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MARSHALL SPACE FLIGHT CENTER
THE UNIVERSITY OF ALABAMA

AN EXPERIMENT TO STUDY FULLERENE FORMATION UNDER REDUCED GRAVITY

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The study of the carbon molecular species known as fullerenes represented by the molecules of C_{60} and C_{70} is at today's frontier of molecular physics and materials science. Research in this area was initiated by the astrophysical considerations of the nature of complex molecules of the interstellar environment and their probable formation in the outer atmospheres of highly evolved stars (Kroto et al. 1985). The ability to produce C_{60} and C_{70} in gram amounts is also the consequence of researchers having astrophysical intent (Kratschmer et al. 1990a). This has resulted in a technique for a gram amount production of fullerenes incorporating an electric carbon arc with inert gas atmospheres at reduced pressure (Kratschmer et al. 1990b, Haufler et al. 1990, Curl and Smalley 1991). A high current electric are is struck between two carbon rods or a carbon rod and carbon plate in an inert gas such as helium at pressure of ~100 torr. The material produced is vigorously convected upward from the arc and collected in a chimney for later solvent extraction of the fullerene component. Other techniques such as flames have also yielded fullerenes (Howard et al. 1991).

While gram amounts of fullerenes are produced in arcs in inert atmospheres, the initial experiments involving laser ablation and yielding amounts detectable only by mass spectrometry (Kroto et al. 1985), provides insight into the role of the inert gas. In that experiment a pulse of helium applied after laser ablation, swept the vaporized carbon out into a vacuum chamber. This supersonic expansion cooled the vaporized carbon atoms condensing them into clusters including substantial fractions of C_{60} and C_{70}. In the arc process a similar thing occurs except in a continuous fashion as carbon atoms move out from the arc region and into the inert gas atmosphere. However, the process is continuous and extensive convection occurs. Convective transport out of the arc region is a significant parameter in the process therefore it is of obvious interest to control it by carrying out the process under microgravity conditions, and determining the effect of doing so on fullerene production. As buoyancy-driven convection is governed by the Rayleigh number $Ra \propto gL^3$ reduction of gravity to 10^{-3} suggests scale length changes of ~10x.

Since the electric arc/inert gas atmosphere process for fullerene production is technically simple, experimenting with it under reduced gravity offered by drop towers, aircraft trajectories, and space flight ought to be an easy task. These experiments would allow sampling of the material produced for fullerene assay afterwards, and study of the arc and plume during reduced gravity by imaging, interferometry, spectroscopy, and inserted probes. Because fullerene production is a rapid process compared to the duration of reduced gravity for aircraft trajectories and even that of a drop towers (Appendix B of NRA-91-OSSA-17), the development of technique and acquisition of usable data should by possible with those methods. Space flight offers an even more attractive environment. Indeed the integration of fullerene research with microgravity appears to as desirable situation one can ask for research into a new field.

The activity of the summer focused on the design and construction of key components of a carbon arc/inert gas reactor for fullerene production, that is suitable for reduced gravity experiments onbord the KC-135 aircraft. The apparatus will be configured for both floor-mount and free-floating operation providing access to reduction to 10^{-2} and 10^{-3} of normal respectively. It is planned to incorporate "seat belt" restraints that will allow a safe transition from reduced gravity free-float to full gravity, at the end of the parabolic.

A spherical chamber housing two carbon rods will be the core of the reactor. The drive mechanism that will maintain a constant current arc through feedback has been constructed.

After initial laboratory measurements of the performance of a bread board version of the arc control system it will be configured for flight experiments. The chamber to be constructed at UAB this fall will be fitted with ports for view and sample extraction. The view ports will be used for a shadowgraph system will incorporate a diode laser with a CCD camcorder having an interference filter with a band pass at the laser wavelength. The filter will reject most of the light emitted by the arc allowing recording of the shadows induced by density variations in the plume. Because operations of an arc at reduced gravity will be a new experience, shadowgraph imaging will be interesting to say the least. Experiments at reduced gravity involving flames can provide some suggestions of what to expect, however, the energy densities of the arc and low pressures of ~100 torr are quite different from those involved in flames. A laser interferometer could be constructed for follow on experiments in the second and third years, and for use in spaceflight.

The extraction of samples for assay of fullerene production and its correlation with plume imagery and other parameters, will be accomplished by offset carousel rotation of circular substrates over sample extraction port (Figure 1). This apparatus with stepping motor circuitry was completed prior to fabrication of the arc mechanism. The circular substrate materials will include silicon allowing FTIR transmission spectroscopy of the soot deposited from the plume (Kratschmer et al. 1990a). Substitution of quartz allows UV/VIS transmission spectroscopy and a reflective metal substrate makes possible reflection spectroscopy of the deposited plume soot over the entire UV-IR range. Because only a portion of the substrate will be over the extraction port, the sampling will be time-resolved. Raman spectroscopy, electron microscopy, and x-ray diffraction of the deposited material will be carried out. the ~100 torr pressures involved allow rapid removal of coated substrates, replacement with fresh substrate surfaces, pump-down and gas replenishment permitting many "shots" per KC-135 flight. Extraction and concentration of fullerenes with solvents will be carried out after spectral and physical measurements have been made of the neat plume deposits.

In addition to imagery and sample extraction/analysis, determination of temperature gradients will be accomplished by placement of thermocouple strings at various geometries in the reactor chamber. The ability to correlate fullerene production with plume density images and thermal gradients under reduced gravity (10^{-2}-10^{-3} "gee") and normal gravity will provide important information for assessing the role of convection in an arc/inert atmosphere reactor.

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Howard, J.B., McKinnon, J.T., Makarovsky, Y., Lafleur, A.L., and Johnson, M.E. 1991, Nature 352, 139.

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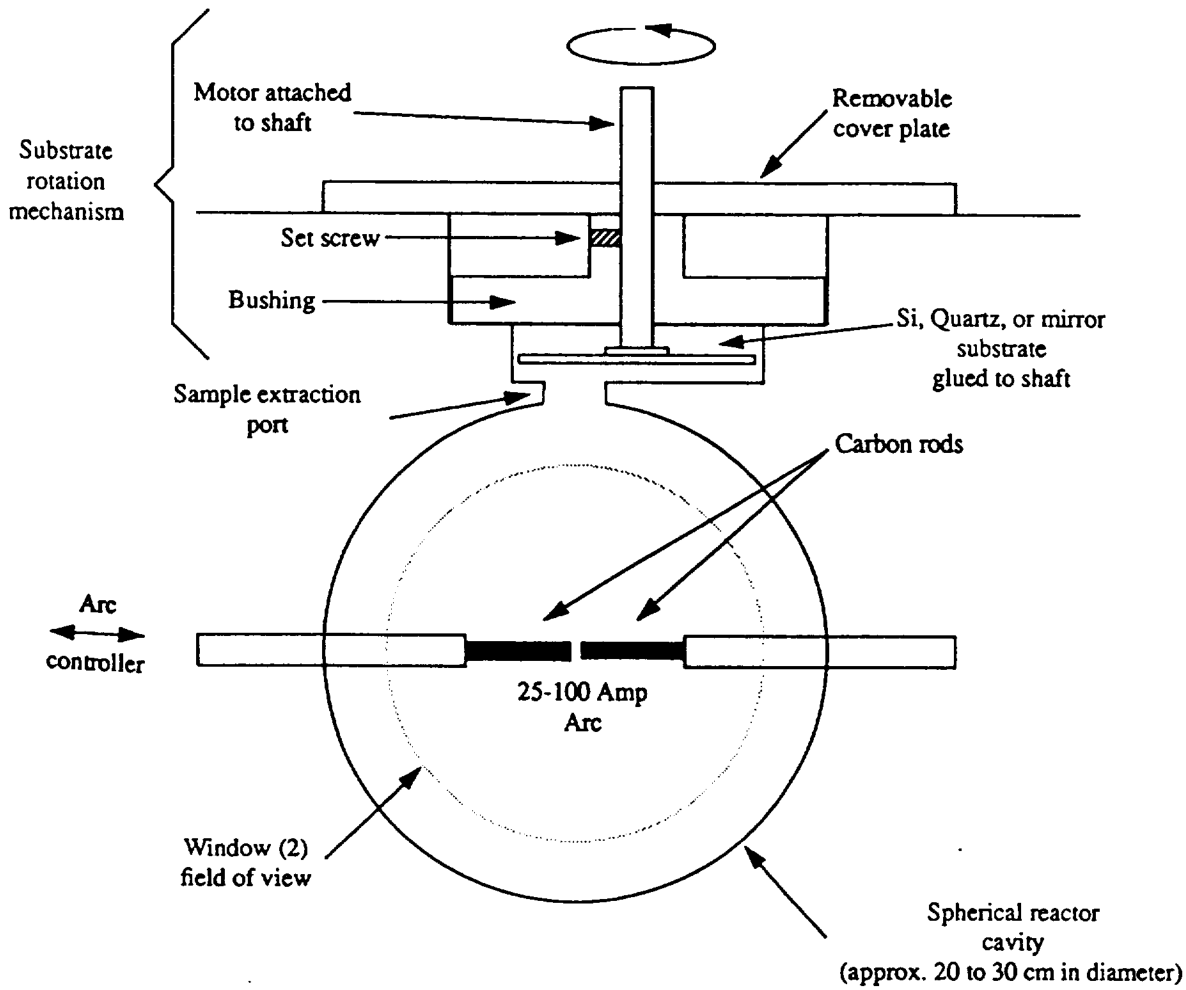

Figure 1. Conceptual configuration (not to scale) of fullerene production reactor for reduced gravity operation. Power for the arc (25-100 amp) to be provided by aircraft supply

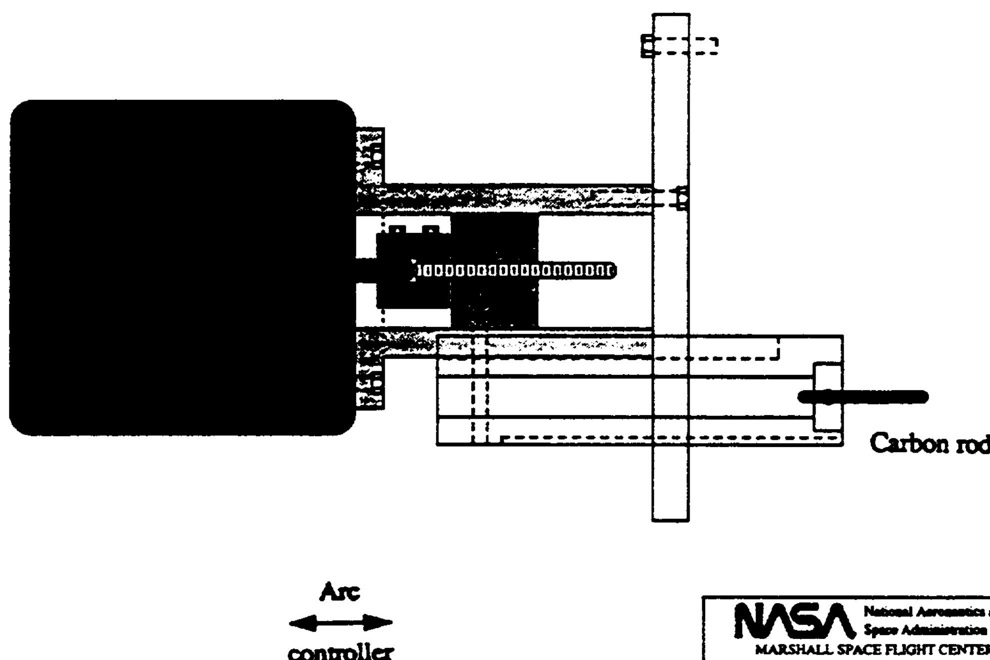

Figure 2

NASA National Aeronautics and Space Administration
MARSHALL SPACE FLIGHT CENTER
CONTROL ELECTRONICS BRANCH

MOTOR SYSTEM ASSEMBLY

DESIGNER: DEAN ALHORN	DWG. NO.

SCALE: 1 / 1	SHEET: 1 OF 2	REV