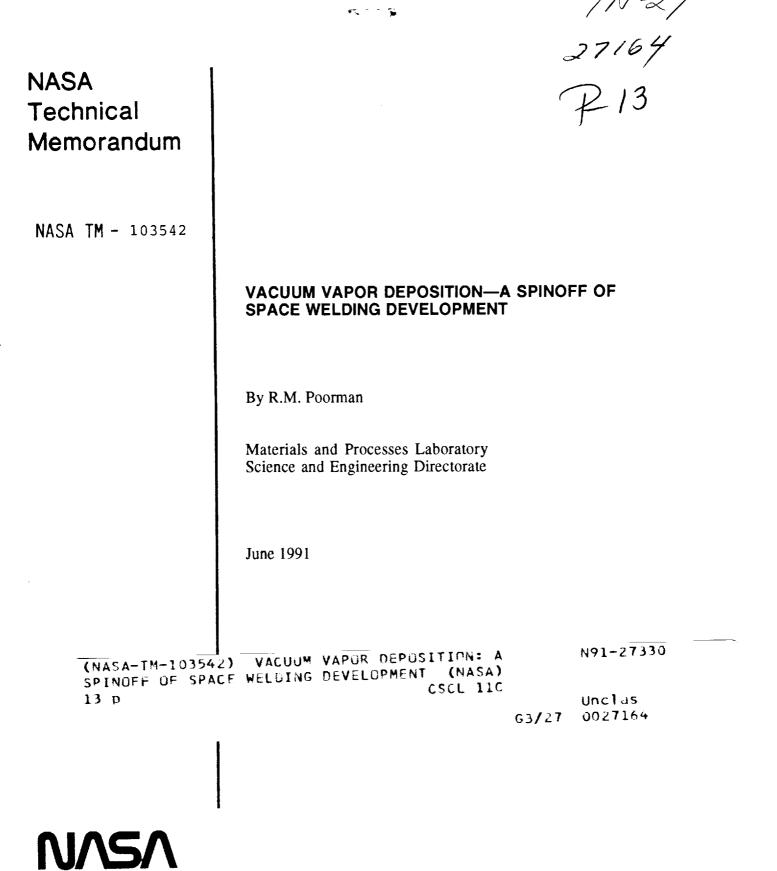
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## TECHNICAL MEMORANDUM

## VACUUM VAPOR DEPOSITION—A SPINOFF OF SPACE WELDING DEVELOPMENT

#### BACKGROUND

The Marshall Space Flight Center (MSFC) has considered many metal joining processes for fabrication, joining subassemblies, maintenance, and repair in space. These were initiated with electron beam welding and exothermic brazing experiments on Skylab I in June of 1973. With Space Station *Freedom* and programs to support other human space exploration, metal joining processes are again getting attention. With these studies, a modified gas tungsten arc welding (GTAW) process is showing much promise. This process utilizes a hollow welding electrode. To weld in a space vacuum environment, an inert gas is fed through the hollow electrode. This allows an arc to be established between the electrode and the work piece via the ionized gas at the electrode. Thus, a GTAW type weld can be made in vacuum. During this work vapor deposits were observed, and this report will describe studies made in producing these vapor deposits.

#### OBJECTIVE

The objective of this work is to characterize vapor deposition produced using a hollow electrode arc in vacuum.

#### APPROACH

The approach is to produce vapor deposits utilizing this unique vacuum welding arc for known and/or some special coating applications. Many coatings were made in this study. No attempt was made to identify all the properties of these coatings or to establish the optimum process conditions. Obviously, there are many process variables, and combinations of these process variables are numerous. This work only attempted to show the potential of the process in some commercial applications.

### **DESCRIPTION OF APPARATUS**

The basic equipment consisted of a vacuum chamber, a uniquely designed vacuum welding torch, a commercial GTAW welding power supply (Lincoln square wave TIG 350), standard (Linde HP20) GTAW welding leads and hoses for water cooling and gas supply. Special "feed throughs" into the vacuum chamber were required for power, gas, and cooling water; both supply and return.

A schematic of the system is shown in figure 1, as well as details of the welding torch in figure 2.

The vacuum chamber (fig. 1, item 11) pressures were normally near 1 micron. With argon gas flowing into the vacuum chamber through the hollow electrode (fig. 1, item 6) at the arc, pressures were maintained by a turbomolecular pump. This pump provides high pumping speeds with minimum back streaming, a condition critical for quality vapor deposition.

The power supply used was a commercial Lincoln square wave TIG 350. Direct current, straight polarity was the primary arc mode for this work. Other modes may be advantageous for special applications. Programmed starts, run current, arc current pulses, and stops are provided with this system. Thus, special effects in the deposits are available.

Power leads and hoses (fig. 1, items 1, 2, and 3) for cooling water and arc gas were those used with a commercial Linde HW20 GTAW torch. The vacuum feed throughs (fig. 1, item 10) need special consideration, primarily to prevent extraneous arcing as the arc is established. Normally, the arc was established through the use of the power supply's high frequency, high voltage start system. However, several other start techniques are known; such as "touch start," preheat of tungsten, and a hot filament in the arc zone. If all electrical leads are not well insulated, random arcing is likely to occur.

### TYPICAL PARAMETERS

The arc current is an important variable in this vapor deposition process. Deposition rate will increase as the arc current increases. Commonly, 100 A were used and currents ranged from 30 to 150 A in several tests. The arc voltage will vary some, 18 to 22 V, over this amperage range. These amperage levels were limited by the electrode size and the torch cooling capacity. The electrode used was 3 mm in diameter. Arc length, gas pressure, and gas composition at the arc will have a minor effect on the required voltage to maintain the arc current.

Gas pressure at the arc is primarily a function of the gas flow. The vacuum chamber pressure will have a minor effect on the gas pressure at the arc but will have a major effect on the vapor deposition. Most of this work was made at chamber pressures near 1 micron. Low pressure improves depositions rates, surface smoothness, cleaning effects of ion bombardment, bond strength, and coating density. The lower vacuum chamber pressure limits are determined by the pumping capacity of the system.

Arc length has a minor influence on the vapor deposition process. Approximately 5 mm was used for most of the work. However, values from 1 to 20 mm have been used. In certain situations, the blowing action of the gas flow may be utilized. The gas is expanding out of the hollow electrode. Short arc lengths obstruct this flow, and a blowing action exists at the surface where the arc is boiling the material that produces the vapor deposit. This blowing action can be used advantageously in producing deposits.

Gas composition is a major variable in this process. Argon was used in most of this work. However, many gases will have special applications. For example, methyl alcohol vapor was used for amorphous carbon coatings containing diamond like structures. Listed below is an often used set of parameters:

Arc voltage - 20 Arc amperage - 100 Arc length - 5 mm Arc gas - argon Flow rate - 0.75 cubic cm per second Vacuum level - 1 micron Material coated - glass Coating - copper Work distance - 8 cm.

#### **PRIOR ART**

Many processes have been utilized in the development of vapor deposition applications. Most, if not all, of these processes are application specific. That is, each process has inherent characteristics that are advantageous for certain particular applications. All tend to have relatively slow deposition rates compared to other nonvapor coating methods.

Most vapor deposition processes are driven by vapor pressure differentials toward the surfaces being coated. And, of course, lower pressure of other gases in the system tends to increase the mass flow of the coating vapors. Lower pressure means fewer obstructions for the vapor flow to the surface being coated.

Nearly all of the commercial processes utilize some techniques for improved deposition efficiency. Electromagnetic and electrostatic fields are often used to direct the vapor flow. These techniques are also used to improve or select the desired particle size utilized in producing the coatings.

The Proceedings of the 14th International Conference on Metallurgical Coatings by R.C. Krutenat, Editor, covered many of the vapor deposition processes commonly used today. They are listed below.

Plasma-Activated Chemical Vapor Deposition Chemical Vapor Deposition Laser Chemical Vapor Deposition Sputtering Cathodic-Spot Arc Coating Electron Beam Evaporation Deposition Steering Arc Evaporation Ion Plating Arc Evaporation Cathodic Arc Plasma Deposition.

### **ADVANTAGES OVER PAST PRACTICE**

This new and innovative vapor deposition process shows a higher deposition rate than perhaps all prior art. This advantage comes by combing low chamber operating pressures, strong pressure differentials within the chamber, and high energies at the melt zone. The pressure differentials result in a flow of gas precisely from the heated source material toward the intended surface to be coated. Thus, this flow of gas helps carry the vapor to the product surface. As this gas flows outward, its pressure rapidly drops from expansion into the surrounding inert vacuum. The inertia of the vapor impinges on and coats the product. In this way, many characteristic advantages of prior art are combined into one new process. That is, this process combines high vapor pressure differentials (high coating rates), directional impetus of the gas flow, a very low absolute pressure at the surface being coated, and an inert high purity environment. All are very desirable conditions for most vapor deposition applications. This is a combination that is not available in any prior art. This process can also support multiple arcs that vaporize more than one material. Thus, manufacturers can produce alloys and/or alternating layers of materials with this process.

#### **COATINGS MADE**

As described in the background section of this report, vapor deposits were observed while making welds in vacuum. The first vapor deposits intentionally produced by this process were a chromium-nickel-iron composition onto pyrex glass. The source material was Inconel 718; a chromium-nickel-iron alloy. However, the deposit was approximately 70-percent chromium, 15-percent nickel, and 15-percent iron. The source material, Inconel 718, is 19-percent chromium, 53-percent nickel, and 18-percent iron. The high chromium content of the coating would be expected when one considers the vapor pressure of these metals at melting temperatures, 1,600 °C. Here, the vapor pressure of chromium is 1 torr and about eight times that of iron or nickel. Thus, a vapor deposited coating by this process is expected to have a composition related to the composition of the vapors over the hot source material.

The chromium-nickel-iron coatings produced were very smooth, highly reflective to light, and thin. The coating rate was estimated at 0.3 mm per hour. Similar coatings on steel showed good corrosion resistance and adhesion.

Coatings of several other materials were made on glass and steel. These included copper, niobium, aluminum, and pure nickel. All showed similar physical properties, e.g., smooth surface, thin but complete coverage, good adhesion, and the natural color of the source material.

Some coatings having special effects were made. One was a copper-niobium coating on graphite fibers. When coated, the fibers were a loosely held bundle. The coating was very thin and essentially surrounded each fiber. Its basic function was to serve as an improved bond to the fibers and as a wetting agent when the fiber bundles were used to form a composite structure. Tests are now being made to assess the relative value in this application.

Another series of coatings was made in an attempt to produce diamond-like structures on stainless steel, glass, and ruby surfaces. The source material for these coatings was graphite. The gas flow through the hollow electrode was methyl alcohol vapor. The resulting coatings were mostly brown-black in color. Scattered through the coatings were small, clear, crystal-like particles of unknown composition. Initial measurements show the coating to be mostly amorphous carbon. The evaluation of these coatings continues.

#### CONCLUSIONS

The applications and volume of business for vapor deposited coatings has grown dramatically within the last few years. From high volume, heavy industry like automotive, to low volume, one-ofa-kind things like research instrumentation. The field for applications is widening. Exotic combinations of property requirements for coatings grow on and on. Perhaps, the largest dollar volume for coatings is within the electronic industry. Here, requirements for different resistivity go from zero for superconductors to infinity for transistor separation. Thermal conductivity is another requirement having a wide range of desired values. Other important properties include reflectivity and/or transmission of different wavelengths of energy, corrosion resistance, stability, absorption, hardness, bond strength, etc.

Although this work has not included detailed assessment of the properties of the various coatings made or the optimum process conditions for any one application, the work has shown a new process for producing vapor deposited coatings. This new process shows high deposition rates, high coating density, a capability for micro thinness, ultrasmooth surface potential, a dramatic reproduction of surface being coated, high bond strength, and ultrahigh coating purity. All are desired characteristics for various coatings applications.

It is concluded that this process can provide a good competitive position for many known and new applications.

- 1. Power and coolant lead
- 2. Welding gas lead
- 3. Coolant return
- 4. Torch body
- 5. Torch bracket
- 6. Hollow electrode
- 7. Target material
- 8. Positioner drive motor
- 9. Substraight (sample)
- 10. Welding feed throughs
- 11. Vacuum chamber wall

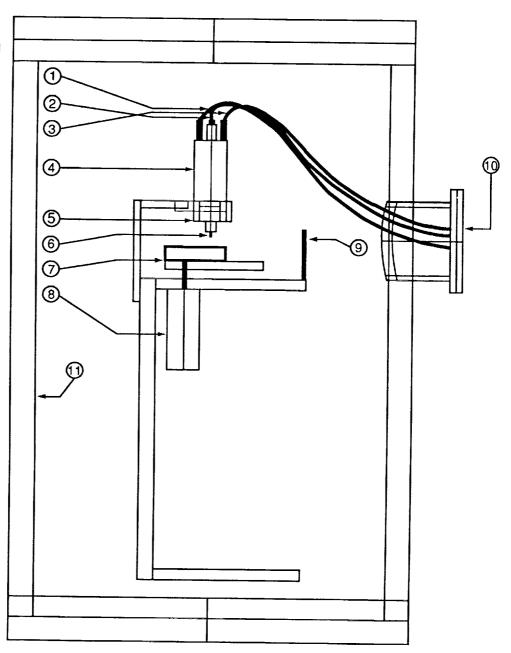


Figure 1. Sketch of vacuum welding system.

- 1. Coolant chambers
- 2. Gas chamber
- 3. Arc start reflector
- 4. Hollow electrode holder
- 5. Hollow electrode gas port

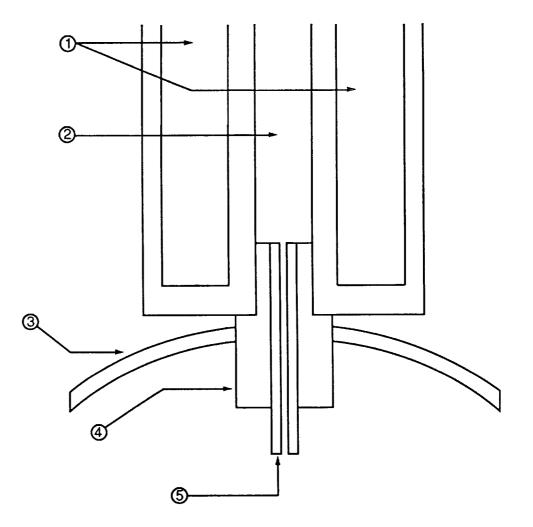


Figure 2. Details of welding torch.

### **APPROVAL**

### VACUUM VAPOR DEPOSITION—A SPINOFF OF SPACE WELDING DEVELOPMENT

By R.M. Poorman

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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