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DEVELOPMENT OF HIGH TEMPERATURE RESISTANT MATERIALS FOR USE IN NAVAL ORDNANCE.

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DEVELOPMENT OF HIGH TEMPERATURE RESISTANT  
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Third Quarterly Report

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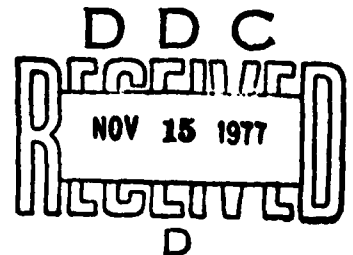
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## 1.0 INTRODUCTION

Pyrostrand<sup>®</sup> graphite composite is a carbon filament reinforced pyrolytic graphite formed by simultaneous pyrolytic graphite deposition and filament addition. A previous program<sup>(1)</sup> demonstrated attractive erosion resistant characteristics for rocket nozzle applications. Additional features of this concept include: (1) the simultaneous deposition of carbides (when desired) to modify the matrix structure, (2) the use of filamentary materials of various textile configurations and mechanical properties and (3) the control of reinforcement placement and content for increased strength and greater erosion resistance in very-high-temperature environments.

The pyrolytic graphite matrix has inherently high resistance to erosion under rocket motor firing conditions. A disadvantage of the material is its tendency to delaminate at moderate or excessive thicknesses. This delamination tendency can be reduced by causing the pyrolytic graphite to grow from surfaces other than the substrate, a filamentary reinforcement for example. The high performance graphite yarns have a very high strength/density ratio and thereby offer the potential for producing composites with a significant degree of reinforcement. In contrast to most materials, the strength of both major components of these composites increases with temperature up to near the sublimation temperature of graphite (6,600°F). The graphite fiber reinforcement sufficiently reduces the anisotropy of the pyrolytic graphite matrix so that freedom from delaminations of the pyrolytic graphite is achieved.

Rocket motor nozzle tests are currently underway in 1 inch throat sizes to establish the applicability of these graphite composite materials (Pyrostrand) to the nozzle conditions of a typical advanced propulsion system. Results to date indicate that, under the test conditions (6500°F propellant, 1000 psi motor pressure), the Pyrostrand graphite composite erodes at an average rate of 2 mils per second for durations up to 46 seconds.<sup>(2)</sup> Work is currently in progress to optimize the yarn reinforcement spacing to further reduce the erosion and, also, to increase the thickness of the composite to provide for durations up to 60 seconds.

Based on the expected erosion rate of 2 mils per second, the pyrostrand graphite composite nozzle appears to be the most desirable throat insert material

for these rocket motor conditions. Other candidates have major disadvantages. For example, tungsten has high weight and vulnerability to nuclear damage, pyrolytic graphite coatings have a maximum thickness capability below that required for the severe C-4 motor conditions and bulk graphites or ablative carbon composites erode excessively. Since the Pyrostrand graphite composite materials are structurally capable of self support, they appear to be desirable throat insert materials.

Past progress has been limited by a small furnace and a small capacity for handling the filamentary material. The results were sufficiently promising to indicate that further work should be conducted to scale up the equipment for the production of larger pieces and to solve the problems identified in earlier studies. The most important of these problems were: (1) voids inside yarn and at yarn crossing points, (2) growth of pyrolytic graphite on broken filament ends, which prevented smooth winding in subsequent layers and (3) insufficient yarn handling capability to prepare thick specimens.

For advanced propulsion systems throat inserts may be required in sizes up to 12.5 inches throat diameter. Assuming an outside diameter of 14 inches, a furnace diameter of approximately 16 inches is indicated, resulting in a fourfold cross sectional area extrapolation. Transition to a fabrication system of this size and demonstration of 7-inch nozzle fabrication and test are the objectives of this program.

A discussion of the program approach was presented in Reference (2). Work accomplished during this report period is described below.

## 2.0 WORK ACCOMPLISHED

Most of the work accomplished related to the procurement of equipment required for the size scale-up. Company funds were used for the purchase of this equipment.

The atmosphere control chamber scheduled for delivery in late February was received on April 10. The induction coil is scheduled for delivery in mid-May.

An objective in both the control chamber and furnace design is to

provide a system suitable for transition to production operation, if required, without significant changes.

The new equipment will consist of three major sections: (1) the vacuum tank to contain the controlled atmosphere, (2) the induction coil and (3) the furnace assembly.

The vacuum tank is 72 inches in diameter and 60 inches high. It was constructed from 304 stainless steel in two sections, split horizontally, and provided with flanges for vacuum sealing. Operations under either vacuum or differential pressure conditions will be possible. A rupture disk system will be installed with a low blow-out rating. This will insure an adequate pressure release in the event of an inadvertent overpressure.

The lower section of the vacuum chamber contains three ports so that the winding of yarn on the mandrel may be observed from different vantage points during a run. All utility connections will pass through the tank wall.

During routine operations the lower half of the tank will remain fixed in place and only the upper tank lid will be removed for access to the furnace area.

The furnace structure will be based upon the 8-inch chamber model presently in use, plus new concepts made possible by the increased size. The maximum furnace chamber diameter was selected to be 19 inches to accommodate nozzle inserts with a throat diameter of 12-1/2 inches. The dimensions of the coil and insulation were proportioned on this basis. The samples to be prepared initially in this furnace will be nozzle inserts of 7-inch throat diameter. To promote efficient deposition of pyrolytic graphite, the free volume between sample and chamber wall must be kept relatively small. Fortunately, with graphite susceptors the chamber diameter may be controlled by varying the wall thickness. The depth of penetration of graphite by the induced current of a magnetic field of 180Hz is approximately 4-1/2 inches. Any wall thickness up to this value will allow the susceptor to operate efficiently. A 4-inch thick susceptor plus a 1/2-inch liner will provide a suitable chamber for the initial furnace studies.

A sealed yarn box will be constructed and positioned on a flange outside of the tank. The yarn box will contain the same pressure conditions that

will exist in the furnace, and also will permit visual observation of the operations within the box.

A copper induction coil 33 inches in diameter and 41 inches high is being fabricated. It will be insulated by wrapping with fiberglass tape and impregnated with high temperature resistant varnish. The first three turns of the induction coil will be used as a cooling coil to provide for temperature gradients in the upper furnace area. The inside of the coil will be lined with alternate layers of refractory cement and fiberglass cloth. A cooling plate on the bottom of the furnace will be bolted to the coil and support the susceptor, work load and lampblack insulation. It will be possible to lift the entire furnace out of the bottom tank section. There will, however, be a certain amount of connections to be taken apart to remove the furnace.

Furnace support systems such as gas dryers, control panels, nitrogen deluge and a closed water cooling system are being actively pursued. Figure 1 shows the schedule for completion of the installation of the new Pyrostrand furnace.

Additional operations under consideration are as follows:

1. Utilization of laser beam illumination to obtain a visual check on the winding operation.
2. A control device for the automatic monitoring of yarn tension.

### 3.0 FUTURE WORK

During the next three month period, all fabrication components should be received. Furnace check-out will then be initiated, followed by the winding of 7 inch prototype nozzles.

Figure 1. 18-inch Pyrostrand Furnace Installation Schedule

	April	May	June
Vacuum Chamber (Arrival)	—		
Induction Coil (Arrival)		—	
Closed Water System (Install)		—	
Furnace Construction		—	
Furnace Test Runs			—
Pyrostrand Test Windings			—
Complete Design Work	—		
Fabrication, Carbon Components		—	

#### REFERENCES

1. Eugent L. Olcott, Atlantic Research, "Development of High Temperature Resistant Materials for Use in Naval Ordnance (U)," Summary Report prepared under Contract No. NO0017-71-C-4401 for Naval Ordnance Systems Command, July 1971. CONFIDENTIAL.
2. "Development of High Temperature Resistant Materials for Use in Naval Ordnance," First Quarterly Report prepared by Atlantic Research Corporation under Contract No. NO0017-73-C-4306 for Naval Ordnance Systems Command, November 15, 1972.
3. "Development of High Temperature Resistant Materials for Use In Naval Ordnance," Second Quarterly Report prepared by Atlantic Research Corporation under Contract No. NO0017-73-C-4306 for Naval Ordnance Systems Command, January 18, 1973.



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13. ABSTRACT

Work continued on the process scale-up to permit the fabrication of filament reinforced graphite composites in sizes up to 16-inch outside diameter. The vacuum chamber was constructed using stainless steel and has been received. The induction coil is being fabricated with delivery scheduled for about the middle of May. Furnace components have been scaled-up and will be available before the next report period.

The scale-up furnace includes several desirable features not available in previous fabrication equipment. These include external filament storage to permit the observation of the filament feed system during fabrication, three view ports to permit the visual observation of the fabrication process inside the furnace and arrangements to permit the use of the equipment for subsequent production requirements.

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