UNIVERSITY OF DENVER
COLORADO SEMINARY

Denver Research Institute
University Park, Denver 10, Colorado

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National Aeronautics and Space Administration
1520 H Street N. W.
Washington, D. C. 20546

Attention: Office of Grants and Research Contracts, Code SC

Gentlemen:

This letter is a report of progress on Research Grant NsG-365, entitled "Theoretical Studies on the Relationship Between the Thermionic Work Function of Refractory Intermetallic Compounds and Their Electronic and Crystal Structures," for the period 1 July 1964 to 31 December 1964.

1.0 Introduction

Intermetallic compounds exist for all the group VI-A metals and it is the purpose of this investigation to measure the thermionic work function of the σ-compounds and the intermediate phase having the α-Mn structure of the molybdenum-rhenium and tungsten-rhenium systems as well as the σ-phase of the chromium-rhenium. The objective of this study is to measure the thermionic work functions of these five intermetallic phases and determine their relationship to electronic and crystal structure.

2.0 Equipment

A. High Temperature Vacuum Furnace

The high temperature vacuum furnace is complete and has worked completely satisfactorily to produce the annealing treatment required for the intermetallic phases. Above 2000°C the furnace electrode electrical supply cables overheat slightly, but this is a minor difficulty which can be overcome easily at a convenient time.

B. Emissivity Apparatus

Previous investigators have, in general, measured the temperature of the electron emitting cathode by attaching a thermocouple head. We have questioned this practice and initial heat transfer calculations indicate that a three mil thermocouple wire attached to a ten mil cathode causes reduction in the temperature of the cathode at the thermocouple bead as much as 25°C

at 1600°K. The thermocouple wires conduct heat away from the cathode and lower its apparent temperature. The magnitude of this effect is sufficiently large that this source of error cannot be neglected, but must be reduced or this method, though commonly used, will introduce large errors into the derived thermionic data.

Two possible methods exist which might be useful to eliminate this source of error. First, AC electrical heating of the thermocouple wire to approximately a matching temperature with the cathode. The heat loss would be eliminated and the depressed thermocouple bead temperature error would approach zero. This method was not used since the presence of additional AC fields inside the collector rings is a very dubious practice. Secondly the use of optical methods completely sidesteps the difficulty.

Optical observation of the cathode is complicated by the fact that no accurate and useful emittance data are available for the five refractory intermetallics under study. Therefore, since the optical temperature measurement method is the most satisfactory method experimental measurement of the emittances of these materials will be undertaken.

The emittance method selected was a modified Worthing tube method. This is a simple but reliable procedure of obtaining the data. The apparatus consists of a hollow tube electrically heated with an axial flow of current to incandescent temperatures. Near the midpoint of the tube is drilled a small blackbody hole. Emittance data are obtained by alternately observing the specimen surface and the blackbody hole with an optical pyrometer.

To measure the thermal emittance of the five intermetallic phases under study, apparatus has been constructed and tested which uses samples 0.25" OD with a 10 mil wall thickness and 10" long. The intermetallics are electrophoretically applied as coatings to the tantalum tubes. Initial work on stainless steel and gold coated stainless for which reasonably good data are available indicate the apparatus to be performing in a perfectly satisfactory manner.

C. High Voltage Pulse Generator

A 15 KV pulse generator, patterned after the design of Haas, capable of producing pulses of a wide range of widths and pulsing rates has been constructed and is currently being debugged. Certain design deficiencies which presented a safety hazard are being overcome. In general, these consist of changes in the types of connectors used at cable terminations.

Cables are required to carry the high voltage pulse from the generator to the diodes. We believe that all difficulties will be rectified within a short time.

3.0 Diode Fabrication

Diodes suitable for thermionic work function determinations by direct electrical measurement require three distinct steps. These are: 1) Intermetallic alloy preparation; 2) Electrophoretic coating of the cathodes; and 3) Assembly of the diodes.

A. Intermetallic Alloy Preparation

Four of the five intermetallics have been prepared by arc melting and annealing treatments. The chromium-rhenium phase has not been prepared by arc melting due to the difficulty of obtaining the required narrow composition range due to the large difference in the volatility of chromium with respect to rhenium in the arc. Currently it is planned to react these materials using standard powder metallurgy methods at much lower temperatures.

B. Electrophoretic Cathode Coating

Since the electrophoretic coating of cathode wires of 5 to 10 mils diameter is difficult due to the high density of refractory intermetallics which causes a rapid vertical concentration gradient within the coating vessel very fine particles are required. The obtaining of five to ten micron diameter particles of intermetallics free of contamination proved to be a major task. No commercial grinding device was found which could perform this task without contamination. Therefore, it was decided to select a grinding system which would introduce a tramp material which could be removed easily and completely. The selected procedure was as follows: the arc button was crushed to minus 60 mesh in an iron mortar. This coarse powder was ground for approximately 24 hours in a vibratory grinder, also constructed of iron. After grinding the major tramp iron was extracted with a strong magnet. Approximately 50% of the ground powder proved to be iron. Subsequently ten consecutive hydrochloric acid treatments were used to chemically leach out all iron smeared over the surface of the intermetallic particles. Chemical analysis indicated less than 0.005% Fe for all except one sample which showed 0.020% Fe and it was purposely acid leached only a few times to provide a known high iron content sample.

The four samples were supplied to Indelco Corporation of Beverly, Massachusetts and coating was completed by Mid-October. A one to two mil coating was applied to a 5 mil tungsten wire to produce the finished cathodes.

A heat treatment at 1700°C bonded the coatings to the wire but this temperature is sufficiently low to prevent significant interdiffusion of the coating and substrate.

C. Diode Fabrication

Diode fabrication requires the use of "clean room" facilities of a type which the University of Denver does not have. Therefore, a contract was let to the ITT Electron Tube Division, International Telephone and Telegraph Corporation located at Easton, Pennsylvania. The first diode with a pure tungsten filament has been received and one serious defect found. The diode design used by ITT is such that the spring used to hold the cathode straight by taking up thermal dilation possesses such a large spring constant and such a short length that as the cathode heats up the tension is completely relaxed in the spring and the cathode bows out of its central position.

ITT has been notified of this defect and we have supplied them with technical references on the proper design of the cathode support spring. ITT will reopen the diode and insert the proper spring. Delivery of the modified diode is supposed to be made within three weeks.

4.0 Discussion of Future Work

Diode fabrication delays and defects have made it impossible to keep the planned schedule and another grant extension will be required.

Respectfully submitted,

Samuel Miller

C. Samuel Miller
Graduate Research Assistant

Jerry D. Plunkett

Jerry D. Plunkett
Project Supervisor