxidized NaK. Although they were not encountered, alkali metal burns were possible and, to date, only highly trained personnel have carried out this NaK cleanup process.

Foam generated by mixing an inert gas (helium or argon) with an aqueous solution of 5 weight percent sodium lauryl sulfate safely and quickly reacted with and dissolved NaK₇₈ and its associated compounds. A bench top test apparatus (Figure 11) was employed to evaluate the foam cleaning process. The foam cleaning chamber permitted visual observation of NaK₇₈ samples during reaction with the foam.

Several foam generator hybrids were designed, fabricated, and tested during this and previous studies.⁽³⁾ A detailed schematic of one generator design is pictured in Figure 12. As illustrated, a high density aqueous foam was generated by the mechanical agitation of the solution of 5 wt. % sodium lauryl sulfate. A helium driven, high speed stirrer was incorporated within the containment vessel. A suitable metal baffle arrangement, coupled with a stainless steel wool plug and foam accumulator, permitted the generation of "dry," compact foams which were injected directly into an in-house current collector test stand

assembly. Dynamic testing of this foam generator design was accomplished by injecting foam into a collector assembly containment which had previously been exposed to and contained significant quantities (>50 ml) of NaK₇₈ (Figure 13).

The cleaning of NaK from the full-scale current collector prototype was done in an area outside the laboratory building. As shown in Figure 14, the collector assembly, fitted with a transparent cover to simulate a closed machine, was set up adjacent to the foam generator/accumulator subsystem. A 1-1/2" diameter partially transparent pipe connected the foam generator to the current collector system. Helium was employed as the driving gas. The view of the collector area presented in Figure 14 shows the presence of significant quantities of NaK and possibly super-oxides* that were removed in the cleaning operation.

During the initial phase of the cleaning operation, the transparent cover was removed from the collector assembly and sodium laury sulfate foam was slowly fed into the collector regions. During this operation, no evidence was noted that suggested NaK burning or hot spotting. As the cleaning operation progressed in a controlled fashion, the cover was replaced on the collector housing and foam was permitted to fill the unit.

The entire cleaning process was uneventful; the interaction of the foam with the NaK/products proceeded in a slow and controlled fashion. The foam cleaning operation was followed by a water wash and air drying. Visual inspection of the entire collector assembly after cleaning revealed no damage as a result of burning and/or hot spotting.

Further experiments utilizing the foam generator schematically represented in Figure 12 revealed that the pressure head developed at the generator outlet was insufficient to drive foam through the inlet

* These photographs were originally color transparencies; definite yellow coloration was noted with the NaK residue suggesting the possible presence of potassium super-oxide.

and outlet ports and annuli of a prototype current collector assembly. Attempts to increase the outlet pressure resulted in a disruption of the foam stream. Additional testing with this foam generator was suspended.

A foam generator as pictured in Figure 15 was fabricated. The mechanism of foam generation consisted of forcing inert gas under pressure through a metal frit into a sodium lauryl sulfate solution. The microscopic bubbles thus formed gathered at the head space above the solution level and were ejected from the foam generator under pressure in a continuous stream. The containment and cap were made of stainless steel to minimize corrosion from the sodium lauryl sulfate solution. The aqueous solution capacity was approximately 2 liters with an additional one liter head space for foam accumulation. With a metal frit size of 0.1 μm and a gas inlet pressure of 40 psi, foam was generated and delivered to and through an in-house collector prototype at rates varying from 0.25-1.0 liters/min. Foam densities as generated by the 0.1 μm frit were in the range of 0.05-0.10 g/cc. Ratios of foam generated to sodium lauryl sulfate solution were 10/1 to 20/1. The "dryness" of the foam, and presumably its reaction rate with NaK, was a function of its density. Tests indicated that foam density decreased with increasing gas inlet pressure.

A series of foam cleaning experiments was performed during this study utilizing a positive pressure foam generator design of the type pictured in Figure 15. Two current collector assembly prototypes were successfully foam cleaned, the Westinghouse or in-house design (Figures 16 and 17), and the Navy or DTNSRDC design (Figure 18). Both prototypes were housed within a controlled environment glove box enclosure during foam cleaning operations. Cleaning was done before and after current collector electrical performance trials with NaK.

Table 2 lists the various tasks of a procedure developed for in-situ foam cleaning of current collector internals. The procedure evolved during initial foam cleaning test runs on the in-house collector prototype pictured in Figures 16 and 17. After isolating the prototype assembly from its liquid metal supply loops and draining out as much of

Table 2

IN-SITU FOAM CLEANING PROCEDURE FOR COLLECTOR PROTOTYPES

-
- Isolate machine from liquid metal supply loops (if applicable)
 - Drain residual NaK from collector internal.
 - Passivate for at least 24 hours by exposing collector internals to the environment (an air pump may be necessary to adequately move air through collector internals)
 - Re-inert the collector assembly until moisture content is less than 500 ppm as determined by hygrometer monitoring
 - Foam clean until a minimum of 10 liters of foam per collector has been pumped through the machine (monitoring with a pH meter or combustibles meter will indicate when the reaction between NaK₇₈ and foam has reached an end point)
 - Water rinse with a minimum of 20 liters/collector
 - Dry with heated air or inert gas
-

the NaK₇₈ as possible from collector internals, the machine and its glove box enclosure were opened to the laboratory environment for a minimum of 24 hours to passivate liquid metal exposed to the glove box environment. Failure to passivate resulted in run-away reactions between the foam and liquid metal. Passivation was followed by mandatory re-inerting of the collector prototype and its glovebox enclosure. Failure to re-inert the environment with helium or argon resulted in hotspots and ignition when foam contacted NaK₇₈. Re-inerting was judged adequate when moisture content within the glovebox was less than 500 ppm, as monitored by a hygrometer. A minimum of 10 liters of foam was then pumped through each collector from the foam generator, entering through the liquid metal inlet line and exiting through either the liquid metal outlet ports or the stator labyrinth seals. When the pH fell below 10, as measured with a pH meter or narrow range pH paper, the reaction between foam and NaK₇₈ was assumed to have reached an end point. The prototype collector assembly was then water rinsed with a minimum of 20 liters per collector and air dried.

Foam cleaning of the in-house prototype was accomplished with rotors turning at tip speeds of 11-22 meters/sec. Figures 19, 20, and 21 show, in succession, foam just beginning to exit the stator labyrinth seals, foam flooding the open volume between collectors, and foam collapsing as water was pumped into the collector internals and ejected from the stator labyrinth seals. The foam was structurally sound enough to survive, intact, the hydrodynamic forces incurred by passage through the collector annuli with the rotor turning and with rotor/stator clearances of less than 0.1 cm. No tests were performed with the rotor stationary and it is therefore not clear whether it is necessary to have the collector rotor turning to force the foam into crevices and recesses within the collector internals.

Foam cleaning of the Navy prototype current collector assembly (Figure 18) was performed using a procedure very similar to the one itemized in Table 2. The Navy prototype was a batch fill design with its liquid metal supply contained within reservoirs at the bottom of its stators. Total liquid metal inventory was approximately 15 ml per collector. Draining of the collectors before cleaning retrieved approximately 4 ml of NaK₇₈ from each reservoir. The remainder (\approx 11 ml) was unaccounted for and was probably retained within the metal braid which filled the stator annuli.

The Navy collector prototype was a "sealed" unit as opposed to the in-house prototype design, which utilized labyrinth seals on the stators to retain liquid metal during operation. Although the Navy design successfully retained liquid metal, foam freely exited the collector assembly through various accesses such as vents, bolts, metal/fiberglass seals, and bearing seals. In all probability, foam penetrated recesses within the collector internals not accessible to the liquid metal.

Post-cleaning inspection of both in-house and Navy prototypes revealed little apparent damage to collector materials. Inspection entailed disassembly of stator halves from rotors, thus exposing current collecting surfaces of both rotors and stators. For the Navy collector

pair, stator braids were removed. All collector surfaces in contact with the foam during cleaning were free of damage. There were no indications of hot spots or uncontrolled reaction between the foam and NaK₇₈. Tin metallized areas showed some deterioration in that the tin was partially removed from certain areas, but it is not yet understood whether the tin was consumed by the foam cleaning procedure or by the liquid metal. Fiberglass seals were unaffected by the cleaning procedure. Inspection of epoxy paints used to electrically insulate specific rotor and stator surfaces indicated some deterioration of adhesion between metal and paint, especially at sharp corners and terminations. It is unclear whether this deterioration was the result of electrical performance tests or foam cleaning procedures. Perhaps of most importance, no traces of NaK₇₈ or its reaction products (oxides and hydroxides) were discovered when the collector prototypes were disassembled after foam cleaning and water rinsing. The cleaning procedure detailed in Table 2 safely and completely removed NaK₇₈ from both collector prototypes tested.

The foam cleaning procedure has been thoroughly appraised and meets the constraints previously listed for use on board ship. It can be used for cleaning machines within closed spaces; controllable quantities of potentially hazardous fumes and gases can be safely and easily vented; the safety and reliability of the cleaning method has been demonstrated with two distinctly different current collector prototype designs; the cleaning method was judged compatible with subsequent current collector operation (a detailed discussion appears in subsequent sections of this report); the cleanup scheme was compatible with machine structural and component materials; the cleaning procedure effectively removed NaK₇₈ from all the hidden recesses within the collectors; and finally, the foam cleaning method can be utilized on board ship by personnel who are only moderately trained.

5.1.3 Electrical Performance Experiments with an In-house Current Collector Prototype

Experiments were conducted with an in-house current collector prototype to evaluate the compatibility between the foam cleaning method previously discussed and electrical performance of the collector assembly. Acceptance of the cleaning method by the Navy (DTNSRDC) for use with ship board liquid metal machines required that test results substantiate little or no increase in current collector electrical losses from prior foam cleaning. An existing current collector test stand and a modified collector assembly were utilized to evaluate pre- and post-foam cleaning performance tests.

5.1.3.1 Collector Test Stand and Assembly

The current collector test stand pictured in Figure 22 was utilized for all the electrical performance and foam cleaning experiments. An in-house collector prototype assembly was installed within an inert cover gas glove box (Figure 23) and coupled to an electric motor (5 hp) through a rotary shaft seal in the glove box wall. A selection of drive pulleys permitted collector rotor tip speeds of 11, 22, and 33 m/sec. An argon atmosphere with less than 20 ppm oxygen and 10 ppm water vapor was maintained by a dual column circulation and purification system. Two electrically isolated liquid metal supply loops (Figure 24) pumped NaK₇₈ at the rate of 100 ml/min to each of the collectors in the collector assembly. A GEC liquid metal homopolar generator, with a maximum power output of 30 kW and a maximum current output of 16,000 amps, was utilized as a direct current power supply for the collector tests.

The collector assembly was an existing prototype design⁽³⁾ modified to conduct electrical current from a stator, through a dual rotor mounted on a common shaft, to a second stator, utilizing liquid metal filled annuli for electrical contacts, and necessitating current flow through four liquid metal-solid metal interfaces. The two stators were electrically isolated by insulating them from the collector housing

and by isolating their individual liquid metal supply loops (Figure 24). To permit the flow of 16,000 amps DC through the collector pair, copper bus bars and straps were fabricated and assembled to the upper stator halves and collector housing (Figures 25 and 26). The bottom stator halves were electrically insulated from the bottom collector housing by placing mylar film between the collector housing and stator halves. All copper bolt penetrations through the bottom collector housing were opened up and electrically insulated with Micarta sleeves. The stator assembly was tested for electrical isolation by placing a DC potential across the bottom stator halves and monitoring for current flow. Electrical shorts were eliminated by loosening stator retaining bolts, applying voltage to the stators from a high current source, and tightening retaining bolts until electrical arcing resulted in burning away slivers of metal responsible for the electrical shorts. This procedure was necessary every time the collector test rig was reassembled.

On the basis of previous investigations(14, 15, 16) and discussions with DTNSRDC personnel(17), V groove and double tongue in groove rotor/stator designs were modified to single rectangular tongue in groove pairs in order to minimize liquid metal ejection and gas entrainment. These changes in collector configuration necessitated disassembly of rotors and flanges from the rotor shaft and remachining of rotors and stators. Reassembly required heat shrinking of rotor flanges to the shaft to prevent slippage on machine start-up. Stator inserts were fabricated to increase liquid metal retention by stator labyrinth seals.

Repeated disassembly and assembly of the current collector prototype was necessary to visually inspect current collecting surfaces and collector materials for damage caused by liquid metal or foam cleaning. Procedures for removing corrosion films from current collecting surfaces were necessitated by unplanned and indefinite exposure to the laboratory environment during disassembly inspections. Electrochemical cleaning and mechanical abrasion were utilized at

various times. Electrochemical cleaning was accomplished by a swab technique where the collector surface was electrically connected to the negative output terminal of a direct current power supply and the positive terminal was connected to a 0.6 cm diameter graphite rod wrapped with a cotton swab and saturated with a solution of 100 g/L NaCl. Sufficient voltage was applied to produce a current density of 0.8 A/cm^2 at the swab/metal interface. Residual cleaning solution was wiped from the surface to prevent reoxidation. Mechanical abrasion also effectively removed corrosion products from collector surfaces. Steel wool and silicon carbide were both used. Mechanical abrasion techniques were followed by solvent rinsing or wiping to remove oils or binders.

Electrical performance tests for in-house and Navy collector prototypes were conducted utilizing tin metallized collector surfaces. Electrochemical methods were developed for selectively stripping tin from collector surfaces without damaging the copper substrates. Both methods utilized swab techniques very similar to those utilized for electrochemical cleaning. The solution and current density utilized for electrochemical removal of tin were the same as utilized for cleaning, with polarity reversed. A tin deposit of $13 \text{ }\mu\text{m}$ was selectively removed from collector surface areas at the rate of $5 \text{ cm}^2/\text{min.}$, utilizing a swab which contacted an area of approximately one cm^2 . The entire in-house prototype assembly was completely stripped of tin in 90 minutes. Tin was plated to a thickness of $2.5 \text{ }\mu\text{m}$ on rotors and stators of the prototype in less than 2 hours utilizing a stannous sulfate solution (30 g/L) at an average current density of 0.2 A/cm^2 . The electrochemical swab techniques proved to be safe and efficient methods for selectively cleaning, stripping, and plating current collecting areas of prototype rotors and stators.

5.1.3.2 Collector Performance Tests

Based on previously discussed flowcell results, two methods were selected for promoting wetting at the liquid/solid metal interface: NaK_{78} doped with barium additive (0.1-0.5 wt. %) contacting copper

collector surfaces (NaK-Ba:Cu), and undoped NaK₇₈ contacting tin metallized collector surfaces (NaK:Sn). Undoped NaK₇₈ contacting copper collector surfaces (NaK:Cu) was also tested to provide a baseline for comparing and analyzing experimental results. Pre-foam cleaning and post-foam cleaning electrical performance tests were conducted with each of the three wetting methods to evaluate the effects of foam cleaning on collector performance.

Table 3 details electrical performance of the in-house collector prototype. Collector current and voltage are recorded for various wetting methods at several rotor tip speeds. Performance tests were conducted before and after foam cleaning. As previously discussed, electrical current was introduced into the liquid metal collector prototype at one stator, through a NaK₇₈ filled annulus to one rotor, collector through a common rotor shaft to the other rotor collector through NaK₇₈ filled annulus, and exiting the collector assembly from the other stator. The two stators were electrically insulated at the stator housing to prevent current shorting from one to the other. Figures 25 and 26 show the collector assembly with high current bus network capable of carrying a maximum of 16,000 amps to and from the rotating collector prototype. Voltage taps were connected to each stator. The voltage difference recorded in Table 3 is the sum of the accumulated voltage drops through 2 stators, 2 rotors, the rotor shaft, 2 liquid metal filled annuli, and 4 liquid metal/solid metal interfaces.

5.1.3.3 Interpretation of Performance Test/Results for the In-house Collector Prototype

Figure 27 compares electrical performance curves for the in-house current collector prototype. Collector voltage loss is plotted against current for several combinations of liquid metal and collector surface metal (wetting methods) both before and after foam cleaning. Test results confirmed a linear relationship between voltage and current in the current range of 0-10,000 amps. The linearity indicated operational stability for the collector assembly, regardless of the choice of wetting system. Instability, as indicated by increases in the

Table 3

ELECTRICAL PERFORMANCE OF THE WESTINGHOUSE PROTOTYPE COLLECTOR

Liquid Metal: Collector Surface	Pre-Foam Cleaning	Post-Foam Cleaning	Rotor Tip Speed (m/sec)	Collector Current (amps)	Collector Pair (voltage) (mV)	Running Time (min)	Collector Temp. (°C) Before/After				
NaK-Ba:Cu	✓		11	2000	30.2	1	25/25				
				4000	64.3	1	25/26				
				6000	137.	1	26/27				
				8000	209.	1	28/30				
				10000	305.	1	34/36				
				12000	--	--					
			22	2000	12.8	1	42/42				
				4000	26.1	1	42/42				
				6000	40.9	1	43/44				
				8000	57.4	1	45/46				
				10000	80.1	1	48/50				
				12000	110.	1	53/56				
NaK-Ba:Cu		33	*	13000	--	--					
								2000	13.8	1	55/55
								4000	26.1	1	55/55
								6000	43.8	1	56/56
								8000	62.4	1	57/58
								10000	90.0	1	62/64
	12000	126.	1	71/75							
	✓		11		2000	32.4	1	30/30			
					4000	62.6	1	30/30			
					6000	102.	1	29/28			
					8000	171.	1	30/32			
					10000	272.	1	33/35			
12000					34?.	1	36/40				

Table 3
ELECTRICAL PERFORMANCE OF THE WESTINGHOUSE PROTOTYPE COLLECTOR

Liquid Metal: Collector Surface	Pre-Foam Cleaning	Post-Foam Cleaning	Rotor Tip Speed (m/sec)	Collector Current (amps)	Collector Pair (voltage) (mv)	Running Time (min)	Collector Temp. (°C) Before/After
			22	2000	42.9	1	42/42
				4000	88.5	1	42/42
				6000	136.	1	44/45
				8000	180.	1	47/49
				10000	226.	1	50/53
				12000	339.	1	54/58
			33	2000	42.8	1	58/58
				4000	84.6	1	58/59
				6000	125.	1	62/64
				8000	165.	1	65/67
			*	10000	210.	1	68/73
				12000	--		
NaK-Ba:Cu (redoped)		✓	11	2000	23.4	1	30/30
				4000	46.9	1	31/32
				6000	71.2	1	32/34
				8000	97.5	1	35/37
				10000	132.	1	39/43
				12000	173.	1	44/48
			22	2000	21.2	1	28/28
				4000	42.5	1	29/29
				6000	67.7	1	30/31
				8000	91.6	1	32/34
			*	10000	118.	1	37/41
				12000	--		43/51

Table 3
ELECTRICAL PERFORMANCE OF THE WESTINGHOUSE PROTOTYPE COLLECTOR

Liquid Metal: Collector Surface	Pre-Foam Cleaning	Post-Foam Cleaning	Rotor Tip Speed (m/sec)	Collector Current (amps)	Collector Pair (voltage) (mV)	Running Time (min)	Collector Temp. (°C) Before/After
NaK:Cu			33	2000	21.9	1	29/29
				4000	44.3	1	29/30
				6000	71.4	1	31/33
				8000	100.	1	34/36
				10000	126.	1	37/40
			*	12000	--		46/52
		✓		2000	67	1	28/28
			11	4000	133	1	29/30
				6000	196	1	30/31
				8000	312	1	31/36
				2000	47	1	31/31
			22	4000	97	1	31/32
NaK:Sn				6000	152	1	32/33
				8000	211	1	34/36
			*	10000	264	1	38/42
				12000	--		
				2000	48	1	39/39
			33	4000	96	1	39/40
				6000	147	1	40/41
				8000	198	1	42/44
			*	10000	254	1	45/49
				12000	--		
		✓		2000	21	1	25/25
			11	4000	27	1	25/26
			6000	83	1	26/27	
			8000	152	1	27/29	
		*	10000	--		31/36	

Table 3

ELECTRICAL PERFORMANCE OF THE WESTINGHOUSE PROTOTYPE COLLECTOR

Liquid Metal: Collector Surface	Pre-Foam Cleaning	Post-Foam Cleaning	Rotor Tip Speed (m/sec)	Collector Current (amps)	Collector Pair (voltage)	Running Time (min)	Collector Temp. (°C) Before/After
			22	2000	40	1	34/35
				4000	84	1	35/35
				6000	133	1	35/36
				8000	185	1	37/38
				10000	237	1	40/42
				11500	305	1	49/53
			33	2000	35	1	36/37
			*	4000	--		
NaK:Sn	✓		11	2000	278	1	24/25
				4000	367	1	26/27
				6000	335	1	29/30
				8000	234	1	36/37
				10000	311	1	41/43
			*	12000	--		
			22	2000	49	1	36/36
				4000	100	1	36/36
				6000	152	1	37/38
				8000	205	1	40/41
				10000	260	1	44/46
			33	2000	49	1	44/44
				4000	98	1	44/44
				6000	149	1	44/44
				8000	203	1	45/47
				10000	272	1	49/55

*Unstable electrical performance; arcing; liquid metal ejection

slopes of the voltage/current curves, were commonplace at currents in excess of 10,000 amps. It was also interesting that collectors ejected atomized liquid metal from stator labyrinth seals when electrical performance instability occurred. Arcing between collectors was a common phenomenon in most performance tests when atomized NaK₇₈ was observed in the vicinity of the gap between collectors. Electrical instability and the ejection of atomized NaK₇₈ suggested that the performance of the in-house collector prototype design was not acceptable at currents in excess of 10,000 amps and that instability was probably the result of incomplete filling of collector annuli. Magnetic or electric fields within the annuli may have forced the liquid metal out the labyrinth seals.

The performance curves of Figure 27 show that electrical power losses within the collector assembly were dependent on the choice of wetting method. NaK-Ba:Cu produced lower power losses than either NaK:Sn or NaK:Cu. NaK-Ba:Cu promoted improved wetting at the liquid metal/solid metal interfaces, which reduced interface power losses and contributed to improved electrical performance. NaK:Sn produced lower power losses than NaK:Cu, but not as low as NaK-Ba:Cu. Tin metallized collector surfaces wetted better than unplated copper surfaces and produced lower interface voltage drops.

Foam cleaning was compatible with subsequent collector operation. Cleaning of the in-house collector prototype produced minimal deterioration of electrical performance. The performance curves of Figure 27 show incremental increases in the slopes of the voltage/current curves for tests run after foam cleaning. For the NaK-Ba:Cu wetting system, the increase in slope was approximately 30%. For the NaK:Sn wetting system, the increase was approximately 12%. One might interpret that the increases in slopes reflect increases in liquid metal/solid metal interface resistances. Increases in interface resistances would suggest that the foam cleaning does slightly interfere with rewetting of the collector surfaces by the liquid metal. Previous testing⁽³⁾ had indicated that rewetting improved with running time and

suggested that any incremental increases in collector resistance due to foam cleaning would, for the most part, disappear with machine running time.

Periodic redoping of NaK₇₈ with barium improved collector electrical performance. As indicated on Figure 27, severe deterioration of performance occurred with the NaK-Ba:Cu wetting method when the collector assembly was foam cleaned. It appeared that the foam cleaning operation was responsible for a substantial increase of >150% in apparent collector electrical resistance. The collector prototype was again foam cleaned and another series of performance tests was run with barium doped NaK, which was redoped with approximate 0.2 wt. % barium. Collector resistance decreased to almost the same value as before foam cleaning. The original charge of barium was apparently depleted during collector operation. Redoping promoted rewetting of foam cleaned collector surfaces. The collector performance study did not include an evaluation of barium depletion as a function of machine operating time, temperature, current density, etc. due to funding constraints.

Operational stability of the in-house collector prototype increased with increasing rotor tip speed. Figure 28 shows that collector losses decreased substantially by increasing the rotor tip speed from 11 m/sec to 33 m/sec. Probable cause for the dependence of electrical performance on tip speed was improved filling of the liquid metal annuli from increased centrifical forces, resulting in better pumping action. The dependence of electrical performance on rotor tip speed would suggest that machines utilizing collectors of this particular design might be limited to some minimum rotor tip speed before electrical losses became too high. There might also be some serious limitations on crash-back operation.

5.1.4 - Electrical Performance Experiments with a Navy (DTNSRDC) Current Collector Prototype

Electrical performance experiments were conducted utilizing a Navy current collector prototype pictured in Figure 18. The collector

configuration consisted of 2 stators electrically insulated from one another and 2 rotors mounted on a common drive shaft. Copper braid inserts were placed within slots in the stator annuli which, on proper alignment of the braid, resulted in "zero" clearance between rotors and stators. Test results obtained at DTNSRDC⁽¹⁸⁾ with this same prototype suggested that "zero" clearance of rotors and stators minimized electrical losses through the liquid metal and at the liquid metal/solid metal interface. Liquid metal was contained within reservoirs at the bottom of the stators, thus eliminating the necessity for external liquid metal loops. The total NaK₇₈ inventory was approximately 15 cm³ per collector.

The collector test stand, utilized for electrical performance and foam cleaning experiments with the in-house prototype, was modified and adapted to the Navy prototype collector assembly. All performance and foam cleaning tests were run under a controlled argon environment, as for the in-house prototype.

5.1.4.1 Interpretation of Performance Test Results for the Navy Prototype Collector Assembly

Table 4 lists the sequence of experimental tests performed with the Navy (DTNSRDC) current collector prototype. The machine was subjected to 6 electrical performance tests, 4 with NaK:Sn as the wetting method, and 2 with NaK-Ba:Sn. The assembly was foam cleaned a total of 4 times. It was disassembled and inspected after the performance tests with NaK:Sn, and again after tests with NaK-Ba:Sn. Recharges of liquid metal were added to collector stators twice during the test sequence to compensate for loss in performance. Copper braid was replaced in the collector stators after the series of tests with NaK:Sn and during the inspection of rotors and stators.

Table 5 lists experimental data for the electrical performance tests. Voltage drop across the collector pair was a function of wetting method, performance test sequence (before or after foam cleaning), rotor

Table 4
Test Sequence for the DTNSRDC Prototype Collector

Electrical Performance with NaK:Sn
↓
Collector Idled for 24 Hours
↓
Electrical Performance with NaK:Sn
↓
Foam Cleaned
↓
Electrical Performance with NaK:Sn
↓
Foam Cleaned
↓
Collector Idled for 96 Hours
↓
Electrical Performance with NaK:Sn
↓
Foam Cleaned
↓
Disassembled and Inspected
↓
Stator Braid Replaced
↓
Electrical Performance with NaK₇₈-Ba:Sn
↓
Addition of NaK₇₈-Ba to Stator Reservoirs
↓
Electrical Performance with NaK₇₈-Ba:Sn
↓
Foam Cleaned
↓
Disassembled and Inspected

Table 5
ELECTRICAL PERFORMANCE OF THE DTNSRDC PROTOTYPE COLLECTOR

Liquid Metal: Collector Surface	Pre-Foam Cleaning	Post-Foam Cleaning	Rotor Tip Speed (m/sec)	Collector Current (amps)	Collector Pair Voltage (mV)	Running Time (min)	Collector Temp. (°C) Before/After
NaK:Sn	✓		11	500	0.58	3	27/27
				1000	1.17	1	28/28
				1500	1.77	1	28/28
				2000	2.39	1	28/29
				2500	3.03	11	29/34
				3000	3.65	44	37/60
				500	0.60	1	55/55
				1000	1.12	1	55/55
				1500	1.73	1	56/56
				2000	2.24	1	56/56
2500	2.83	1	56/57				
3000	3.37	1	57/57				
NaK:Sn			*22	500	0.60	1	38/38
				1000	1.06	1	39/39
				2000	2.13	1	39/39
				3000	3.23	1	40/40
				500	0.60	1	38/38
				1000	1.16	1	39/39
				1500	1.68	1	40/40
				2000	2.35	1	41/41
				2500	2.91	1	42/42
				3000	3.39	29	43/69
NaK:Sn	✓		11	560	0.54	2	29/29
				1000	0.99	1	29/29
				1500	1.52	1	29/29

Table 5

ELECTRICAL PERFORMANCE OF THE DTNSRDC PROTOTYPE COLLECTOR
(continued)

Liquid Metal: Collector Surface	Pre-Foam Cleaning	Post-Foam Cleaning	Rotor Tip Speed (m/sec)	Collector Current (amps)	Collector Pair Voltage (mV)	Running Time (min)	Collector Temp. (°C) Before/After
				2000	2.03	1	29/30
				2500	2.55	1	30/30
				3000	3.08	1	31/32
			22	500	0.48	1	33/33
				1000	1.01	1	33/33
				1500	1.62	1	34/34
				2000	2.09	1	34/35
				2500	2.68	1	35/36
				3000	3.19	1	36/36
			33	500	0.61	1	38/38
				1000	1.11	1	39/39
				1500	1.79	1	40/40
				2000	2.47	1	41/41
				2500	2.97	1	42/42
				3000	3.50	1	43/44
				1000	2.17	13	45/51
				500	20.0	1	25/25
NaK:Sn	✓		**22	500	0.51	1	26/26
			**22	1000	0.94	1	26/26
			*	1500	1.46	1	27/27
				2000	1.98	1	28/28
				2500	2.49	1	30/30
				3000	6.21	1	35/36
			22	1000	1.03	160	29/34
				5000	5.57	1	36/40
				10000	11.30	1	49/68

Table 5

ELECTRICAL PERFORMANCE OF THE DTNSRDC PROTOTYPE COLLECTOR
(continued)

Liquid Metal: Collector Surface	Pre-Foam Cleaning	Post-Foam Cleaning	Rotor Tip Speed (m/sec)	Collector Current (amps)	Collector Pair Voltage (mV)	Running Time (min)	Collector Temp. (°C) Before/After
NaK-Ba:Sn (braid replaced)	✓		11	500	0.52	1	29/29
				1000	1.03	1	29/29
				1500	1.47	3	30/30
			2000	2.05	1	30/30	
			2500	2.55	1	32/32	
			3000	3.12	1	32/32	
			22	500	0.55	1	33/33
				1000	1.06	1	33/33
				1500	1.63	1	33/34
			2000	2.21	1	34/34	
			2500	2.70	1	35/35	
			3000	3.32	4	35/37	
			33	500	0.58	1	37/38
				1000	1.13	1	38/39
				1500	1.63	1	39/40
2000	2.55	1	40/41				
2500	3.15	1	42/42				
3000	3.82	1	43/43				
** 33 *	1000	0.98	1	45/46			
	2000	2.04	1	47/47			
	3000	3.14	1	48/49			
	5000	5.32	1	50/51			
	10000	11.13	1	56/58			
	12000	13.98	1	66/70			

*Test assembly held idle for 24 hours before restart

**Test assembly held idle for 96 hours before restart

**Addition of 5cc liquid metal per collector
*

tip speed, and collector current. Running time and collector temperature before and after each test are also listed. The voltage drop across the collector pair was the sum total of voltage losses through the stators, across 2 liquid metal annuli, through 4 liquid metal/solid metal interfaces, through the rotors, and across the common rotor shaft. The stators were electrically isolated at the collector mounts to prevent a shorting path.

Two wetting methods were evaluated for electrical performance, NaK:Sn and NaK-Ba:Sn. Both wetting methods utilized tin metallized current collecting surfaces. The collector prototype was supplied by the Navy with tin plating on the rotors and on the stator braid. No attempts were made to determine the effective lifetime of the tin plating, but visual inspection after several electrical performance trials and foam cleaning tests suggested that tin was slowly consumed by the NaK₇₈ and/or the foam cleaning process.

The addition of 1.0 wt. % barium to NaK₇₈ contributed little or nothing to collector electrical performance for the Navy prototype. The "zero" clearance between rotor and stator braid apparently promoted wetting between liquid metal and solid metal, thus camouflaging any contribution to wetting by the barium. Although current collecting surfaces for rotors and stators were tin plated, it is assumed that wetting would occur in the absence of tin plating and barium doping, if the clearance between rotor and stator were close enough to mechanically abraid collecting surfaces. If clearances were to open up between the rotors and stators during collector operation, additions of 1.0 wt. % barium might significantly improve electrical performance.

To evaluate the compatibility of foam cleaning with collector performance, pre- and post-cleaning electrical performance trials were conducted. For the performance trials, rotor tip speeds of 11, 22 and 33 m/sec were utilized. For the foam cleaning experiments, rotor tip speeds of 11 m/sec were used to force the foam through the collector annuli, the stator braid, and the liquid metal reservoirs.

Figure 29 shows performance curves for the Navy collector prototype. Experimental data for the two wetting methods tested (NaK:Sn and NaK-Ba:Sn) fell within a narrow range, regardless of whether the tests were conducted before or after foam cleaning. Foam cleaning did not contribute to measurable deterioration of subsequent collector electrical performance. Disassembly and inspection of collector internals after foam cleaning revealed no corrosion products on either rotor or stator current collecting surfaces. The collector braid was clean of any traces of NaK₇₈ or its salts.

Idling the collector assembly for one or more days with liquid metal within the collector reservoirs promoted severe deterioration of electrical performance on start-up. Figure 29 shows greater than an order of magnitude increase in voltage drop across the collector pair. Electrical performance was restored by the addition of 5 cm³ of liquid metal to each collector. The deterioration and subsequent restoration of performance was obtained with undoped and barium doped NaK₇₈, indicating that deterioration of performance was not caused by loss of wetting. Apparently not all the liquid metal returned to the reservoirs when the collector assembly was shut down. Sufficient quantities may have been retained within the stator braid and within the collector internals to deplete the supply and prevent complete filling of collector annuli on start-up. Although the problem was temporarily solved by the addition of more liquid metal, a more permanent solution might be to redesign the collector reservoirs to hold more liquid metal.

Collector performance was unaffected by changes in rotor tip speed in the range of 11-33 m/sec. Figure 30 shows very little increase in load resistance for the collector assembly as the rotor tip speed was increased from 11 to 33 m/sec. The test data was accumulated from all the experiments conducted with the Navy prototype. As is evident in Figure 30, all the data fell in a narrow range, indicating that test results were independent of not only rotor tip speed, but also wetting method and foam cleaning. Although no experiments were performed at tip speeds less than 11 m/sec., the test results imply that the Navy proto-

type collector design should operate satisfactorily at tip speeds approaching stall speed and that electrical performance might not significantly deteriorate during crash-back tests.

Electrical performance was independent of the running time for the Navy prototype. Several tests were run for extended time periods from 13 minutes to 4 hours at constant rotor tip speed and current flow. No increase in voltage drop occurred with time. Although these tests were only of relatively short time durations, the results suggest that deterioration of electrical performance is not related to the operating time for the DTNSRDC collector assembly.

Electrical performance was independent of operating temperature for collector temperatures between ambient and 60°C. Comparisons of test data taken at various operating temperatures (Table 5) show no measurable changes in collector resistance. Previous studies⁽³⁾ had determined that one of the primary contributions to deterioration in collector performance was loss of wetting at the liquid metal/solid metal interface, and that one way to improve wetting was to increase substrate temperature. The Navy prototype, with its "zero" clearance between rotors and stators and its stator braid inserts, promoted wetting at the liquid metal/solid metal interface and retained liquid metal in the annuli at all operating temperatures tested.

5.2 Task II - Machine Materials/Liquid Metal Compatibility Study

Materials compatibility with the current collector fluid is of critical importance in guaranteeing continuous long term machine operation. This task investigated the effects of exposure of various machine materials to barium-doped NaK₇₈ liquid metal at 100°C for 250 and 500 hours. As with previous studies^(2, 19) to identify potential compatibility problems involving liquid metals reacting with current collector machine materials, these determinations are also required for similar materials when NaK₇₈ with the addition of barium for increased surface wetting, is utilized. The results of this investigation are expressed quantitatively; characterizations of exposed specimens were compared with data obtained on as-received and thermally-aged materials.⁽²⁰⁾

5.2.1 Definition of Materials

Materials studied were:

- NEMA grade G-10 fiberglass laminate, flat stock
- Buna N rubber, sheet stock
- Type 316 stainless steel, sheet stock
- OFHC copper, sheet stock
- Nickel plated (0.5 mil) OFHC copper, sheet stock
- Typical ball bearing steel (AISI E52100, 1.0 C, 1.5 Cr, 0.3 Mn, trace P, S, Si balance Fe)
- Mild steel

The basis for this selection for compatibility evaluations with NaK:Ba is that some of these materials are used in NaK current collectors today, and previous compatibility studies(2,19) explored the capabilities and limits of these machine and current collector materials when NaK was utilized as the slipping fluid. Morphological, weight change, dimensional, and hardness properties were determined in quantifying the materials studied.(2)

5.2.2 Sample Preparation and Exposure

The specimens were cleaned, weighed, mounted on specimen trees, Figure 31, and sealed in stainless steel capsules, Figure 32, containing barium doped NaK (saturated sol. at 100°C). A constant temperature oil bath maintained at 100°C was utilized for isothermal specimen exposure. The eight test conditions under which the materials were exposed are detailed in the test matrix in Table 6.

5.2.3 Specimen Characterization

Scanning Electron Microscopy (SEM), coupled with Energy Dispersive Analysis by X-ray (EDAX), was utilized to evaluate exposed samples for surface chemistry changes or corrosion effects; surface morphology and elemental compositions were also obtained. Rockwell

Table 6

Machine Materials/Liquid Metal Compatibility Study Test Matrix*

Materials	As-Received	Thermally Aged (Argon) 500 hrs	NaK ₇₈ + Ba					
			250 hrs		500 hrs		**500 + hrs	
			Vapor	Liquid	Vapor	Liquid	Vapor	Liquid
Fiberglass Laminate	✓	✓	✓	✓	✓	✓	✓	✓
Buna N Rubber	✓	✓	✓	✓	✓	✓	✓	✓
316 S.S.	✓	✓	✓	✓	✓	✓	✓	✓
OFHC Cu	✓	✓	✓	✓	✓	✓	✓	✓
Ni Plated Cu	✓	✓	✓	✓	✓	✓	✓	✓
Ball Bearing Steel	✓	✓	✓	✓	✓	✓	✓	✓
Mild Steel	✓	✓	✓	✓	✓	✓	✓	✓

* All tests made at 100°C.

** Extended exposure time to be determined after characterization of specimens exposed for 500 hours.

hardness measurements were carried out on all materials except the Buna N Rubber.

5.2.4 Experimental Practice

5.2.4.1 Doping NaK₇₈ with Barium

The NaK₇₈ used for exposure was doped with an approximate 0.5 weight percent barium. This was done in an argon glovebox by adding a predetermined weight of barium rod to two liters of NaK₇₈. The NaK was heated for approximately eight hours a day for five days at a maximum temperature of 200°C. Daily measurements of the dissolution of the barium are given in Table 7. The undissolved barium rod was removed from the NaK and the NaK was loaded into the specimen exposure capsules at a temperature of ~ 550°C. Upon transferring the NaK into the exposure capsules, a slurry was observed in the bottom of the NaK container.

5.2.4.2 Sample Preparation

Specimens were cut from the materials to appropriate sizes (1" x 1.5"). The metallic material, all exposure capsules, and sample trees were degreased ultrasonically in acetone, ethanol washed, and oven dried. The organic base materials were used as-received. The specimens were mounted on the specimen trees and with the associated capsules, transferred into the ante-chamber of an argon glovebox. They were baked in the ante-chamber at 100°C for approximately 24 hours to degas, then allowed to cool to ambient temperature and transferred into the glovebox proper. The material specimens were removed from the specimen trees, weighed, and remounted. Each specimen tree held samples for NaK liquid and vapor exposure. In the argon glovebox barium doped NaK was placed in each capsule (except for capsules containing materials for thermal aging only) at a level to completely submerge the lower samples, while the top samples remained above the NaK level (vapor). The caps of the exposure capsules which contained the organic materials (Fiberglass

TABLE 7

Measurements of Barium Rod Dissolution in NaK₇₈
(22 of NaK)

Day	Time	NaK Temp.	Weights (g)						w/o Ba
			w ₁	w ₂	w ₃	w ₄	w ₅	w ₆	
1	11:00 a.m.	185°C	38.35	5.60	32.75	35.47	2.72		0.16
2	9:00 a.m.	185°C	36.75	5.64	31.11	35.47	4.36	1.64	0.26
3	10:00 a.m.	210°C	35.64	5.68	29.96	35.47	5.51	1.15	0.33
4	10:00 a.m.	180°C	33.91	5.76	28.15	35.47	7.32	1.81	0.44
5	8:50 a.m.	17°C	30.59	5.82	24.77	35.47	10.70	3.38	0.62

Note: Wetting of copper occurred at ~ 0.1 w/o Ba
Wetting of stainless steel occurred at ~ 0.25 w/o Ba

w₁ - wetted Ba and pan

w₂ - pan

w₃ - wetted Ba

w₄ - original Ba

w₅ - total weight of dissolved Ba

w₆ - weight Ba dissolved since previous measurement

w/o - weight percent Ba in the NaK₇₈

Laminate and Buna N Rubber) were fitted with valve assemblies to facilitate capsule venting. The specimen trees were loaded into the capsules, capped by hand tightening. The capsule assemblies were transferred out of the glovebox and final sealed by applying 150 ft. lbs. torque to the closure fittings. All capsules were then lowered into the constant temperature oil bath. The temperature of the oil bath was maintained at $100 \pm 1^\circ\text{C}$.

5.2.4.3 Specimen Characterization

At the completion of the liquid metal exposure, the exposure capsules were transferred into the argon glovebox, and the excess NaK emptied out. In preparation for characterization, the materials (still mounted on the trees) were decontaminated outside the glovebox using a high velocity water flush, followed by a distilled water/ethanol wash, and oven drying.

Scanning Electron Microscopy (SEM) and the corresponding Energy Dispersive Analysis by X-ray (EDAX) were used to characterize all materials in the test matrix. SEM micrographs were obtained at 500X and 2000X to evaluate the surface features. EDAX spectra were obtained from a 100X rastered area to determine the elemental composition, specifically looking for traces of Na, Ba, and K.

5.2.5 Results and Discussion

At the completion of exposure times it was found that all samples submerged in the NaK liquid were entirely wetted with barium doped NaK except the mild steel, which was striated. Some oxidation of the NaK took place in the 250 hour OFHC copper and nickel plated OFHC copper capsule. Oxidation products migrated up the sample tree and onto the other samples held there for vapor exposure.

Table 8 shows the weight determinations made before and after the specimens were exposed. Essentially, no significant weight changes were noted. The Buna N rubber is the only material which had a measurable weight change (loss after exposure) under every test

TABLE 8

Weights of Samples Before and After Exposure to
NaK₇₈ (0.5 w/o Ba) at 100°C

Exposure Time	Material	wt. Before Exposure (g)	wt. After Exposure (g)	Difference*
500 hrs, Thermal	Laminate	1.60	1.61	+0.01
	Buna N	1.66	1.61	-0.05
250 hrs, Vap.	Laminate	1.81	1.81	
	, Vap. Buna N	1.57	1.52	-0.05
	, Liq. Laminate	1.82	1.82	
	, Liq. Buna N	1.62	1.56	-0.06
500 hrs, Vap.	Laminate	1.84	1.84	
	, Vap. Buna N	1.66	1.55	-0.11
	, Liq. Laminate	1.74	1.73	-0.01
	, Liq. Buna N	1.72	1.51	-0.21
500 hrs, Thermal	Stainless Steel (S.S.)	5.54	5.54	
	Bearing Steel (B.S.)	44.07	44.07	
250 hrs, Vap.	S.S.	5.80	5.80	
	, Vap. B.S.	43.04	43.04	
	, Liq. S.S.	5.73	5.74	+0.01
	, Liq. B.S.	44.36	44.37	+0.01
500 hrs, Vap.	S.S.	5.73	5.73	
	, Vap. B.S.	43.04	43.04	
	, Liq. S.S.	5.63	5.63	
	, Liq. B.S.	44.36	44.36	
500 hrs, Thermal	OFHC Cu	14.15	14.15	
	Ni/OFHC Cu	14.23	14.24	+0.01
	Magnetic Steel	13.87	13.87	
250 hrs, Vap.	OFHC Cu	14.05	14.05	
	, Vap. Ni/OFHC Cu	14.14	14.14	
	, Liq. OFHC Cu	14.12	14.12	
	, Liq. Ni/OFHC Cu	14.03	14.03	
500 hrs, Vap.	OFHC Cu	14.08	14.08	
	, Vap. Ni/OFHC Cu	14.13	Sample Lost	
	, Liq. OFHC Cu	13.91	13.91	
	, Liq. Ni/OFHC Cu	14.22	14.22	

(continued)

TABLE 8

Weights of Samples Before and After Exposure to
NaK₇₈ (0.5 w/o Ba) at 100°C

(continued)

<u>Exposure Time</u>	<u>Material</u>	<u>Exposure (g)</u>	<u>Exposure (g)</u>	<u>Difference</u> [*]
250 hrs, Vap.	Magnetic Steel	12.22	12.22	
, Liq.	Magnetic Steel	13.10	13.09	-.01
500 hrs, Vap.	Magnetic Steel	11.66	11.65	-.01
, Liq.	Magnetic Steel	12.50	12.48	-.02

Vap. - Vapor exposure

Liq. - Liquid exposure

*+, wt. gain after exposure

-, wt. loss after exposure

condition. The largest weight changes also occurred with the Buna N rubber samples. Material was also lost from some of the liquid metal exposed Buna N rubber samples when they ignited during cleanup. For the remainder of the samples, the maximum weight change was + 0.02 grams. On a percentage basis, this ranged from 0.08 to 0.63%.

Samples of all materials in the as-received condition, after 500 hours liquid NaK₇₈ (Ba) exposure, and after thermal aging were characterized by SEM and EDAX. The SEM micrographs and the EDAX spectra are shown in Figures 33 through 53. For the Nema Grade G-10 Fiberglass Laminate, Figures 33-35, the SEM micrographs showed the removal of matrix material from the fiberglass after 500 hours. Also, the EDAX spectra obtained from a 100X rastered area showed traces of Ba and K after 500 hours exposure. The 500 hour NaK (+Ba) exposed Buna N rubber, Figure 37, also exhibited a loss of material when compared with the as-received sample, Figure 36 and thermally aged, Figure 8. The EDAX spectra, see Figure 37, also revealed traces of Ba.

No apparent surface chemistry changes or corrosion occurred after exposure of the 316 stainless steel, Figures 39-41; the OFHC copper, Figures 42-44; the nickel plated OFHC copper, Figures 45-47; the ball bearing steel, Figures 48-50; and the mild magnetic steel, Figures 51-53. The SEM micrographs of the 316 stainless steel and the ball bearing steel exposed to NaK (+Ba) for 500 hours, Figures 48 and 49, show the surface to be cleaner (as a result of NaK exposure) when compared to the as-received samples, Figures 39 and 48. Micrographs and EDAX spectra of the OFHC copper revealed the presence of a residue containing Ba in the micro-pitted areas of the sample. This may be due to a failure to completely remove the NaK₇₈ (+Ba) from these micro-pits during cleanup. As a result, during handling in air, the NaK leached out to the surface and was detected by EDAX. Thermal aging without NaK for 500 hours had no visible effect on the surface morphology or elemental composition of any of the materials; this is shown in Figures 41, 44, 47, 50, and 53.

The materials' Nema Grade G-10 Fiberglass Laminate, 316 Stainless Steel, OFHC Copper, Nickel plated OFHC Copper, and Magnetic Steel were tested for hardness by the Rockwell Method. The Buna N Rubber was too soft to be tested by this method. The measurements and the Rockwell Hardness Scale on which they were tested are given in Table 9. The observed hardnesses are also plotted in Figure 54. The 316 Stainless Steel and the Bearing Steel had virtually no change in hardness under any exposure condition. The Magnetic Steel underwent an apparent and not expected increase in hardness for all exposure conditions. The Nickel plated OFHC Copper could not be fully evaluated by hardness due to some samples lost from the test matrix. The samples available had a very small increase in hardness. The OFHC copper and the Fiberglass Laminate had similar patterns in hardness changes, as seen in Figure 54. All exposed samples had an increase in hardness from that of the as-received specimen, but the hardness of the 500 hour liquid NaK exposed samples was less than that of the 250 hour exposed sample.

Table 9

Rockwell Hardness Measurements of Compatibility Test Materials

<u>Exposure Condition</u>	<u>Material</u>	<u>Scale</u>	<u>Hardness</u>
As-received	OFHC Copper	Rockwell "F"	83
500 hrs., thermal	Cu	1/16" Ball	87.5
250 hrs., vap.	Cu	60 Kg	88
, liq.	Cu		86
500 hrs., vap.	Cu		83.5
As-received	Ni plated OFHC Cu	Rockwell "B"	Not Available
500 hrs., thermal	Ni/Cu	1/16" Ball	42.5
250 hrs., vap.	Ni/Cu	100 Kg	42
, liq.			40.5
500 hrs., vap.			Sample Last
, liq.			41
As-received	Magnetic Steel (M.S.)	Rockwell "B"	58
500 hrs., thermal	M.S.	1/16" Ball	63.5
250 hrs., vap.	M.S.	100 Kg	65
, liq.	M.S.		62
500 hrs., vap.	M.S.		69
, liq.	M.S.		69
As-received	Laminate	Rockwell 30X	79
500 hours, thermal	Laminate	1/4" Ball 30 Kg	87.5
250 hrs., vap.	Laminate		88
, liq.	Laminate		86
500 hrs., vap.	Laminate		89.5
, liq.	Laminate		83.5
As-received	Stainless Steel (S.S)	Rockwell "B"	84
500 hrs. thermal	S.S.	1/16" Ball 100 Kg	83.5
250 hrs., vap.	S.S.		84
, liq.	S.S.		84
500 hrs., vap.	S.S.		83
, liq.	S.S.		84
As-received	Bearing Steel (B.S.)	Rockwell "C"	62.5
500 hrs., thermal	B.S.	Diamond Pyramid	62
250 hrs., vap.	B.S.	150 Kg	62
, liq.	B.S.		62
500 hrs., vap.	B.S.		62
, liq.	B.S.		62

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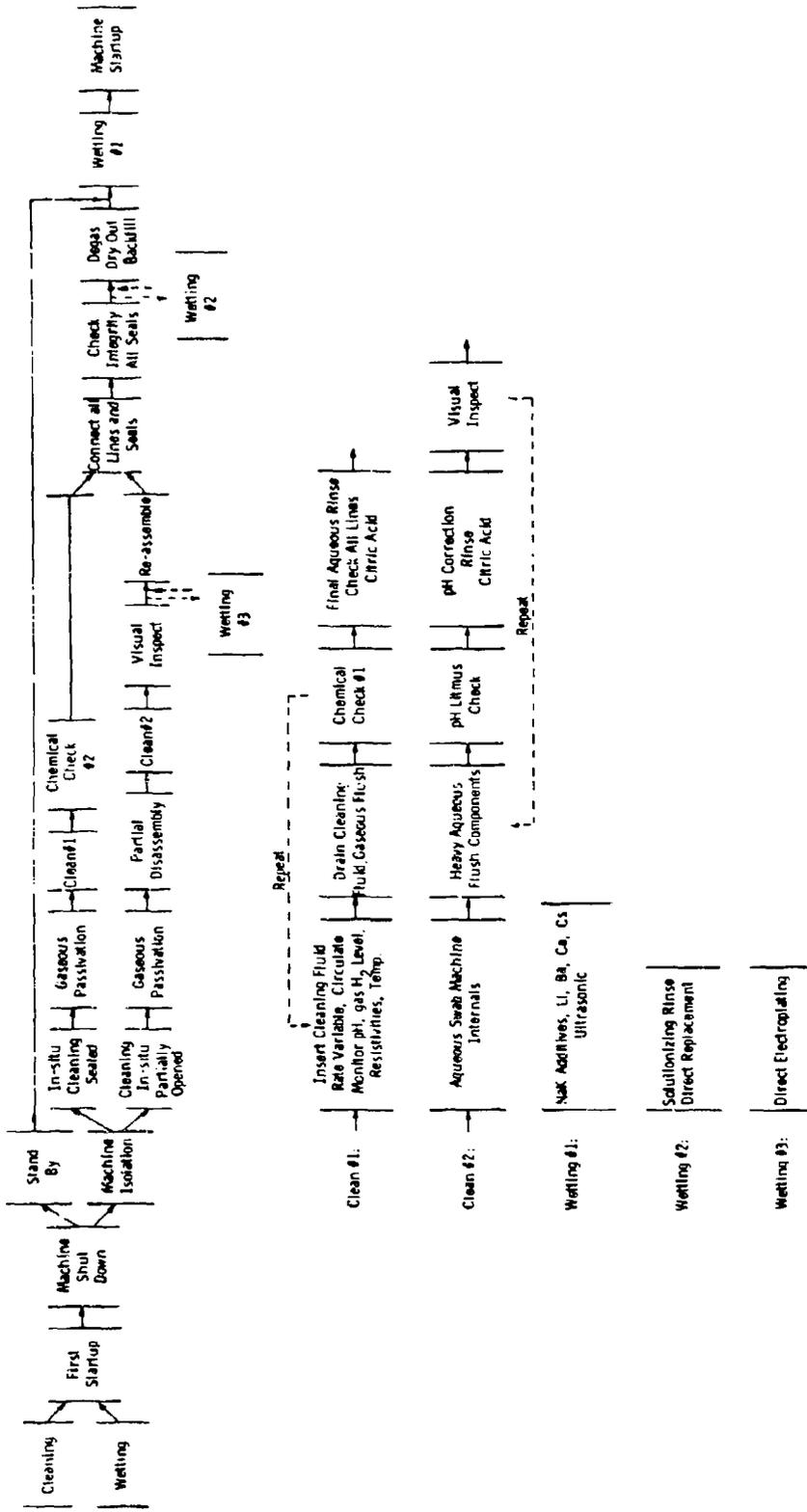
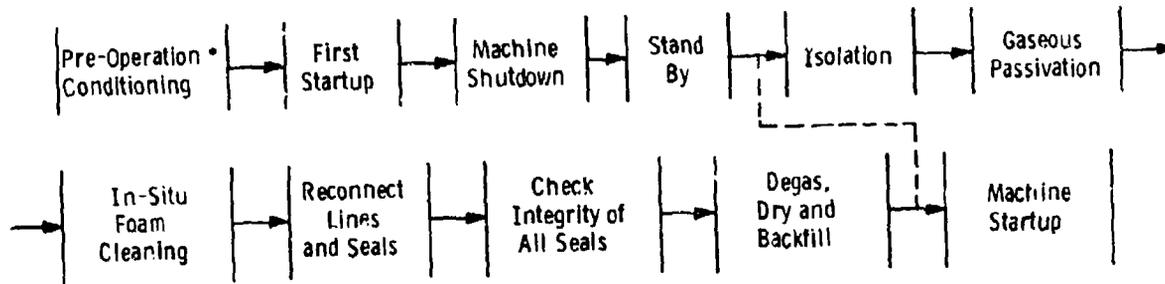


Fig. 1—Defining sequencing and interfaces

Dwg. 4246827



- Pre-Operation Conditioning: 1) Tin Plating of Collectors
- 2) Barium Doping of Liquid Metal
- 3) Installation of Stator Braid

Fig. 2--Updated flowchart defining sequencing and interfaces

Dwg. 6376A79

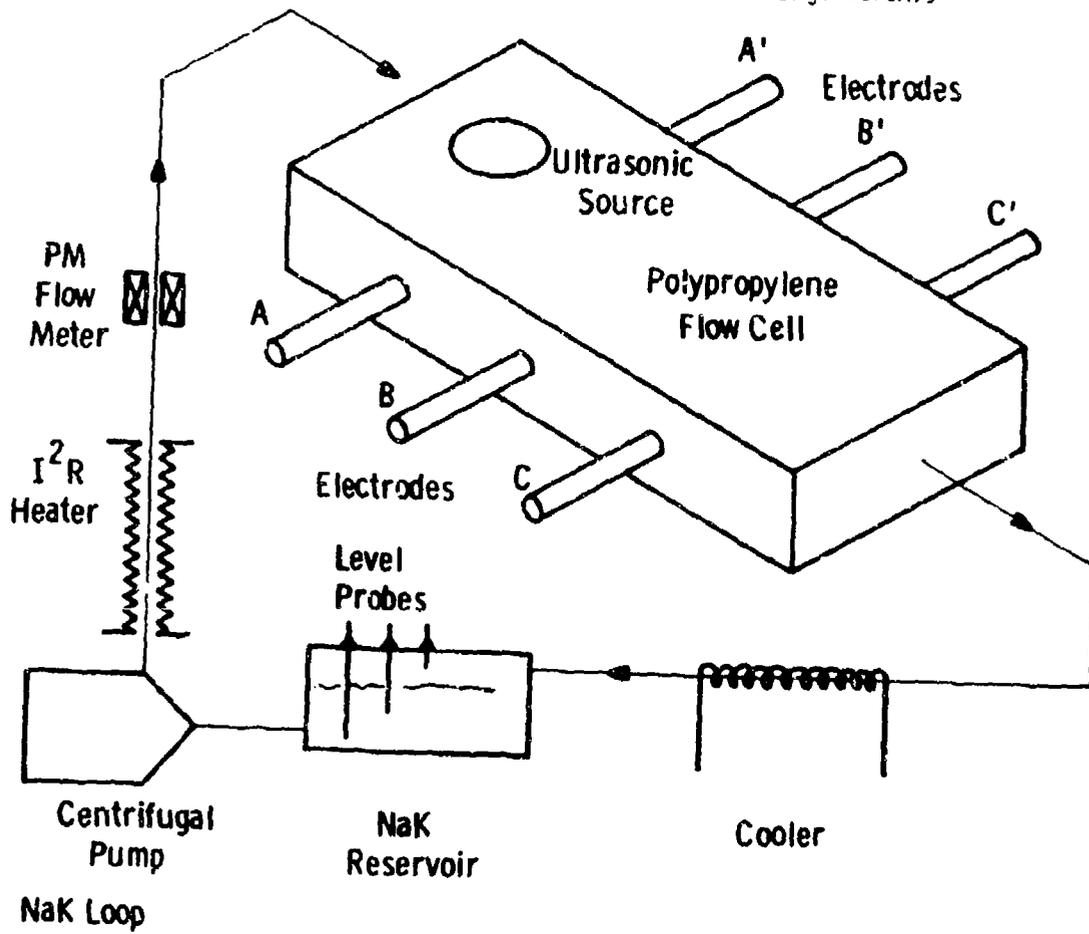


Fig. 3—Flow-cell test apparatus for the evaluation of specific contact resistance

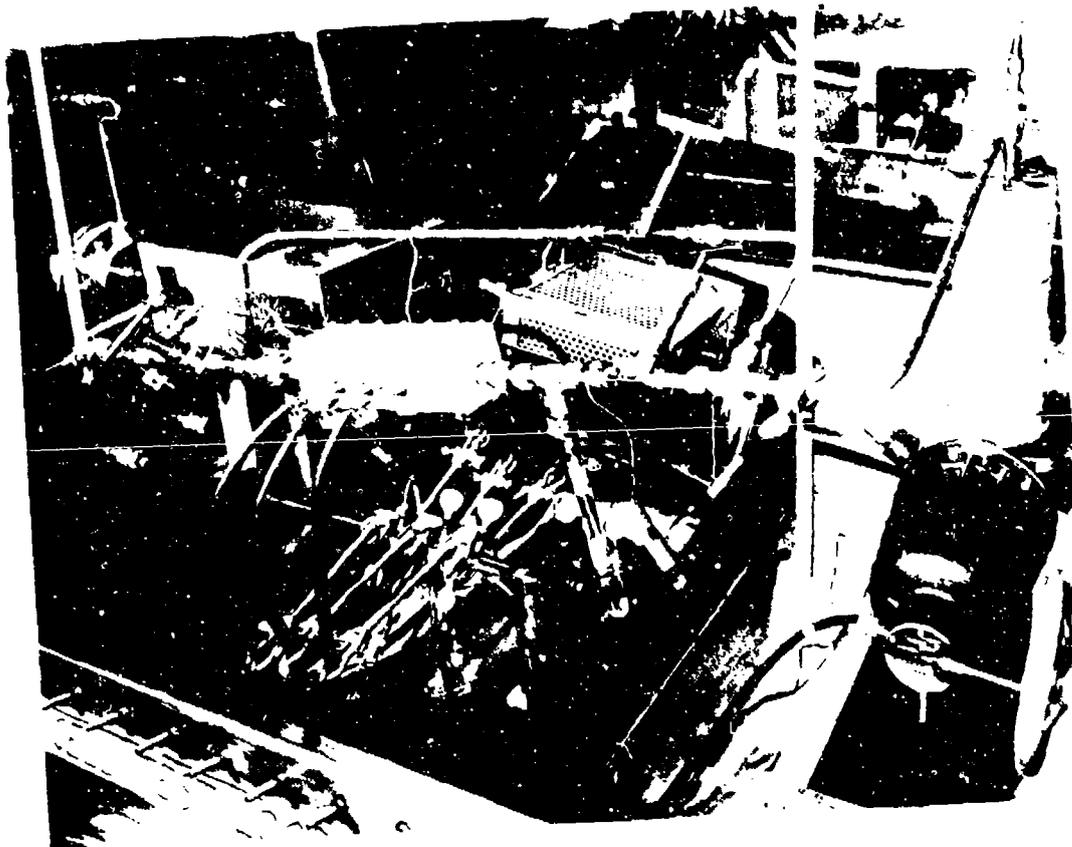
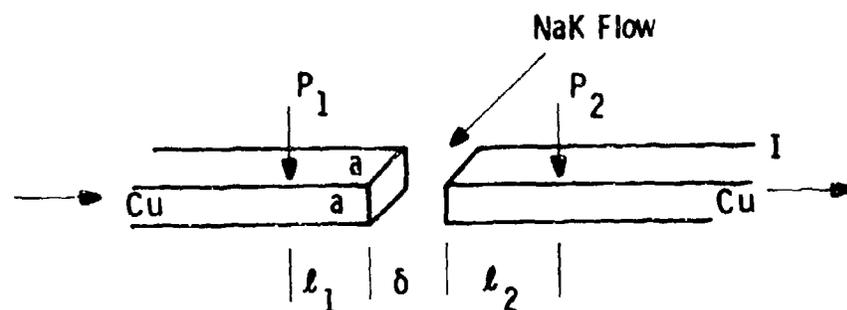


Figure 4. Flow cell loop.

DWG. 6376A80



DC Current (Amps)	I
Current Density (I/a^2)	J
NaK Gap Width	δ
Copper Electrode Area	a^2
Couple Potential Drop	$P_1 - P_2$
Specific Contact Potential	ϵ_k

$$\epsilon_k = \frac{(P_1 - P_2)_{\text{meas}} - (V_{SM} + V_{LM})}{J}$$

where

V_{SM} = Potential Drop in Solid Metal

$$= \frac{I (l_1 + l_2)}{\sigma_{SM} a^2}$$

V_{LM} = Potential Drop in Liquid Metal

$$= \frac{I \delta}{\sigma_{LM} a^2}$$

Fig. 5—Flow-cell schematic for measurement of specific contact resistance ϵ_k

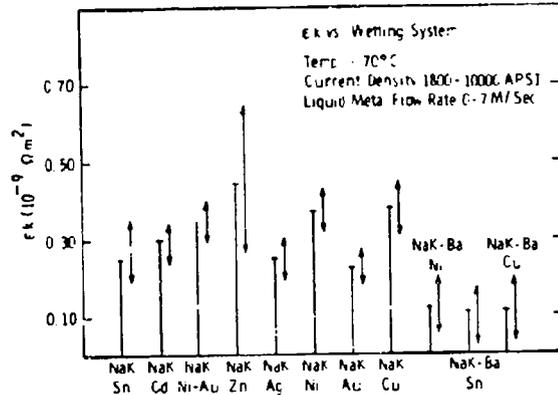


Figure 6

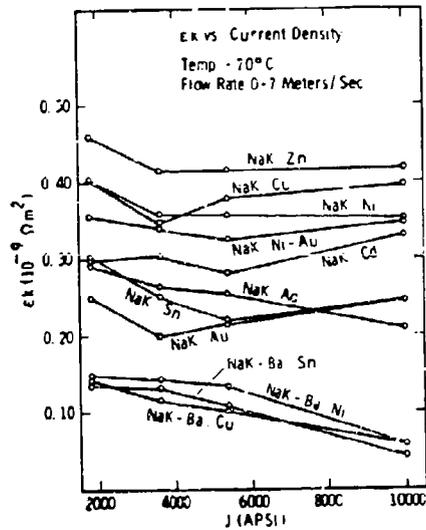


Figure 7

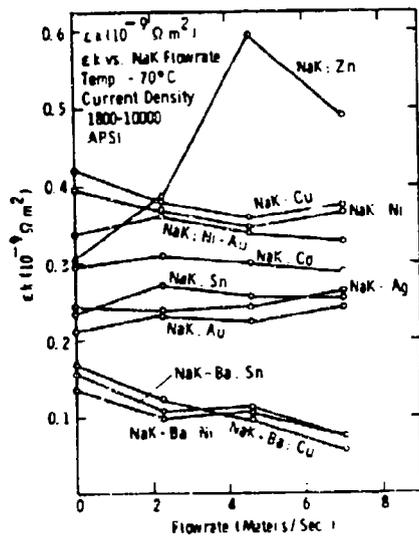


Figure 8

Curve 728142-B

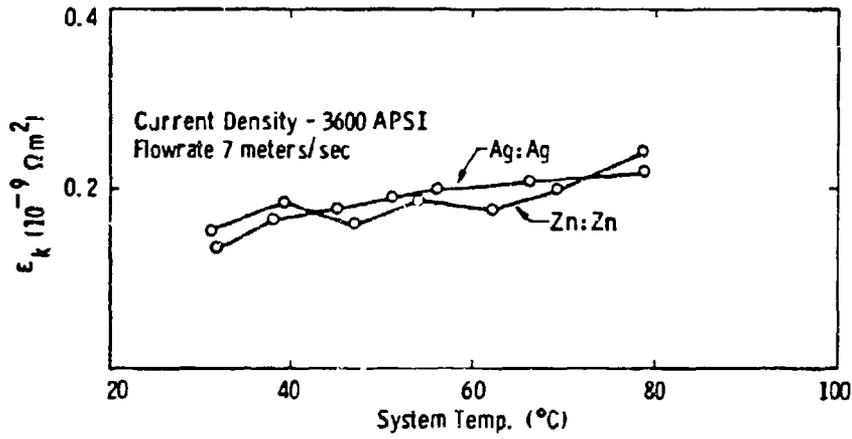


Fig. 9 - ϵ_k vs. liquid metal temperature

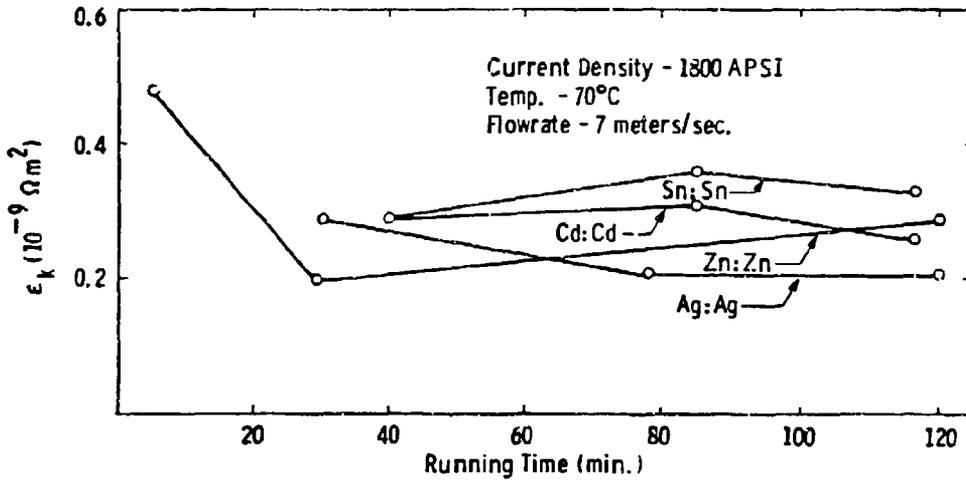


Fig. 10 - ϵ_k vs. running time

Dwg. 6.399A73

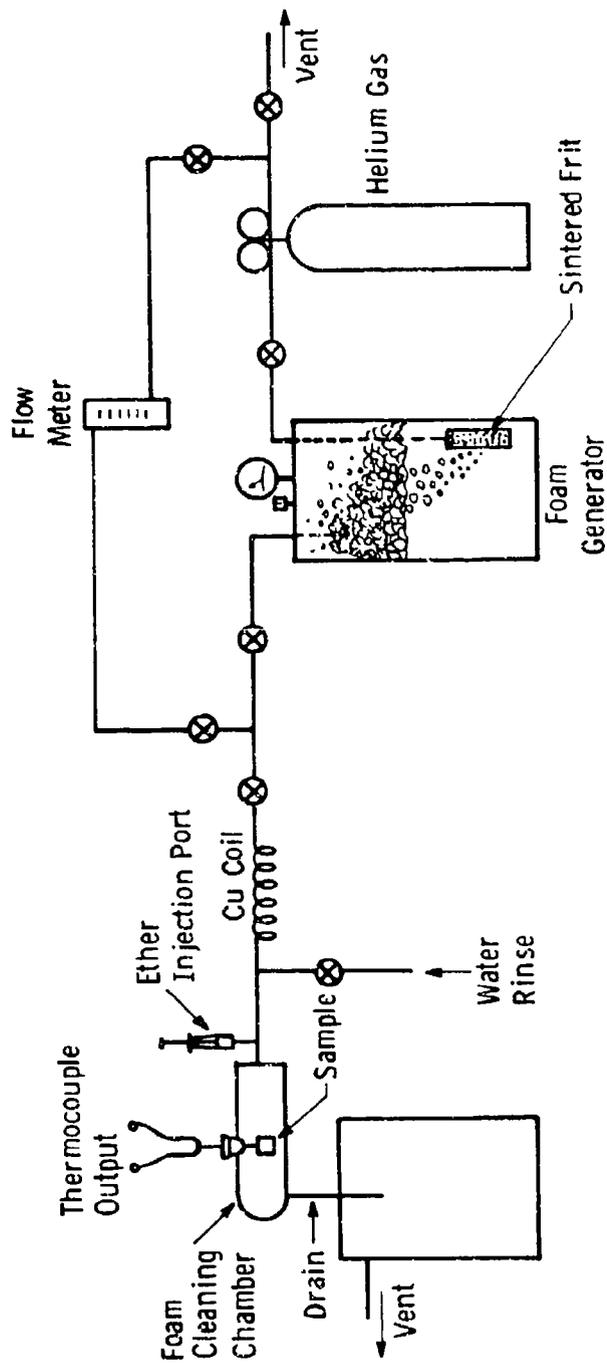


Fig. 11- Foam generator and associated apparatus for studying the cleaning of NaK from materials using a 5% sodium lauryl sulfate aqueous foam

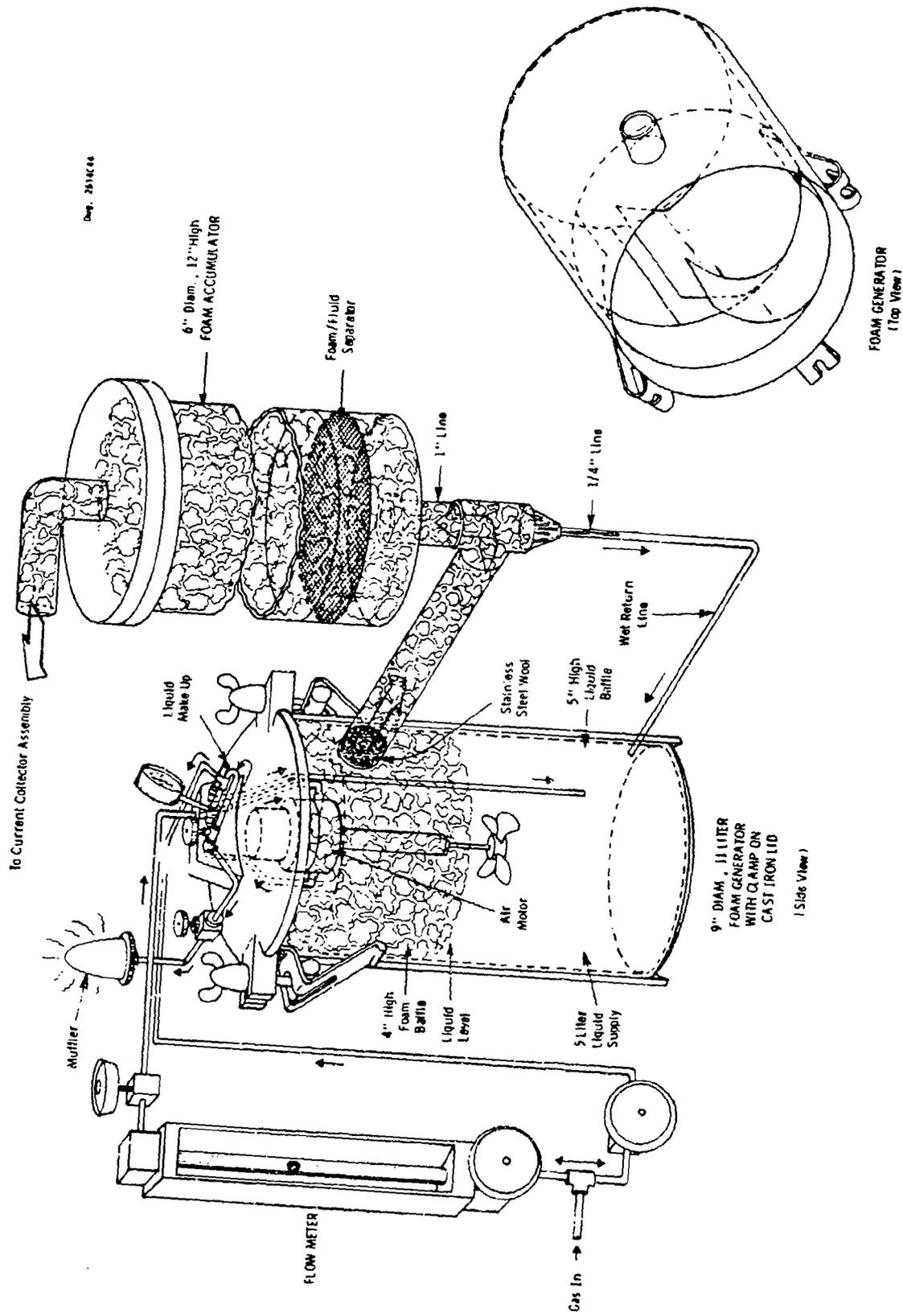


Fig. 12—foam generator for use in cleaning NAK from the internals of machines which utilize liquid metal current collectors

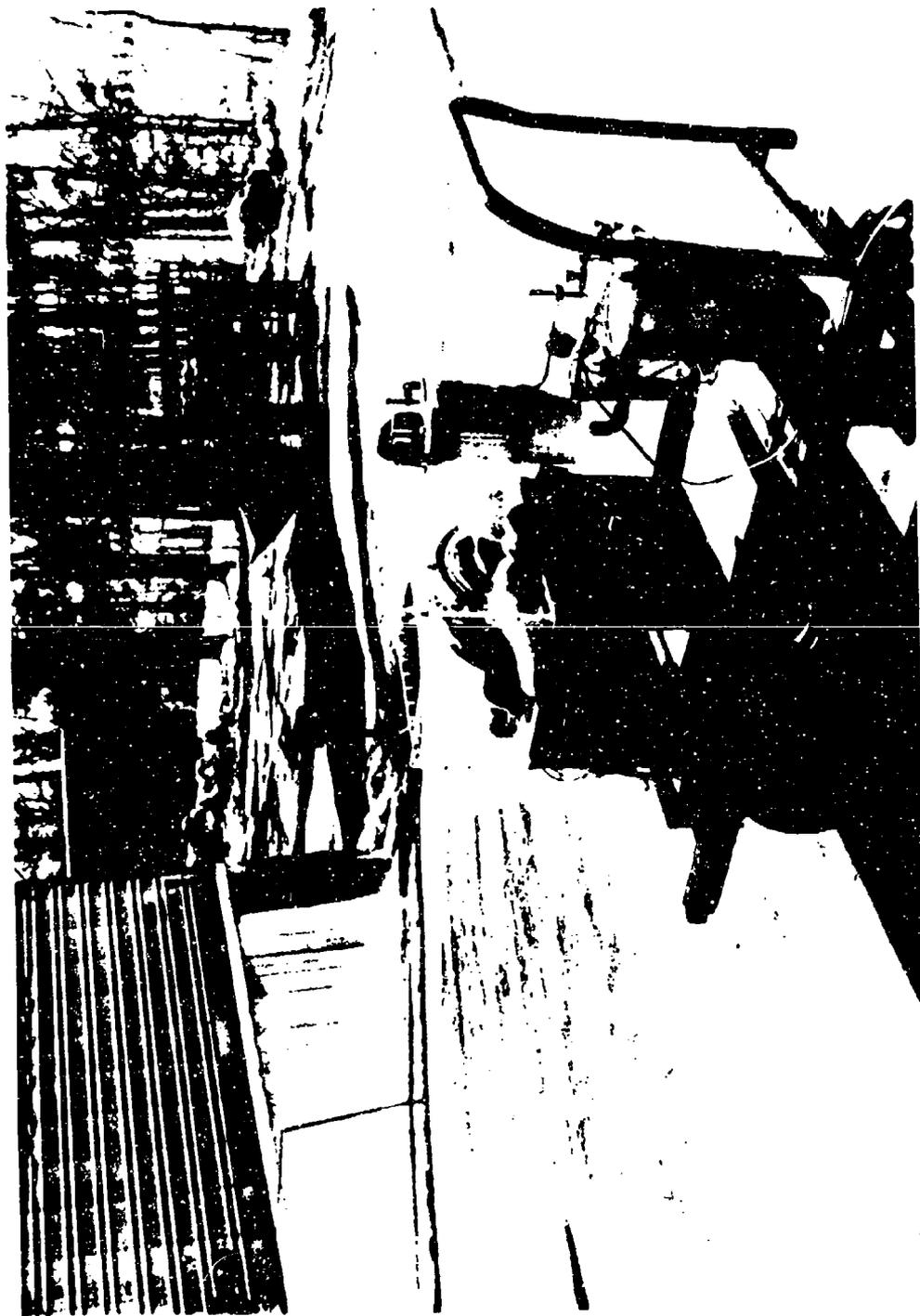


Figure 13. Overall view of four cleaning operation. This photo was taken near the end of the cleaning operation.



Figure 14. Close-up view of NaK contaminated current collector assembly. Note presence of NaK reaction products.

Dwg. 7701A21

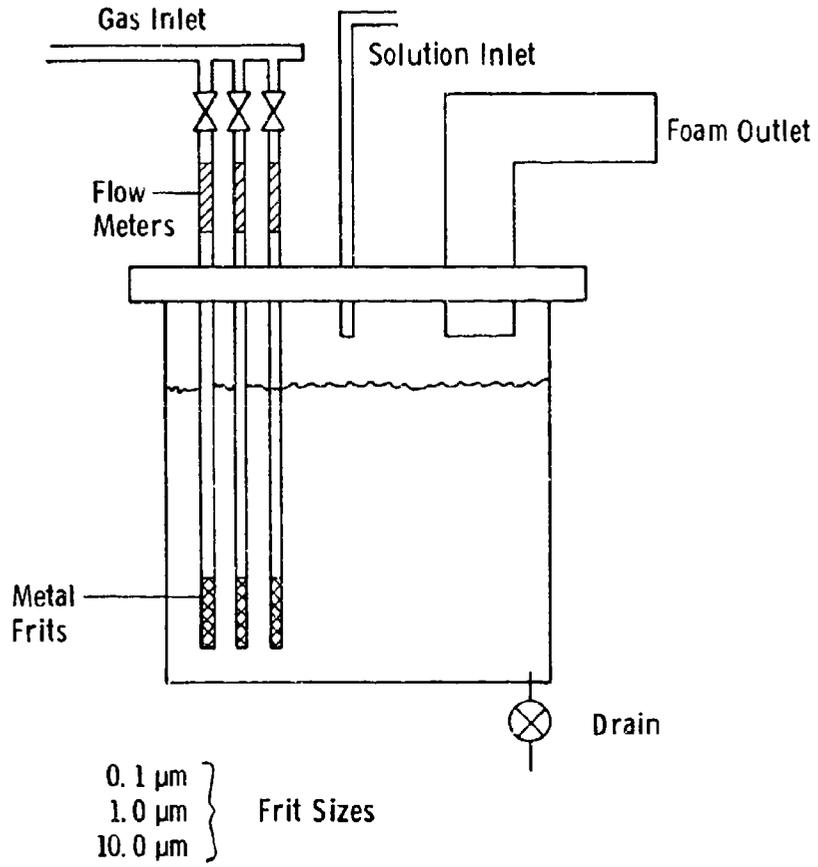


Fig. 15- Foam generator

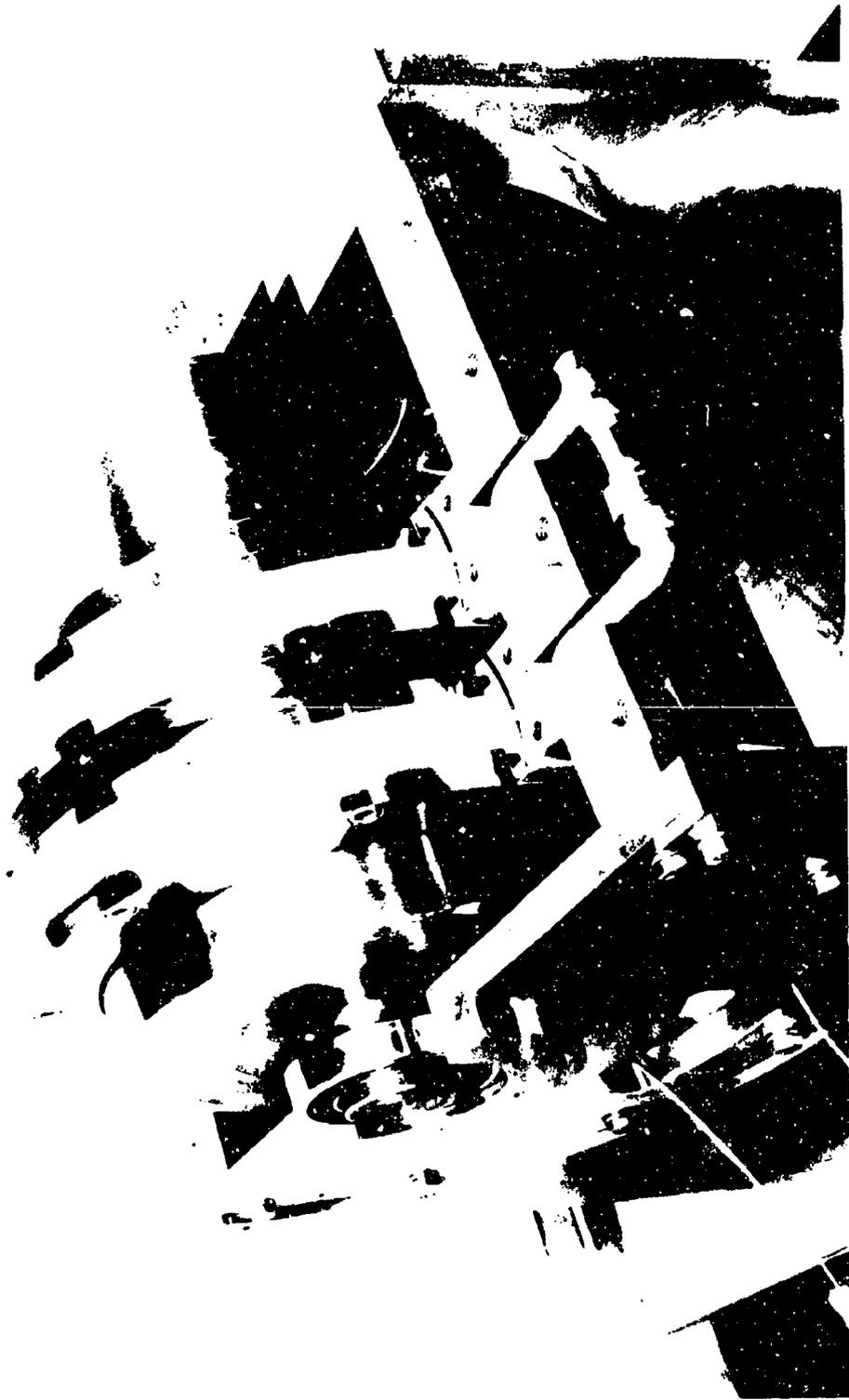


Figure 16. In-house current collector prototype: rotors exposed.

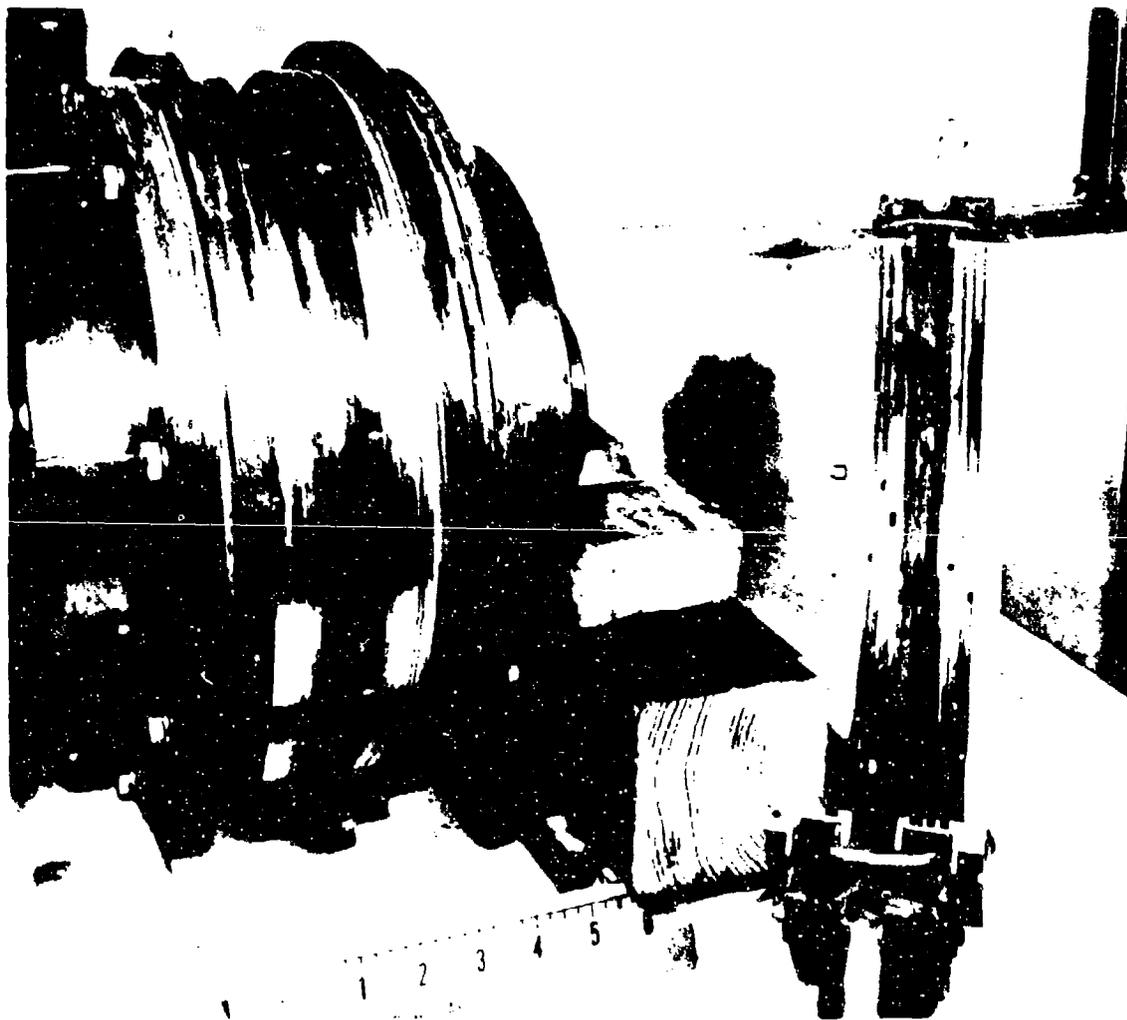


Figure 17. In-house collector prototype: rotor pair and stator hall.

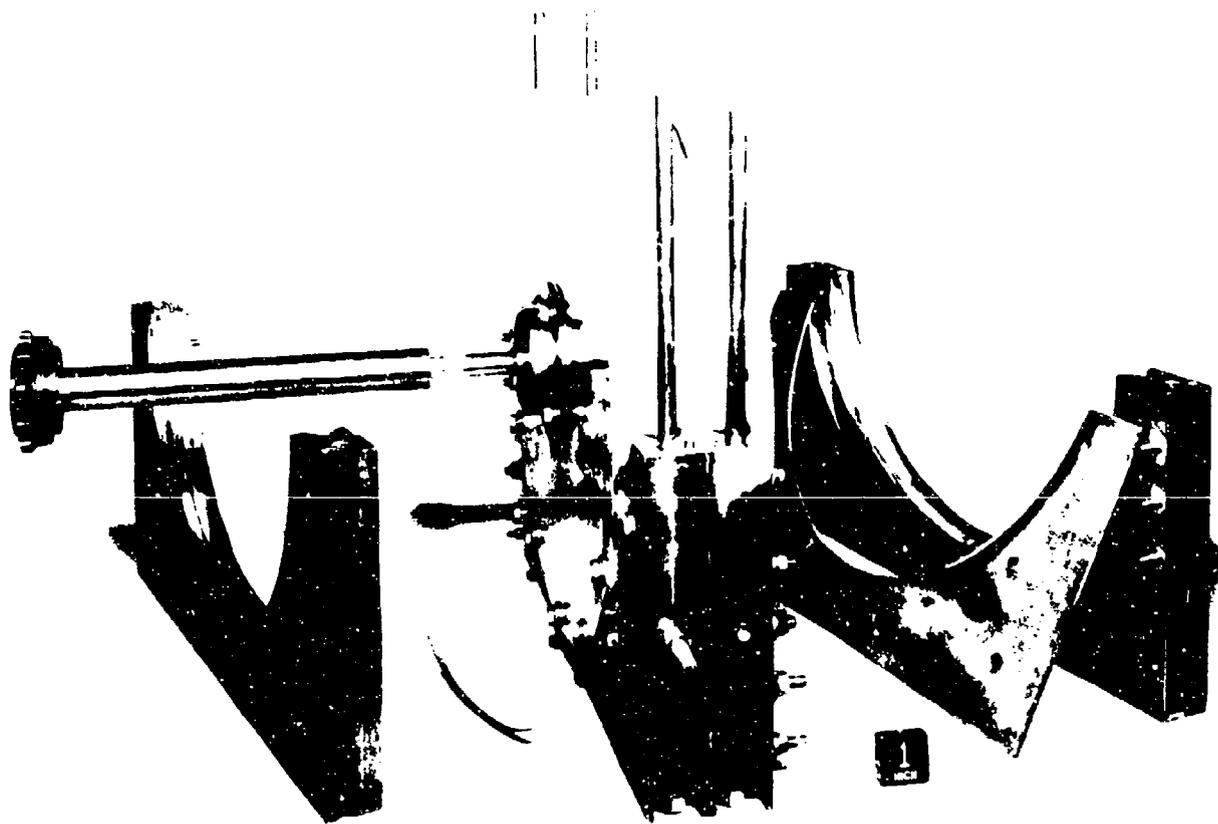
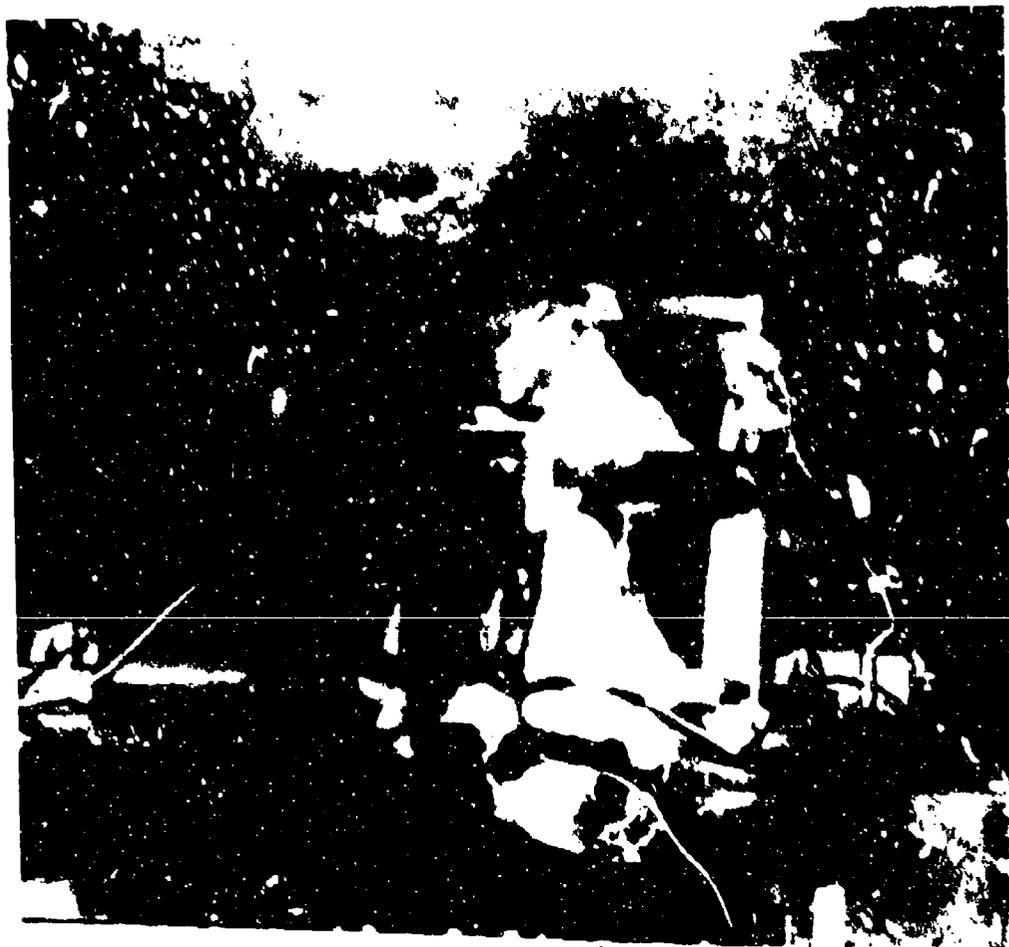


Figure 18. Navy collector prototype: disassembled.



COLLECTOR TEST STAND

Figure 19. Foam exiting collector labyrinth seals.



COLLECTOR TEST STAND

Figure 30. collector unit saturated with foam.



WATER COLLECTOR TEST STAND

Figure 21. Collapsing of foam with water.

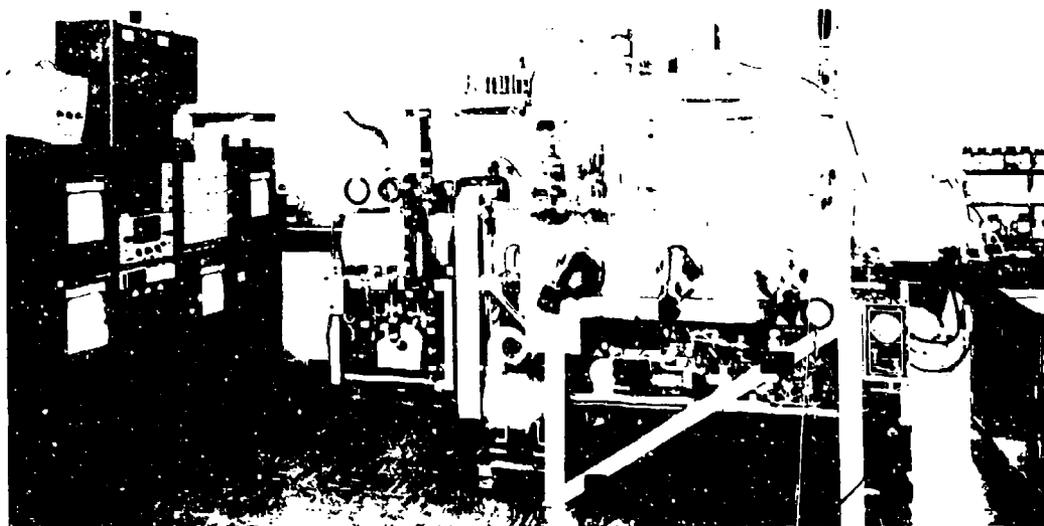


Figure 22. Current collector test stand with control panel and auxiliary systems.

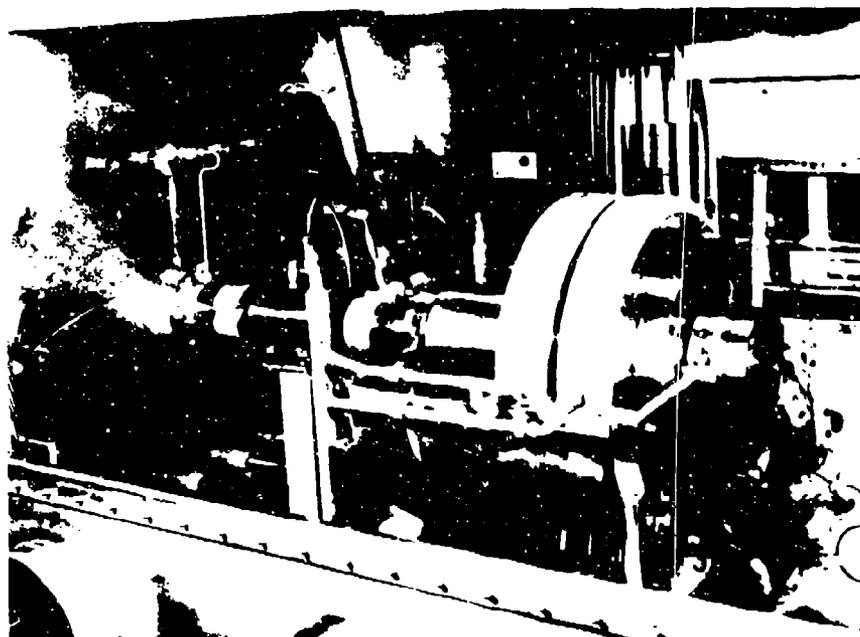


Figure 23. Test rig for an in-house liquid metal current collector housed in glove box and coupled to drive train.

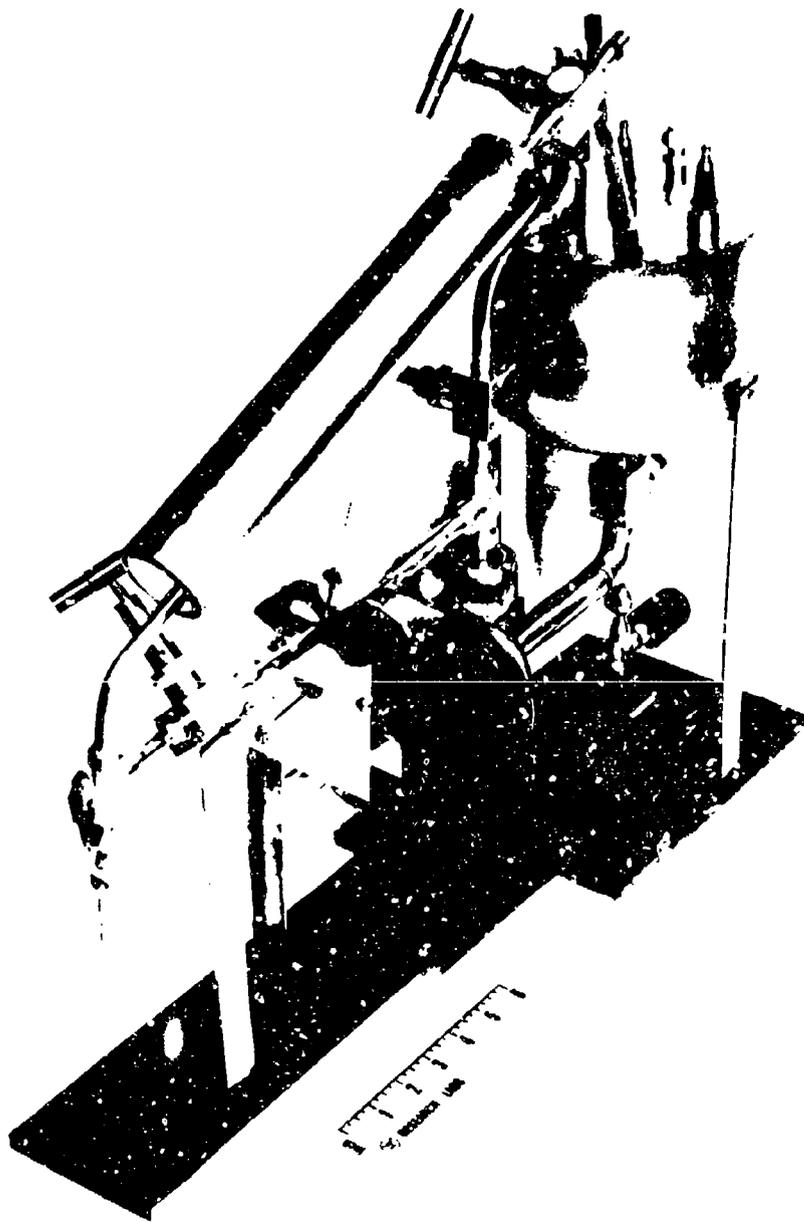


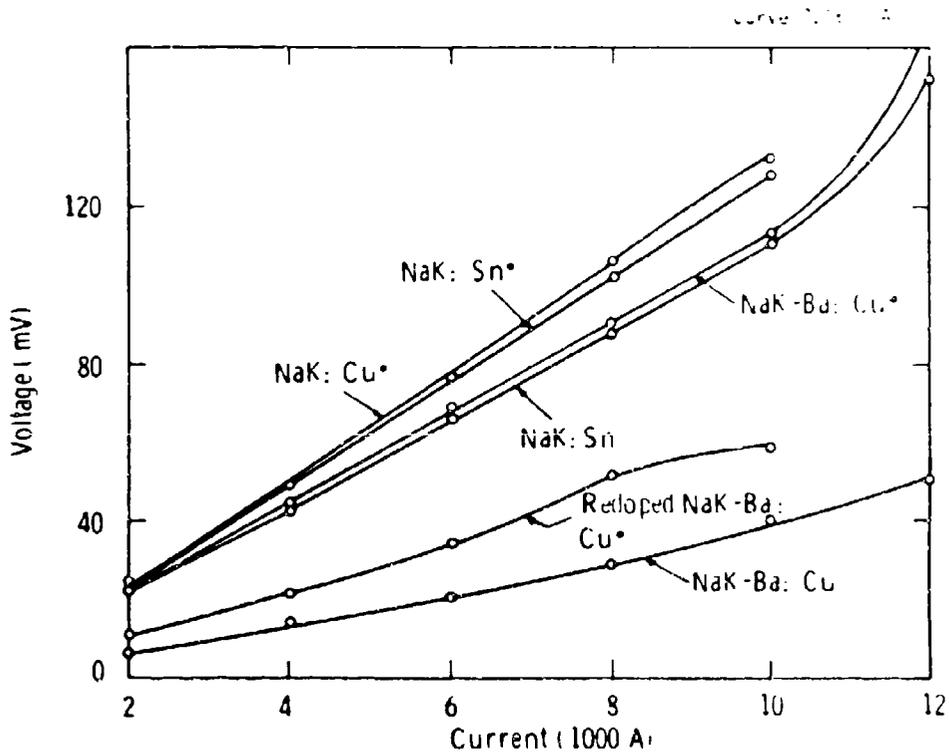
Figure 24. Liquid metal supply loop.



Figure 25. High current bus network.



Figure 26. Upper and lower stator half conductors.



*Post-Cleaning Electrical Test

Fig. 27—In-house current collector prototype performance curves: before and after foam cleaning

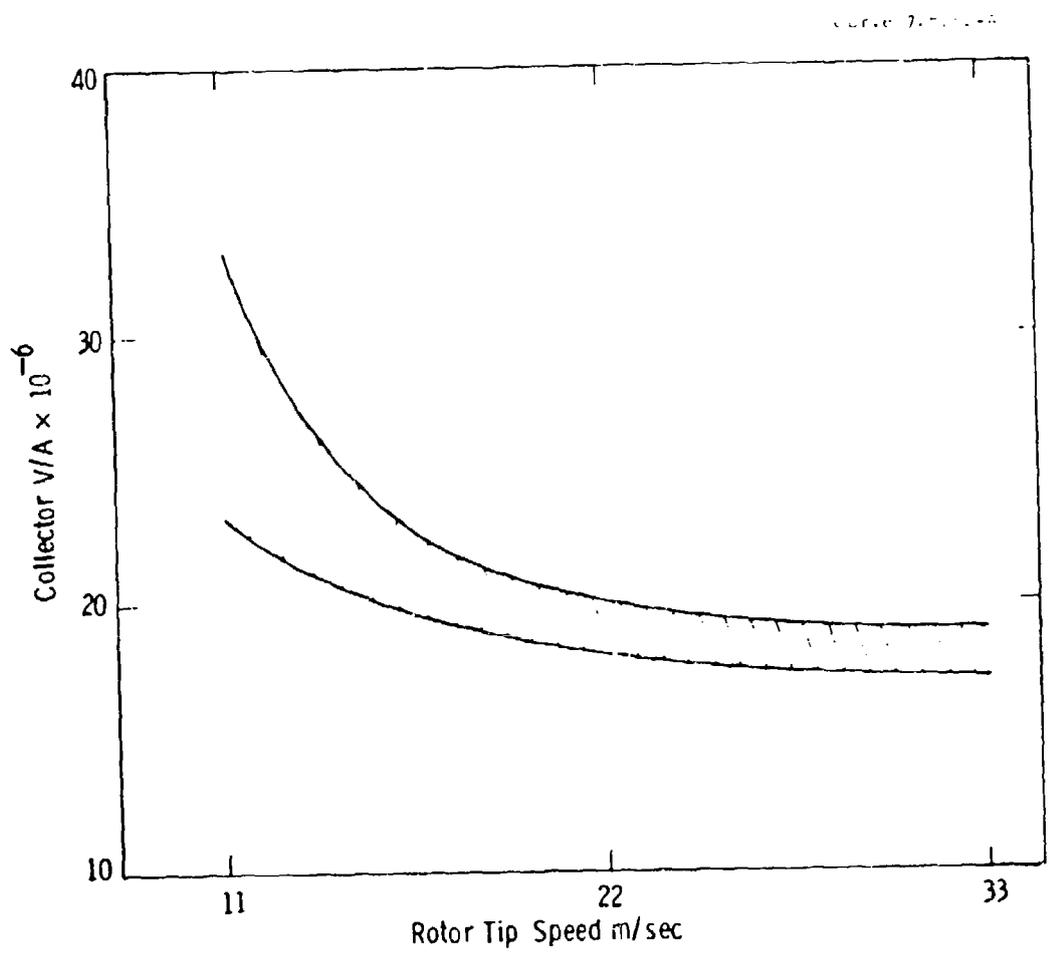


Fig. 28—Electrical performance as a function of rotor tip speed: In-house collector prototype

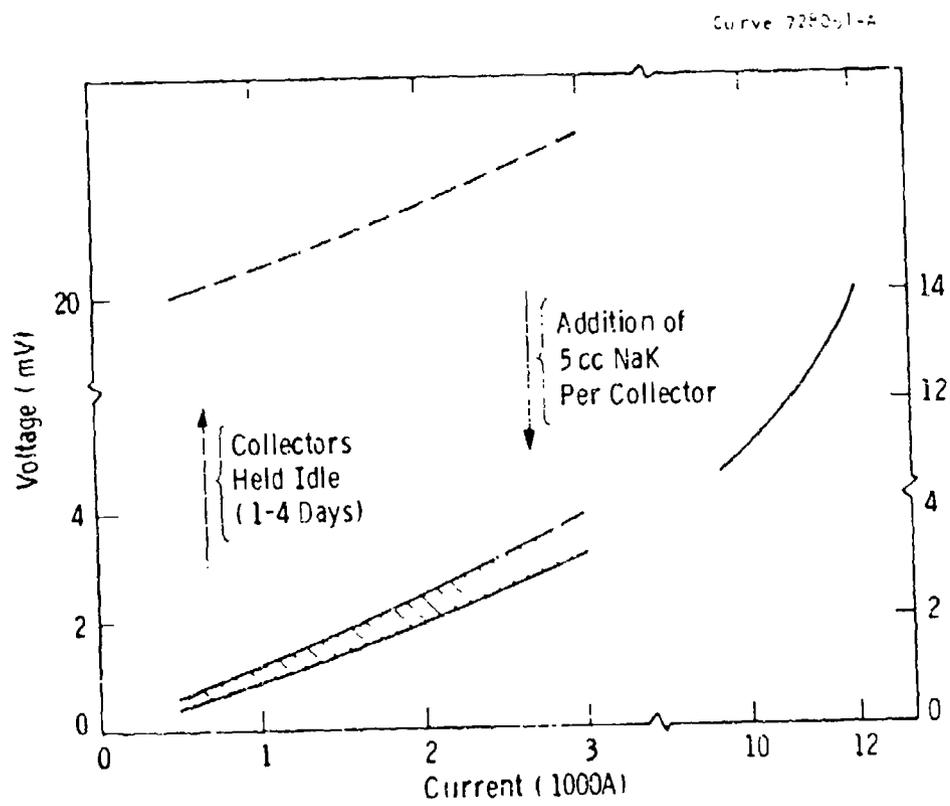


Fig. 29—Navy current collector prototype performance curves

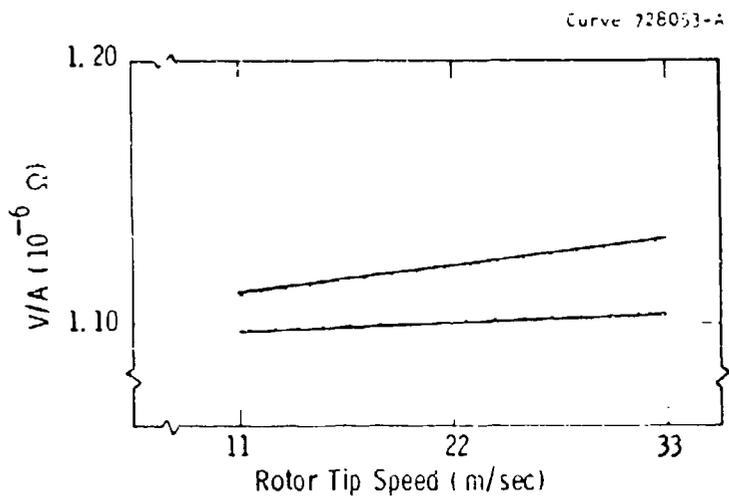


Fig. 30—Electrical performance vs rotor tip speed:
navy collector prototype

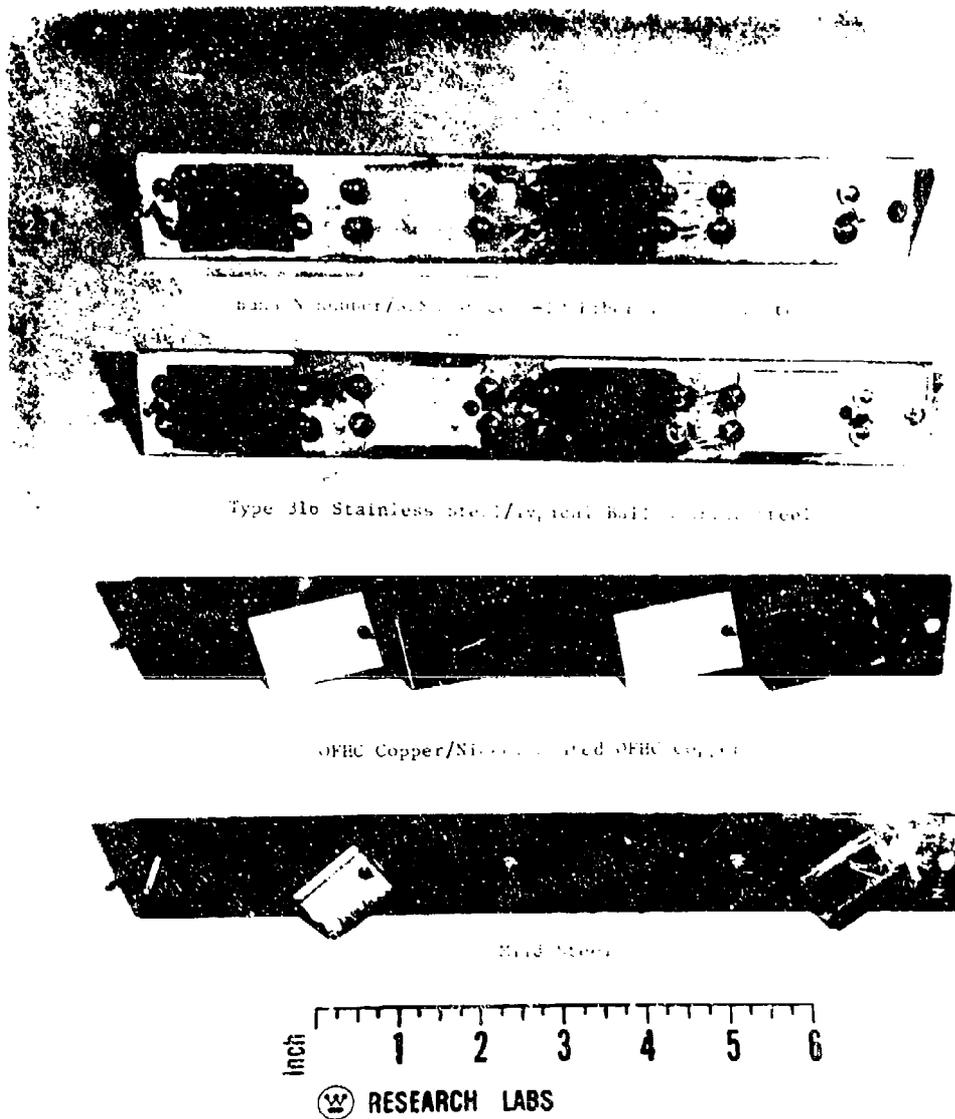


Figure 31. Machine materials mounted on specimen trees.

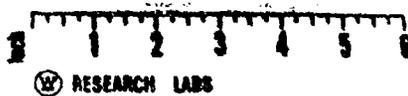
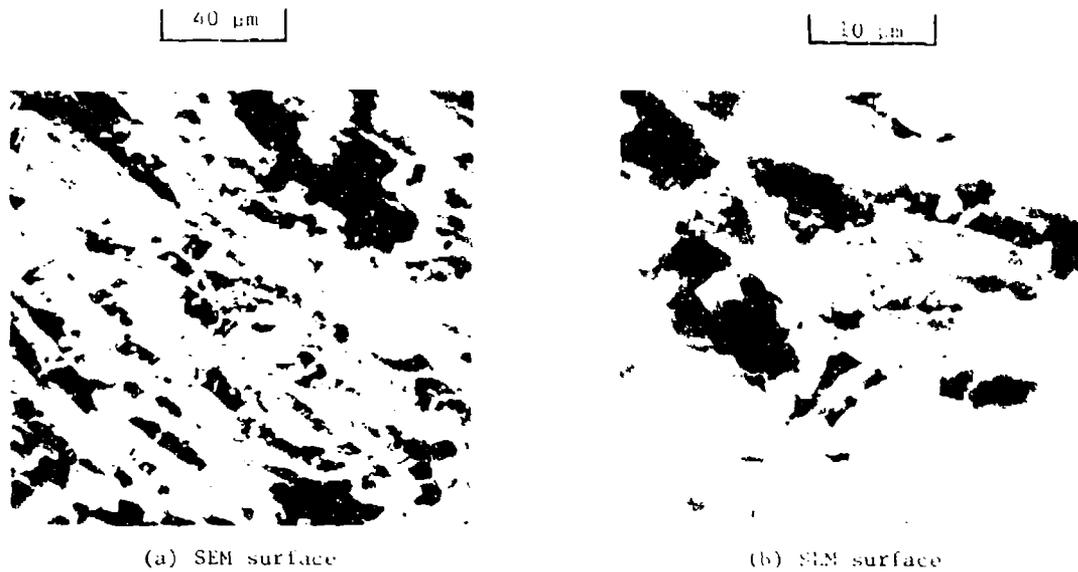
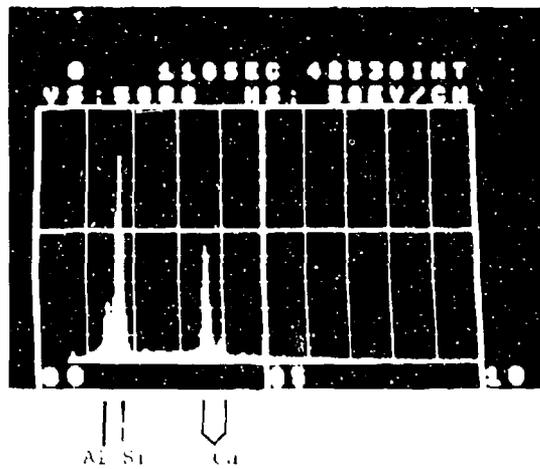


Figure 32. Example of the stainless steel exposure capsules and specimen trees.



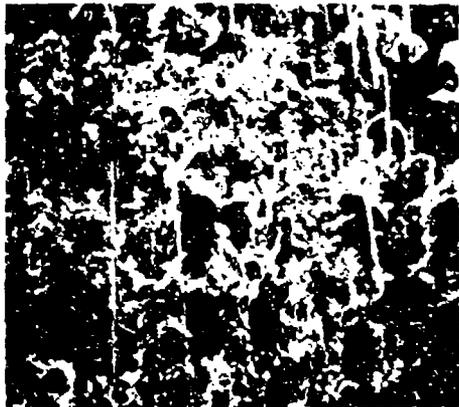
as-received



(c) Electron excited x-ray spectrum.
(100X rastered area)

Figure 33. Nema Grade G-10 Fiberglass Laminator. Surface features and composition. (100X rastered area). (48A) (exposed)

40 μm



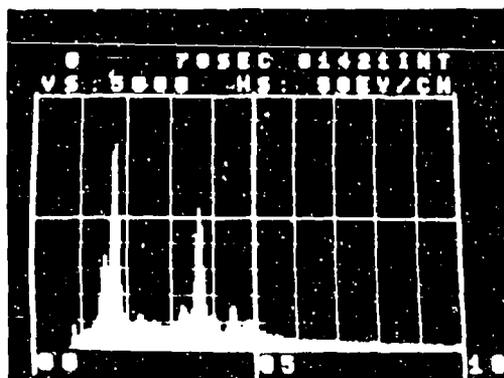
(a) SEM surface

10 μm



(b) SEM surface

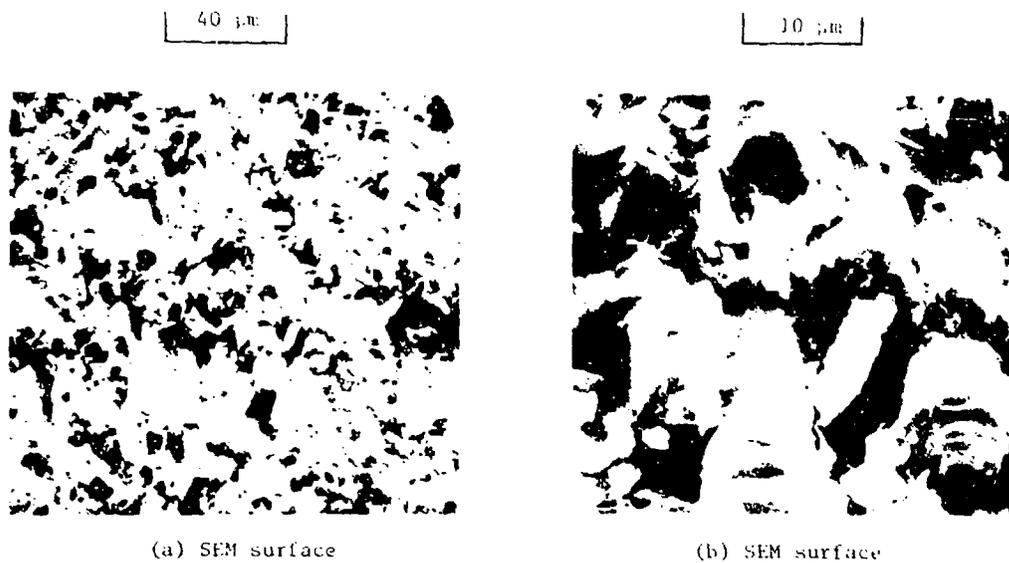
500 hrs, NaK_{78} , 100°C



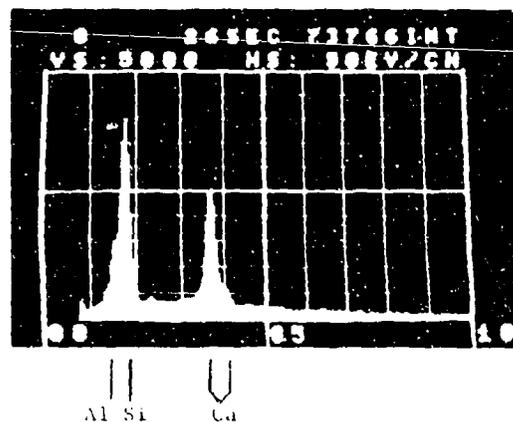
Al | S | Ca | Ba
Si | K

(b) Electron excited x-ray spectrum
(100X rastered area)

Figure 34. Nova Grade C-10 Fiberglass Laminate: Surface features and composition after NaK_{78} (4%) exposure.

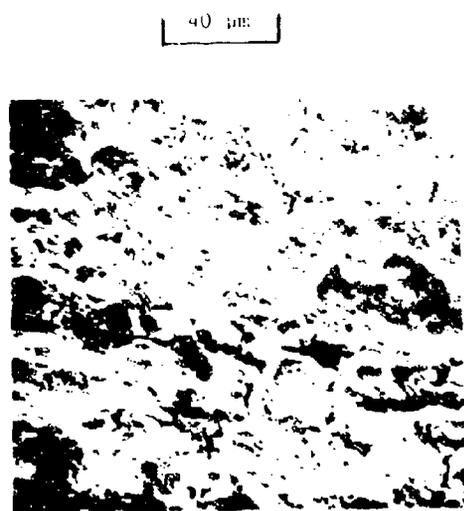


500 hrs., 100°C

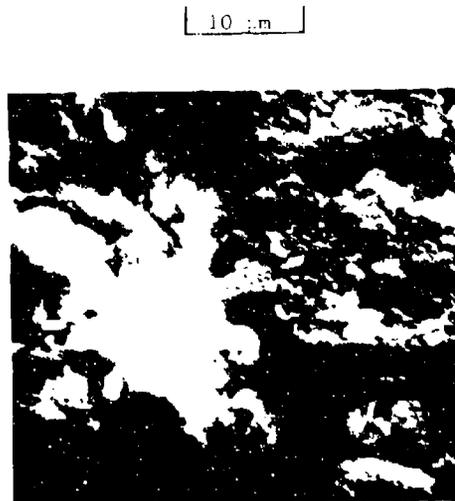


(c) Electron excited x-ray spectrum
(100X rastered area)

Figure 35. Nema Grade G-10 Fiberglass Laminator: Surface features and composition after thermal exposure.

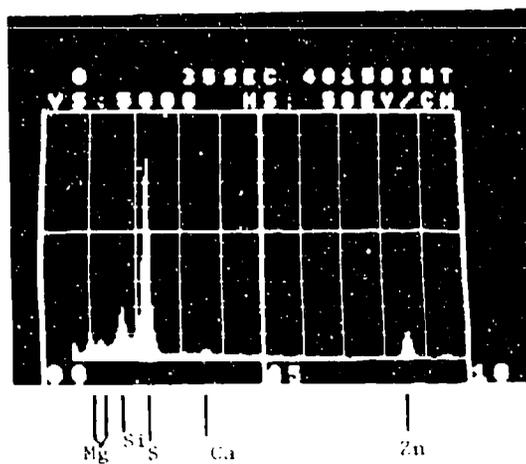


(a) SEM surface



(b) SEM surface

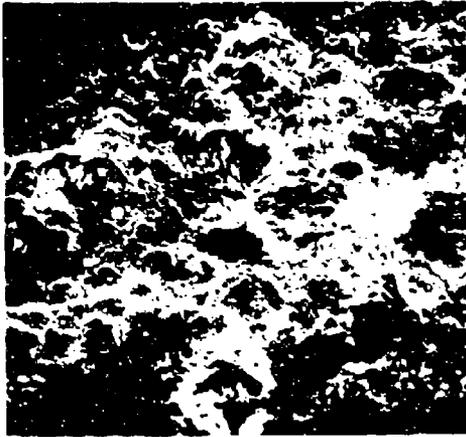
as-received



(c) Electron excited x-ray spectrum
(100X rastered area)

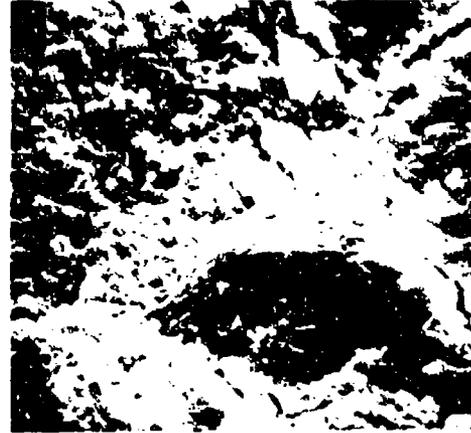
Figure 36. Buna N Rubber: Surface features and composition before NaK₇₈ (EB) exposure.

40 μ m



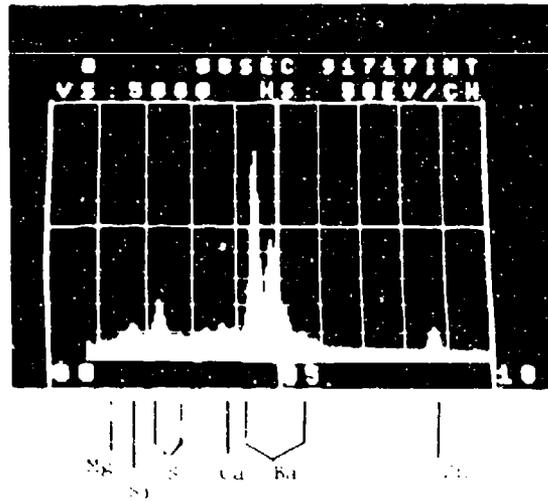
(a) SEM surface

20 μ m



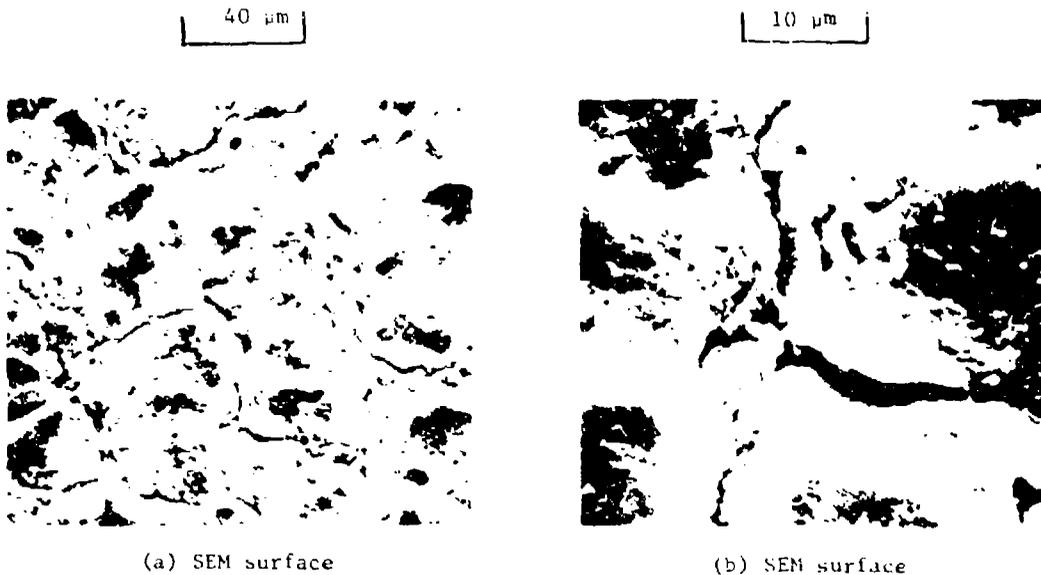
(b) SEM surface

500 hrs., NaK₇₈, 100°C

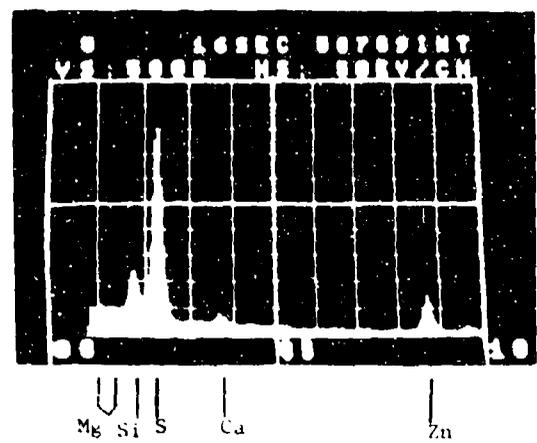


(c) Electron excited x-ray spectra
(only listed area)

Figure 37. Barium Rubidium surface composition of $\text{Ba}_{0.5}\text{Rb}_{0.5}\text{Ti}_2\text{O}_{10}$ after 500 hrs. in NaK_{78} at 100°C. (SEM surface)

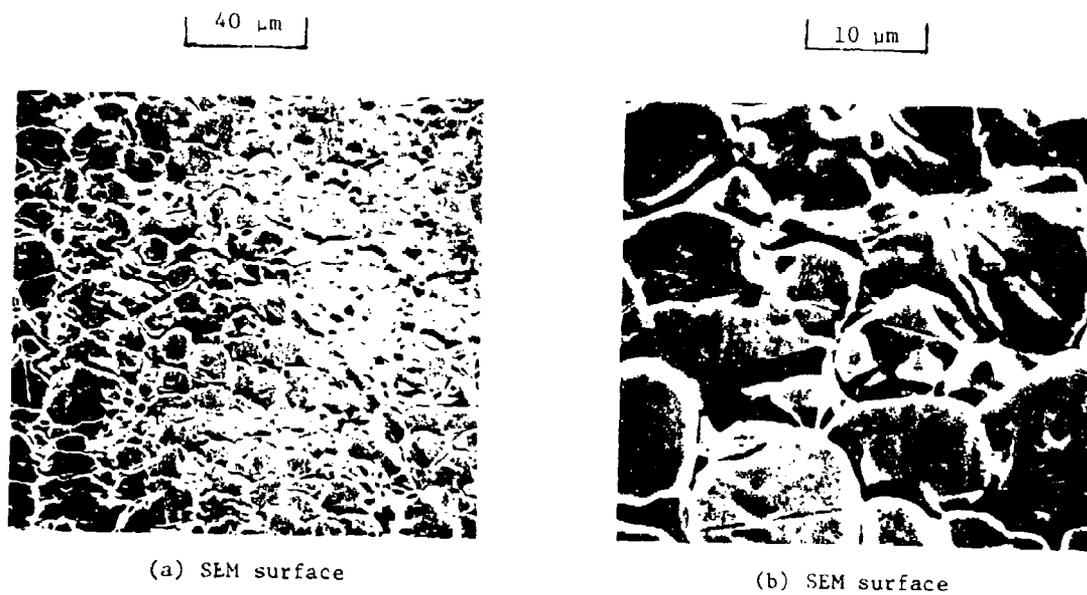


500 hrs., 100°C

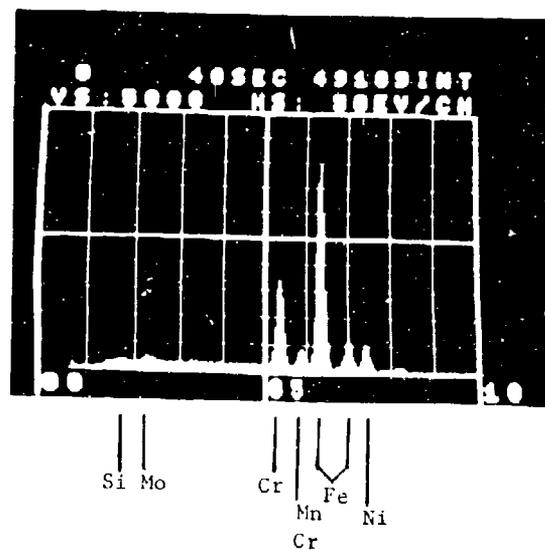


(c) Electron excited x-ray spectrum
(100X rastered area)

Figure 38. Buna X Rubber: Surface features and composition after thermal exposure.



as-received



(c) Electron excited x-ray spectrum
(100X rastered area)

Figure 39. 316 Stainless Steel: Surface features and composition before
NaK₇₈ (+Ba) exposure.

40 μm

10 μm

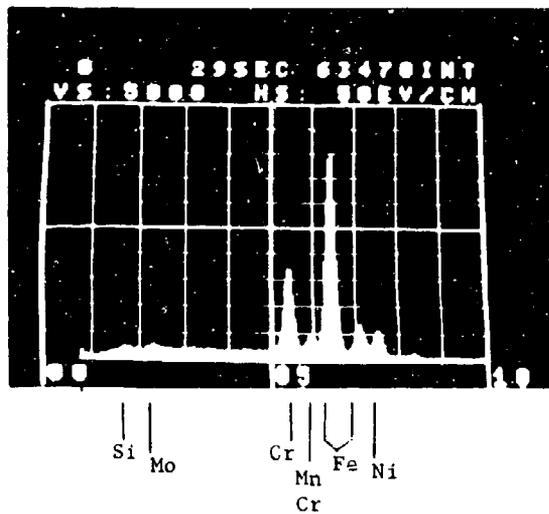


(a) SEM surface



(b) SEM surface

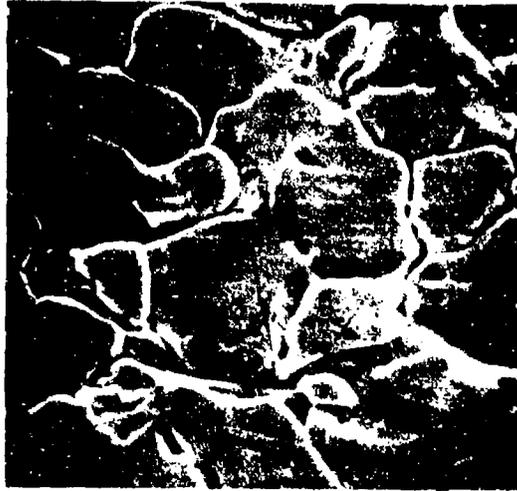
500 hrs., NaK₇₈, 100°C



(c) Electron excited x-ray spectrum
(100X rastered area)

Figure 40. 316 Stainless Steel: Surface features and composition after NaK₇₈ (+Ba) exposure.

10 μ m



(a) SEM surface

500 hrs., 100°C

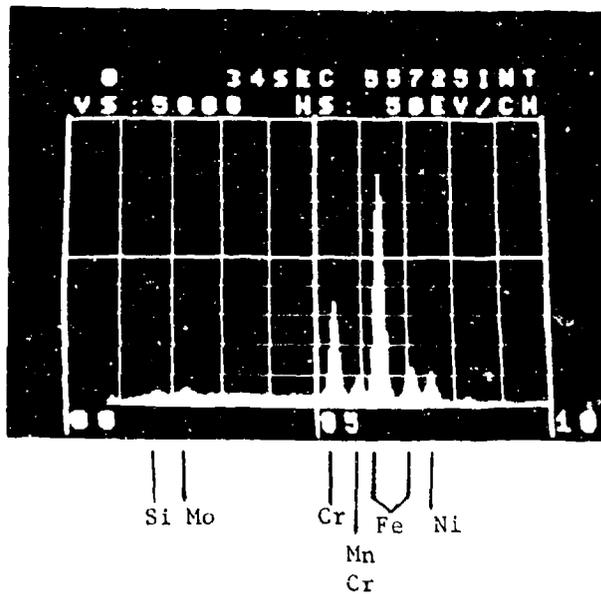


Figure 41. 316 Stainless Steel: Surface features and composition after thermal exposure.

40 μm



(a) SEM surface

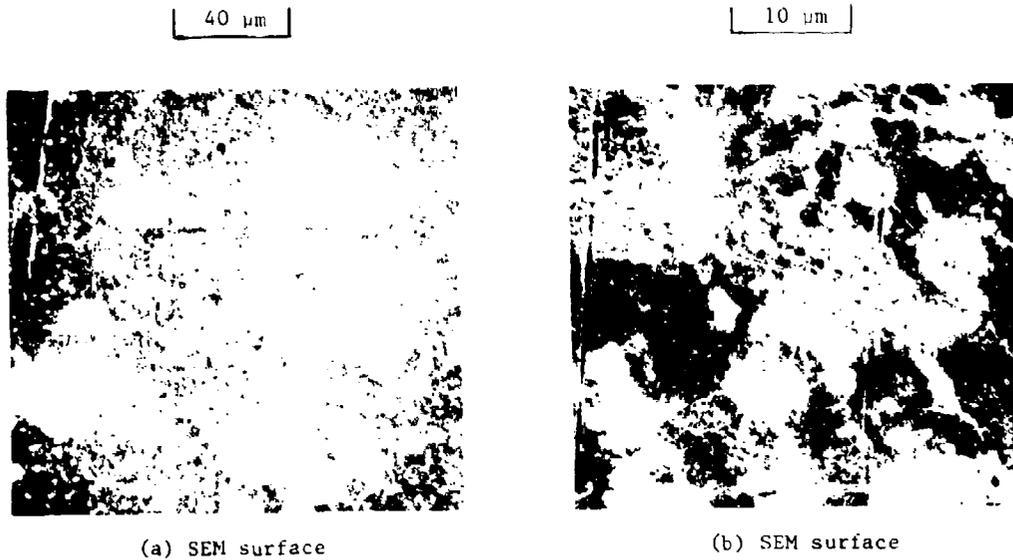
10 μm



(b) SEM surface

as-received

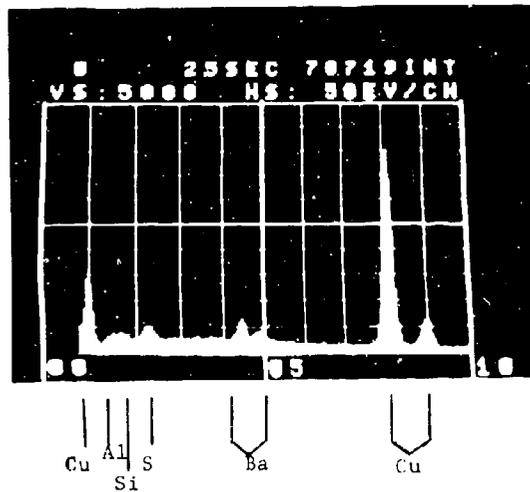
Figure 42. OFHC Copper: Surface features before NaK_{78} (+Ba) exposure.



(a) SEM surface

(b) SEM surface

500 hrs., NaK₇₈, 100°C



(c) Electron excited x-ray spectrum
(200X rastered area)

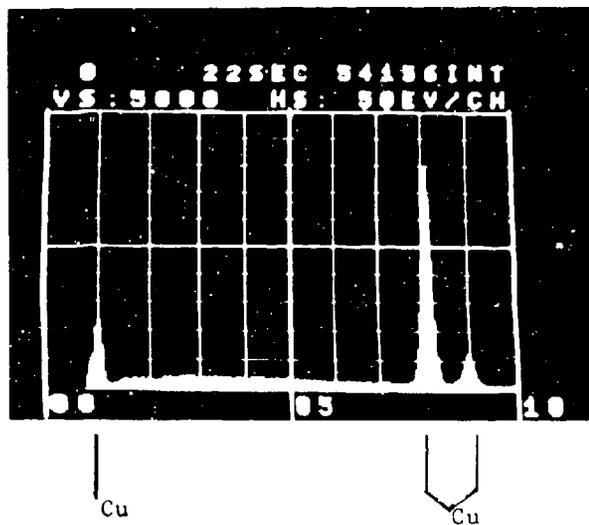
Figure 43. OFHC Copper: Surface features and composition after NaK₇₈ (+Ba) exposure.

40 μ m



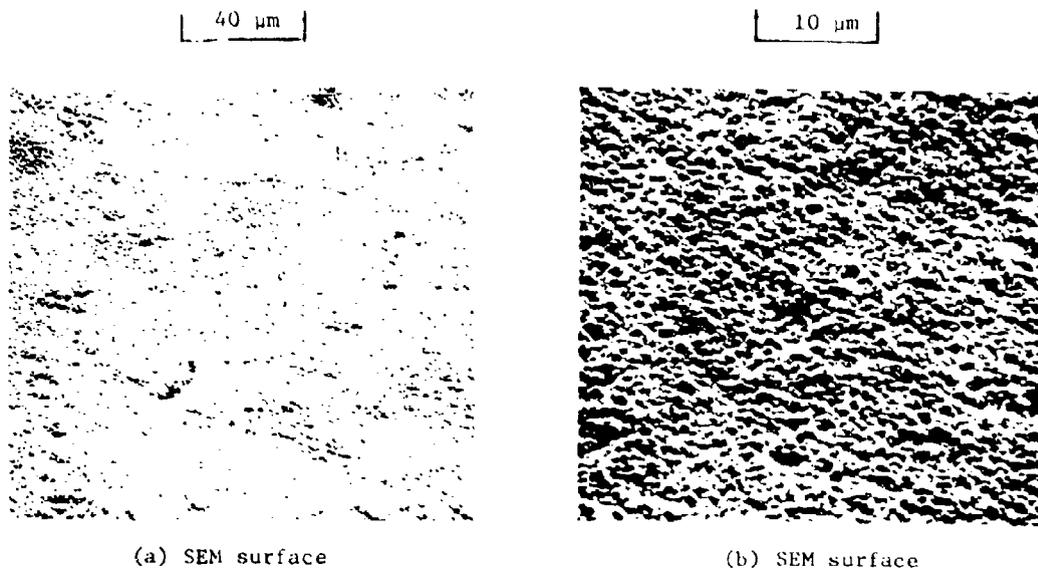
(a) SEM surface

500 hrs., 100°C

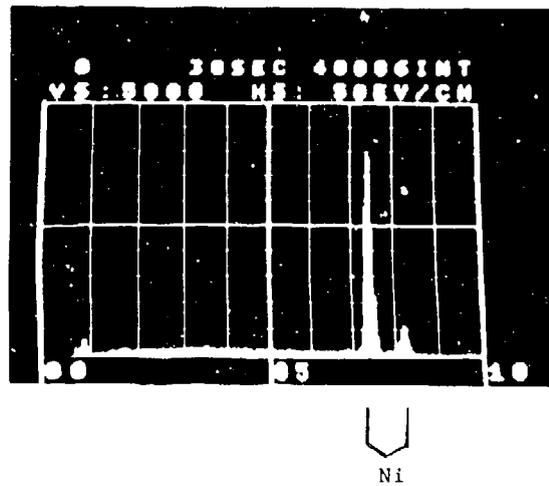


(b) Electron excited x-ray spectrum
(2000X rastered area)

Figure 44. OFHC Copper: Surface features and composition after thermal exposure.

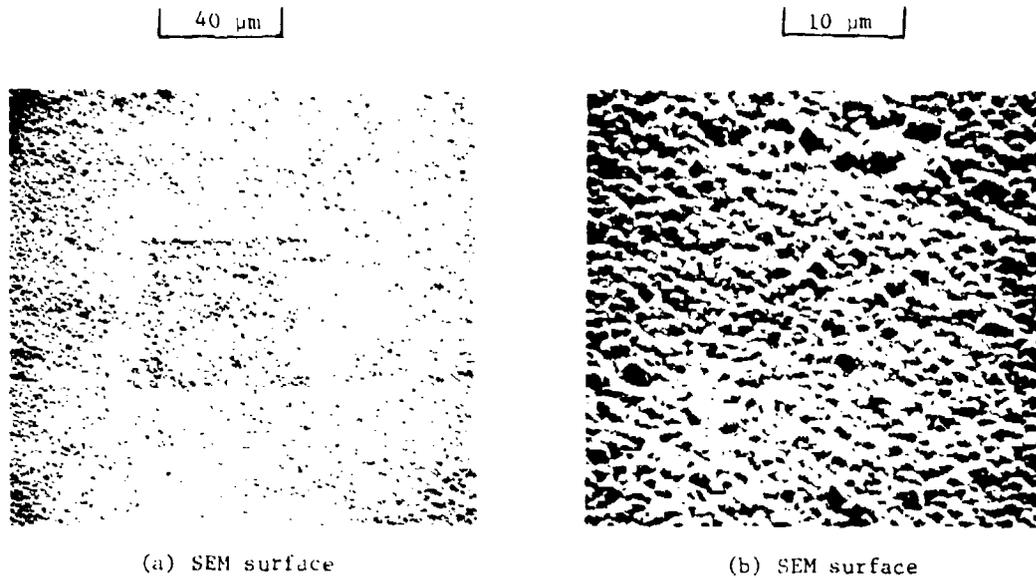


as-received



(c) Electron excited x-ray spectrum
(100X rastered area)

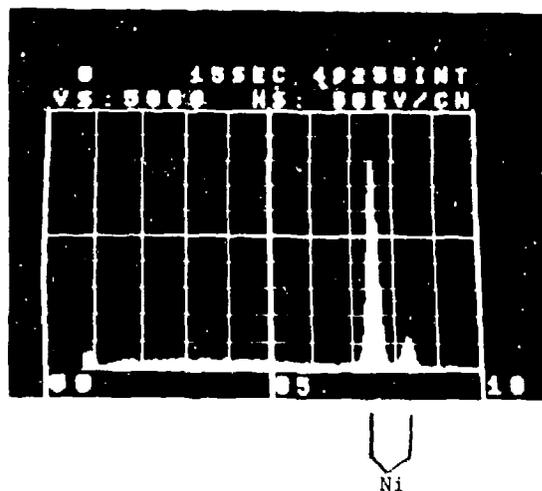
Figure 45. Nickel plated OFHC Copper: Surface features and composition before NaK_{78} (+Ba) exposure.



(a) SEM surface

(b) SEM surface

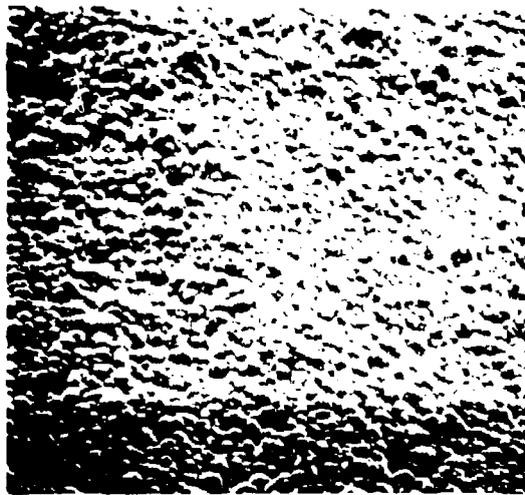
500 hrs., NaK₇₈, 100°C



(c) Electron excited x-ray spectrum
(100X rastered area)

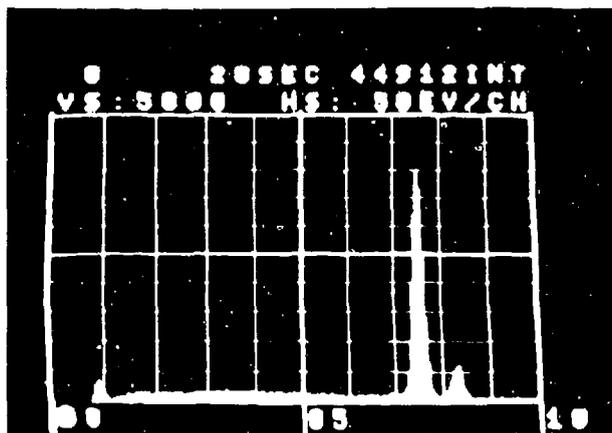
Figure 46. Nickel plated OFHC Copper; Surface features and composition after NaK₇₈ (+Ba) exposure.

40 μ m



(a) SEM surface

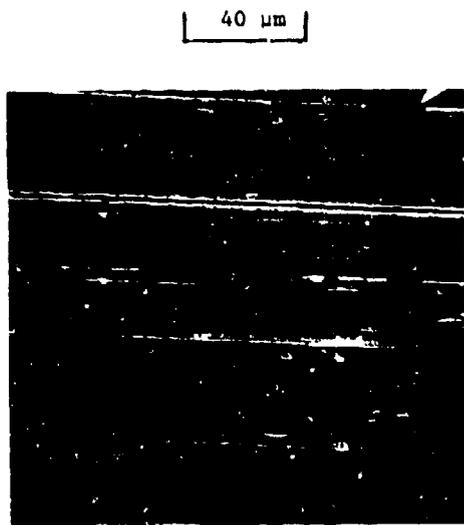
500 hrs., 100°C



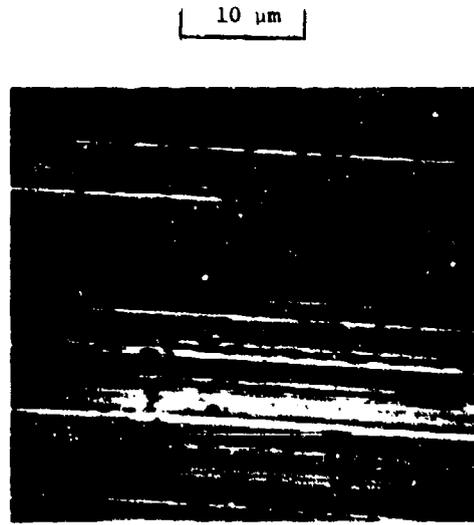
Ni

(b) Electron excited x-ray spectrum
(100X rastered area)

Figure 47. Nickel plated OFHC Copper; Surface features and composition after thermal exposure.



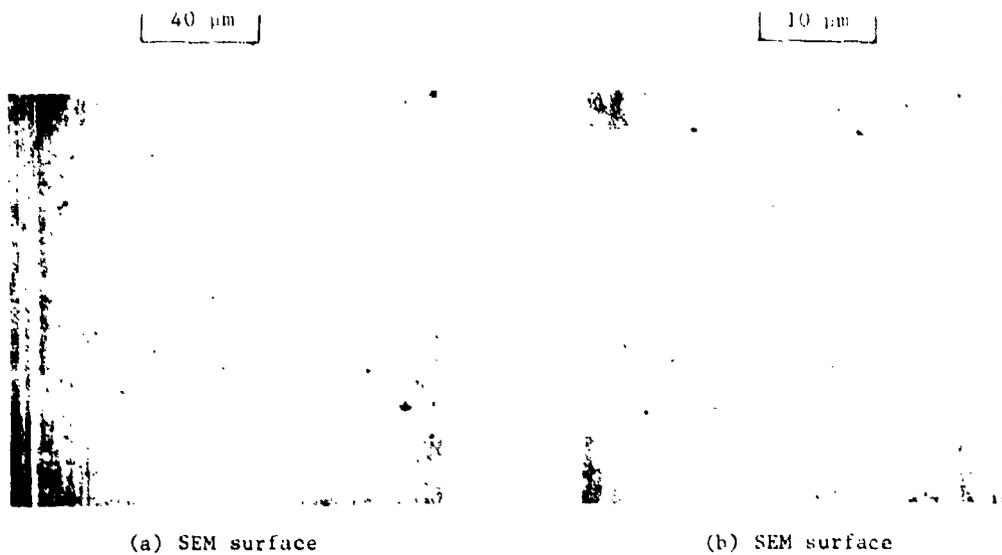
(a) SEM surface



(b) SEM surface

as-received

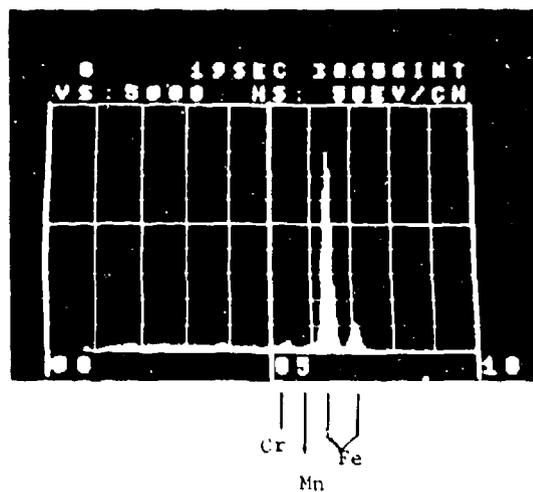
Figure 48. Ball Bearing Steel: Surface features before NaK_{78} (+Ba) exposure.



(a) SEM surface

(b) SEM surface

500 hrs., NaK₇₈, 100°C



(c) Electron excited x-ray spectrum
(100X rastered area)

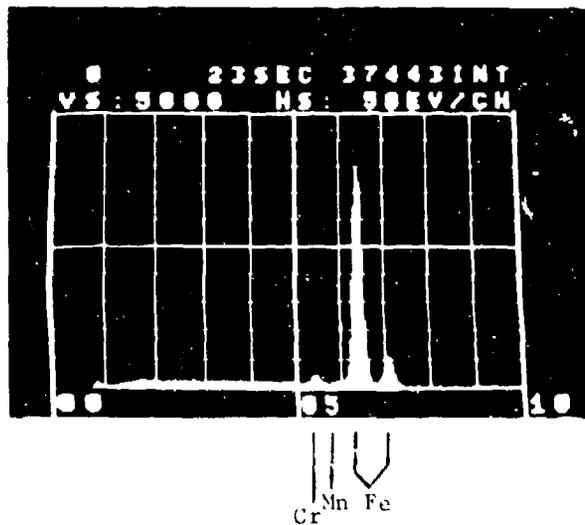
Figure 49. Ball Bearing Steel: Surface features and composition after NaK₇₈ (+Ba) exposure.

40 μ m



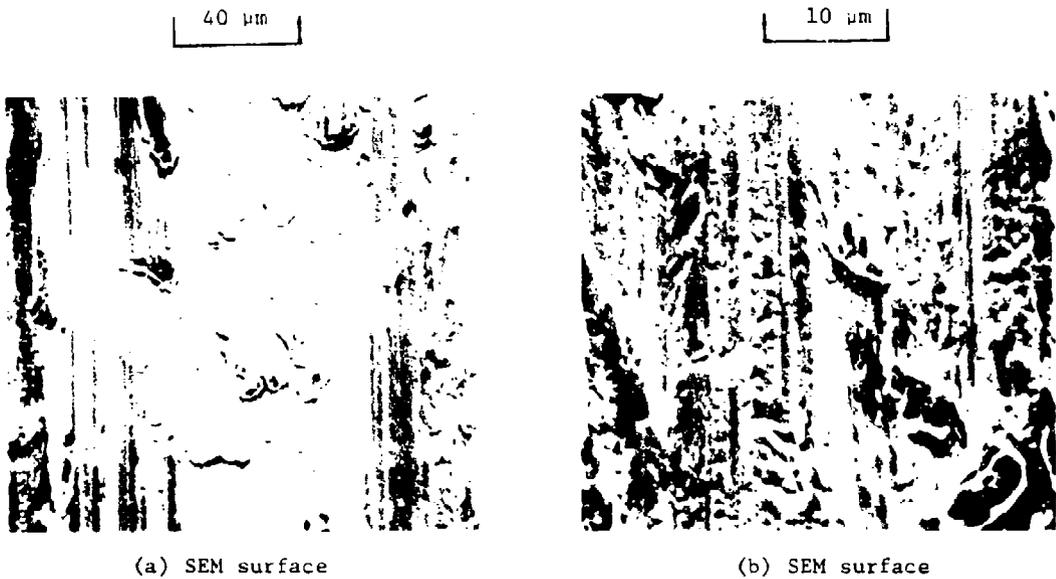
(a) SEM surface

500 hrs., 100°C



(b) Electron excited x-ray spectrum
(100X rastered area)

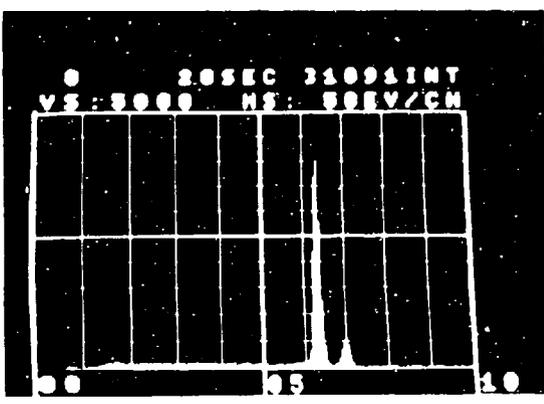
Figure 50. Ball Bearing Steel: Surface features and composition after thermal exposure.



(a) SEM surface

(b) SEM surface

as-received



Fe

(c) electron excited x-ray spectrum
(100X rastered area)

Figure 51. Mild Magnetic Steel: Surface features and composition before NaK₇₈ (+Ba) exposure.

40 μm



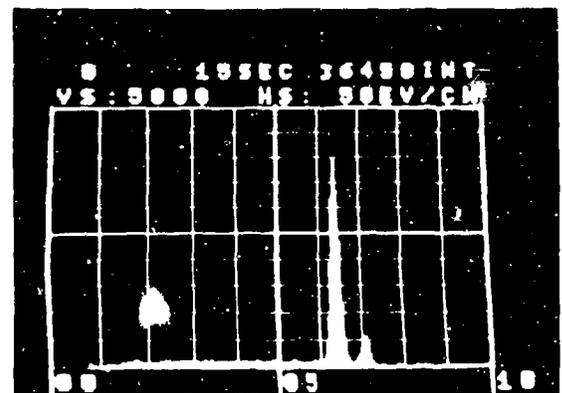
(a) SEM surface

10 μm



(b) SEM surface

500 hrs., NaK₇₈, 100°C

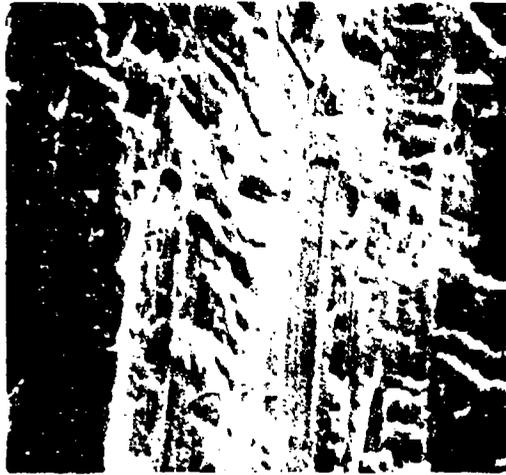


Fe

(c) Electron excited x-ray spectrum
(100X rastered area)

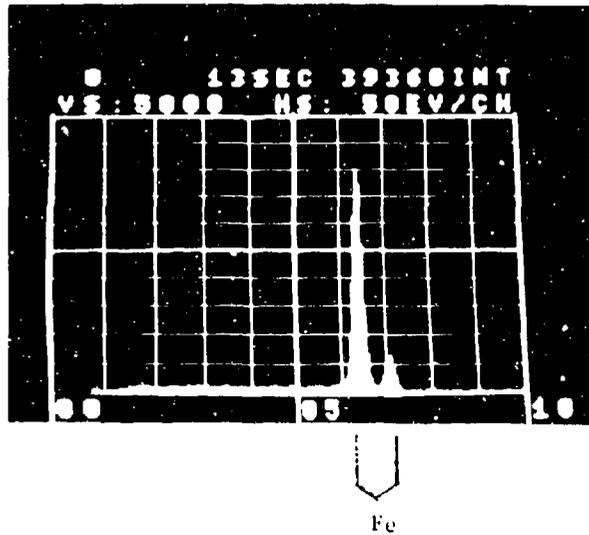
Figure 52. Mild Magnetic Steel: Surface features and composition after NaK₇₈ (+Ba) exposure.

40 μ m



(a) SEM surface

500 hrs., 100°C



(b) Electron excited x-ray spectrum
(100X rastered area)

Figure 53. Mild Magnetic Steel: Surface features and composition after thermal exposure.

Curve 72805-1-1

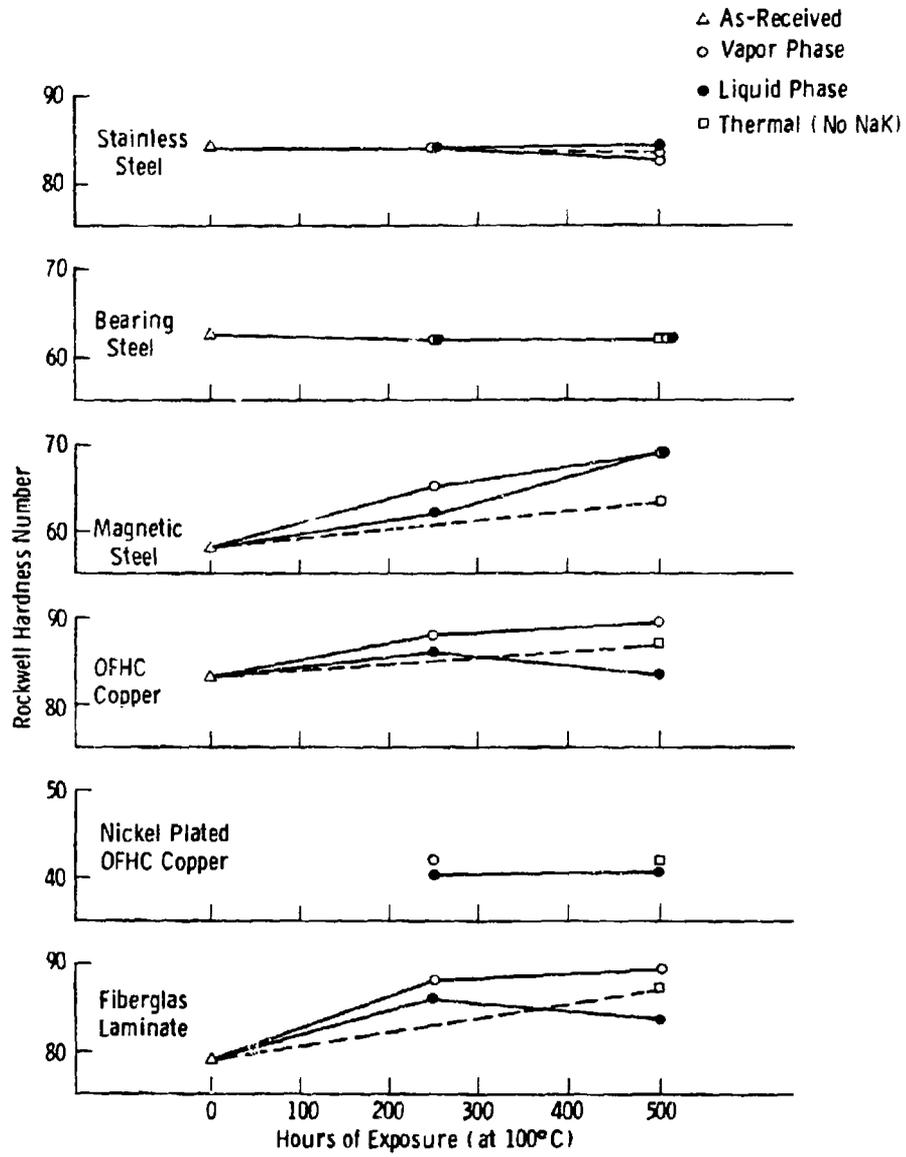


Fig. 54—Hardness profiles of machine materials after exposure to Nak (liquid and vapor) and thermal exposure

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