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# Technology Thresholds for Microgravity: Status and Prospects

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December 1996



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## DEFINITION OF CHEMICAL SYMBOLS

Ag	silver
Ag in Pb	silver and lead
Al	aluminum
Al-Cu	aluminum copper
Al <sub>2</sub> O <sub>3</sub>	aluminum oxide
AlGaInSb	aluminum gallium indium quaternary semiconductor compounds
AlPc-F	fluoro-aluminum
CaO	calcium oxide
CcdB protein	complimentary protein
Cd <sub>1-x</sub> Zn <sub>x</sub> Te	cadmium zinc telluride, ternary semiconductor compounds
CdS	cadmium sulfide
CeMg <sub>3</sub>	cesium manganese
CoSO <sub>4</sub>	cobalt sulfide
Cu	copper
Ga <sub>2</sub> O <sub>3</sub>	gallium oxide
GaAs	gallium arsenide as highly perfect III-V substrates for heterostructures
GaInAsP	gallium indium arsenide phosphorus quaternary semiconductor compounds
GaPAsSb	gallium phosphorus arsenide quaternary semiconductor compounds
GaPc-Cl	chloro-gallium
GaPc-Cl and AlPc-F	phthalocyanines
GaSb	gallium
Gd <sub>2</sub> O <sub>3</sub>	glass oxide
GeSe	germanium selenide
GeSe-GeI <sub>4</sub>	germanium selenide-germanium iodide

GeSe-Xenon	germanium selenide-xenon
Hg <sub>1-x</sub> Cd <sub>x</sub> Te	mercury cadmium telluride, ternary semiconductor compounds
HgCdTe	mercury cadmium telluride
InPAsSb	indium arsenide phosphorus quaternary semiconductor compounds
InSb	indium
La <sub>2</sub> O <sub>3</sub>	lanthaneum oxide
Li <sub>2</sub> O-SiO <sub>2</sub>	lithium oxide, silicon oxide
MLR	monodisperse latex reactor
MnBi	manganese bismuth
MnBi-Bi eutectics formed by MnBi fibers in a Bi matrix	manganese bismuth
Na <sub>2</sub> O-B <sub>2</sub> O <sub>3</sub> -SiO <sub>2</sub>	sodium oxide, boron oxide, silicon oxide
Nb <sub>2</sub> O <sub>3</sub>	niobium oxide
Nb <sub>3</sub> Ge	niobium germanium
Palladium doped TiO <sub>x</sub>	titanium oxide
Pb with 10-percent Ag and BaO particles	lead with silver and barium oxide
PbSnTe	lead tin telluride, ternary semiconductor compounds
Si-Ge	silicon germanium alloy crystals with chemical homogeneity for infrared (IR) detectors
SiC	silicon carbide
Sm <sub>2</sub> O <sub>3</sub>	glass oxide
Ta <sub>2</sub> O <sub>5</sub>	tallic oxide
Th and MgO	thorium and magnesium oxide
ThO <sub>2</sub> particles with Mg particles	thorium oxide
Y <sub>2</sub> O <sub>3</sub>	ytrium oxide
ZrO <sub>x</sub>	zirconium oxide





## TECHNICAL MEMORANDUM

### TECHNOLOGY THRESHOLDS FOR MICROGRAVITY: STATUS AND PROSPECTS

#### INTRODUCING A MARKET SHARE APPROACH

Historically, the space program has drawn wide support based on a number of factors unrelated to applied research and potential benefits. First and foremost, the progress of space flight has centered on the agenda for exploration and discovery. As President Kennedy summarized: "We choose to go . . . not because it is easy, but because it is hard." The pursuit of fundamental science and new frontiers has been and remains at the heart of the space program's efforts.

This document surveys the tangible commercial sectors with a history of priorities that overlap those of microgravity research. The emphasis is on what has been accomplished in the field already; wherever possible these results are demonstrated in a quantifiable fashion compared to the equivalent Earth-based and space-based processing.

The microgravity materials and biophysics program addresses the use of reduced-gravity, vacuum, or radiation effects to improve processing of materials through space-based research. The National Research Council (NRC) identified the area of improvements in materials processing as a national scientific priority for the 1990's. Their report<sup>1</sup> identified the synthesis and processing of novel materials as America's most serious weakness compared to achieving its international goals. The report drew particular attention to this weakness because it "specially impedes the ability to transform America's R&D entrepreneurship into commercial entrepreneurship." This report addresses the concerns for space-based processing of novel materials to usher in technology for potentially large-scale applications.

#### **The Skyline, the Bottom-Line, and the Technology Threshold**

While a number of interesting spinoff applications are featured within the space station era, the thrust of this examination is not single products. Rather, the emphasis is on what would constitute a microgravity-based shift in market share. For this discussion, the market share approach can be summarized as identifying technologies and commercial areas where research can substantially provide an incremental improvement in an existing or planned market. In advanced technologies, the growth and size of the market is large enough that even a few percent improvement in a process or component translates into hundreds of millions of dollars in creating new U.S. and worldwide investment.

The substantial U.S. investment at stake in these critical technologies includes six broad categories: aerospace, transportation, health care, information, energy, and the environment. The breadth of microgravity research addresses each area with current and future experimental programs. As an example, the 1995 United States Microgravity Laboratory (USML-2) crystallized proteins and infrared imaging components for health care screening and the design of therapeutic treatments, formed chemical catalysts (zeolites) for the energy industry including petroleum refining, and conducted research into semiconductor substrates. Each of these contributions will be expanded in some detail, but in general, they underscore the scope of space-related shifts in large markets.

## Technology Aspects of Space Research

For space research, the Government portion contributed to commercial sectors is the majority. For every dollar spent on the Apollo program, \$7 in economic activity were generated.<sup>2</sup> At the core of this multiplier were more than 30,000 spinoffs and applications of the space program—home and automotive design, robotics, programmable implanted medical devices and other advances in medical technology that saves lives, computers, and solid-state electronics (Fletcher, Vital Speeches, 1987). These 30,000 products represent a ten-fold increase in technological innovation since the end of the Apollo program, when the space program amounted to 4 percent of the Federal budget compared to the less than 0.8 percent currently allotted. Therefore, although the program currently receives less than a fourth of its prior funding, the harvest of space applications continues to mount, rising exponentially since 1970.

Before discussing the possible paybacks in detail, an evaluation of the costs is required. Pre-*Challenger* space transportation costs and operations, along with hardware development, averaged about \$5,000/kg.<sup>3</sup> For this most expensive part of doing space research—namely, transportation costs and access to space—the 1995 adjusted costs of the Apollo program translates to more than 100 space shuttle missions like the 1995 USML-2. On a per-flight basis, this brings the economics of space access down by a factor of 9 times compared with the space era peaks in Federal funding.

To date, the commercial viability of selected space-related industries has primarily centered in communications and satellites. Already, combined revenues from all aspects of the commercial space industries are expected to reach \$6.5 billion, an increase of nearly 23 percent over the \$5.3 billion in 1993, mainly from satellites and related communication and natural resource applications. In contrast, present noncommunications space commercial activity is embryonic in size. In 1985, far less than one-hundredth of 1 percent of the American capital markets (\$300 billion in 1985) went to noncommunications space projects, including the payload assist modules, Earth Observation Satellite (EOS) projects, the transfer orbit stage, etc.

The considerable investment in doing space experiments must be accounted for from the outset. Chait<sup>4</sup> estimates that for a space experiment, between 3 and 10 years in total project time and typical investments ranging from \$250,000 to \$10 million are required. For the best use of resources, this lead time and expense requires considerable planning and selection—a route somewhat distinct from the Earth-based scientific methods of experimental trial-and-error. To reduce the cost of candidate material selection and anticipating probable benefits from conducting an experiment in low-Earth orbit (LEO), computer and numerical simulations are increasingly relied upon.

To put these costs on a comparative basis with typical private sector expenditures is revealing. Within the consumer spending market, the cost of a single shuttle mission is a fraction of what Americans commonly consider highly discretionary expenditures. For example, a shuttle mission such as USML-2 costs less than 10 percent of what Americans spend annually on dog food, less than 20 percent of purchases of opera tickets, and a mere 2 to 3 percent of purchases of cigarettes and cosmetics. That same shuttle mission represents a 2-percent fraction of the entire consumer market for toys, of which more than 2 percent of the income-generating sales represents space toys. Apparently the children are willing to buy the shuttle, but the question is: are the adults clear in choosing the real one before the toy ones?

## The Market Share Approach

The market share philosophy is that incremental improvements in a market's efficiency is a tangible reward from space-based research. The strength of a market share approach draws on the historical strengths of NASA science. The space program has never been a commercial vendor, in the sense of the way the Department of Energy (DOE) fostered power utilities or the way the Department of Commerce acts as a direct connection to industry. Rather, NASA has traditionally conducted large-scale collaborative ventures, sharing the results freely with industry, universities, and other Government and

international agencies. While the number of such spinoffs—exceeding 30,000 products—now encompasses all major economic arenas, NASA's singular contribution is to aid the scientific and industrial community, while leading the world in access to space and new innovative concepts. In this regard, assessing the future prospects for microgravity research, a market share approach with a space-based shift in those shares acknowledges both the substantial scope of anticipated Government investment and also the considerable demands of a rather short-term horizon for both Federal budgets and industry interests.

The space program provides the laboratory and a wealth of new ideas, but is not primarily taking the status of a vendor; instead the market share approach provides a way to recognize these contributions.

### **Innovative Aspects of Microgravity Research**

The economics underlying the microgravity gains in market share were discussed critically in a 1987 report in which Nobel Prize winner Sir Brian Pippard analyzed the future of such research. He concluded that while the research was risky, even a small percentage return amounted to a positive balance sheet: "When we turn to the possible technological benefits of microgravity, there can be no guarantee of a reward commensurate with the cost, even in the longer term. But should there be a reward in the form of a better aircraft engine or an improvement of 0.2 percent in the efficiency of a power station, the cost of the research will easily be covered." In other words, a shift in market share is sufficient incentive to conduct research.

For near-term investment in microgravity science, materials science, biotechnology, and fundamental physics provide the bulk of candidates. Because the stakes are so large in many of the advanced technologies now considered promising for space research, even a small fractional improvement in the processing of an electronic component, for example, can deliver a niche.

The remainder of this report will provide concrete examples illustrating this basic approach of incremental changes amounting to tangible returns on investment.

### **Meaningful Metrics for Assessing Progress**

The space program, as a research venture, compares with university and industrial counterparts in both its scope and productivity. MIT and the University of Utah both create spinoffs at about the same rate: 10 percent of licensed inventions enter commercialization. In general, for both universities and the Government, the industrial interest in new startup companies that feature a market advantage is slow. Currently, venture capitalists fund less than one-third of 1 percent of all new commercial enterprise—just 1,600 out of the 300,000 to 600,000 new businesses created every year.<sup>5</sup>

This scarcity of U.S. industrial money is accentuated by the high demands on return. The expected rate of return on venture capital is 100 percent per year, thus all but eclipsing the prospects for unknown or uncertain research to enter the marketplace. While sobering, this analysis does, however, suggest ways to assess Government contributions to tangible consumer benefits, such as the number new startups supported through Small Business Innovative Research (SBIR) grants, the number of outside participants in NASA research from universities and industry, and the return on investments.

In a funding-scarce investment community, the essential role of discretionary funding in fostering innovation is now recognized in a number of university-sponsored consortiums. Several universities already have funds for prototype or proof-of-principle work, with awards in the range of \$5,000 to \$50,000. The structural incentives put in place to foster this kind of startup and innovation include technology incubators, research parks, centers of excellence, manufacturing extension services, venture clubs, forums, and angel networks.

A number of special concerns are important to space-based research. Access to space is the most commonly repeated constraint, but in conducting solicitations or planning for future ventures, the conflict between short-term versus long-term priorities is the crux of the research and development (R&D) dilemma. Furthermore, a different commercialization path can take advantage of a shortest agent to market, which can mix private industry, Government, university, etc.

One interesting observation related to the initiation of new projects is motivation. According to a 1995 Technology Access Report, motivation for entrepreneurs undertaking ventures are, most notably, simple "fear and trembling" of being unprepared in the face of a competitor, followed by reaching to new areas of finance, control of a product or individual's destiny, or pursuit of new gains in an entrepreneurial spirit.

### **Examples of Technology Innovations: Previous Results and Near-Term Prospects**

The attempts to make long-term forecasts are notoriously optimistic or, in some isolated fields like molecular biology, wildly conservative compared to what has already been achieved. Much of the present emphasis on improving the production of novel materials was foreseen as early as 1976, when the seven NASA research centers reviewed the potential for space-based materials processing. As authored more than 20 years ago, "The Forecast of Space Technology: 1980-2000" set priorities for the year 2000.<sup>6</sup> As the Agency enters the last quarter of that projection, it is worth taking account of progress to date.

The Agency-wide goal was then for: (1) an order of magnitude improvement ( $\times 10$ ) in the homogeneity of semiconducting materials, (2) orders-of-magnitude improvements ( $\times 100$ ) in the purity of processing, and (3) containerless processing. As will be described herein, although considerable research objectives remain, these goals have largely been met or exceeded for many example materials, including as much as a thousand-fold reduction in crystalline defects (e.g., as evidenced most recently in cadmium zinc telluride (CdZnTe)) and the containerless processing of many metals, alloys, and in some cases, even proteins. These efforts include contributions to microgravity science R&D, which received, for example, 0.69 percent of the total NASA budget in the 1985 to 1989 period. Thus, while numerous questions remain, the goals of 20 years ago, as outlined by all the NASA research centers, appear to be on schedule to meet or exceed long-term forecasts. A case-by-case examination will look for and highlight virtues, vices, and blank-spots or omissions in attempts to set the next 20-year goals for space-based processing.

As early as 1984, the Microgravity and Materials Processing Facility Definition Study was conducted by Teledyne Brown and identified the major areas for future R&D: (1) electronic materials, (2) metals and alloys, (3) glasses and ceramics, (4) biotechnology, (5) combustion sciences, and (6) polymers.

### **MONODISPERSE NANOMATERIALS**

The first space product sold commercially from the microgravity program (June 1984) was monodisperse latex spheres (fig. 1) used and sold as calibration standards by the National Bureau of Standards. To be used in calibrating electron microscopes, laser light scattering instruments, and particle counters, the observed polydispersity of the uniform space-produced latex spheres was less than 0.4 percent for 10-micron sphere sizes. This small deviation in sphere sizes contrasted markedly with the Earth-based products and defined an effective market niche for the monodisperse latex reactor (MLR). The MLR flew on eight shuttle flights and operated for sphere sizes between 10 and 30 microns.

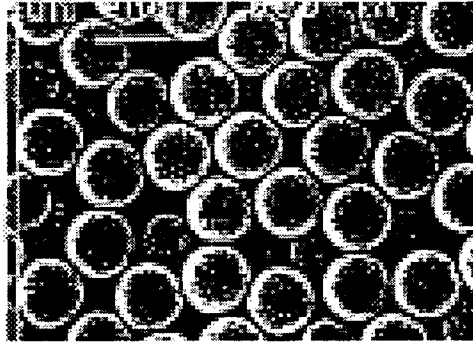


Figure 1. Monodisperse latex spheres.

Because of the commercial history of this space-based processing technique and the road map for its successful marketing, the production of uniform calibration standards is the starting point for considering next steps for returns on research breakthroughs. The next major commercial vista for monodisperse calibration standards is nanomaterials with sphere sizes below 100 nm. This is an effective sphere size 100 times smaller than the MLR's smallest production sizes (10 microns). To serve as standards for miniaturizing devices and components to nanometer scales, these nanoscale powders must deliver both spherical and size uniformity.

Calibration standards are extreme examples of value-added products, whose price range depends highly on their characteristic sizes and dispersity, but can range in costs per pound from \$1,000 to \$100,000. These standards are valued for the following properties: (1) spherical shape, (2) mean sizes ranging from 5 nm to 30 microns, (3) chemical purity and inert, (4) negligible agglutination in both water and air, and (5) monodispersity with a coefficient of variation of less than 0.03.

There are currently no monodisperse or spherical standards below 100 nm. One reason for this gap has been attributed to gravity effects of sedimentation and convection during vapor condensation and powder cloud distortion. Nanomaterials are distinguished within materials sciences as having an average grain size or other domains less than 100 nm. These small sizes present a number of promising changes in physical behavior, in particular when the characteristic length of a process to be transmitted within the nanomaterial exceeds the grain size. These confinement effects have led to considerable interest in new mechanical, optical, electronics, and chemical applications.

Notable examples for nanoscale materials include catalysts with extremely high surface area and chemical activity;<sup>7</sup> optical properties based on visible wavelengths 7 to 10 times higher than the typical nanograin sizes;<sup>8</sup> and extremely strong, hard materials with grains sizes much smaller than the typical Frank-Read dislocation that contributes to traditional yield stresses.<sup>9</sup> It has been estimated that nanocrystalline metals with crystalline grains of the order of a few nanometers would deliver up to a tenfold increase in the stress required to fracture a sample.<sup>10</sup> The research thrust in the field has accelerated to include starting nanomaterials of CdS, palladium doped  $\text{TiO}_x$ ,  $\text{ZrO}_x$ , one-dimensional copper nanomaterials, and nanoalloys.<sup>11</sup>

One identified application for nanoscale standards is the semiconductor industry. Typical defect sizes for the next generation of computer chips will be less than 60 nm. At the present rate of development in semiconductors, the next 5 years will require 1-Gbit devices with 180-nm design rules. For the sizing of these 180-nm design etchings, or for their critical defects in the 60-nm range, no current product on Earth can meet such strenuous requirements. Given the historical commercial success of monodisperse latex in the micron range, the application of nanomaterials for microgravity processing represents an economically attractive area for scientific and technological resources. For multibillion semiconductor economies, a 1-percent contribution in the nanomaterials field amounts to markets sized in tens of millions of dollars. In 1992, semiconductor R&D alone garnered \$14.2 billion in investment

capital, not including revenues generated by the \$123 billion per year worldwide sales by the computer industry.

## MONODISPERSE MICROMATERIALS

In addition to nanomaterials markets, dispersions created in the next highest class of micron-sized matrices of metal-metal dispersions is an obvious area for avoiding uneven gravity distribution. Most metallic alloys for applications in mechanical engineering must be both ductile and hard. Such behavior can be optimized by reinforcing a matrix with a very fine, regularly dispersed phase (1 micron in diameter). Several experiments have been performed in space to produce more uniform dispersions by avoiding sedimentation of the particles in the liquid matrix.

Kawada et al.<sup>12</sup> performed an experiment aboard *Skylab* that was directed toward preparing a high-density, uniform dispersion of silicon carbide whiskers in a silver matrix from the melt. Two-to-10-volume-percent whiskers that were 0.1 microns in diameter and 10 microns in length were mixed with silver powder grains that were 0.5 microns in diameter, compacted and sintered at 900 °C in a hydrogen atmosphere. A spring-loaded plunger was placed in the flight cartridges to avoid the formation of voids. The samples were melted and solidified in space. The whiskers were rather uniformly distributed, whereas in the ground samples they floated to the top of the crucible and agglomerated. The microhardness was found to be higher and more uniform.

Several SPAR rocket experiments were performed to produce dispersion hardened magnesium by adding thoria particles. Raymond<sup>13</sup> compacted 5-micron ThO<sub>2</sub> particles with Mg particles, or alternatively used Th and MgO to form thoria directly by a chemical reaction during the process. After melting and resolidifying in space, the composite material obtained showed considerably greater uniformity in the structure and greater average hardness. Voids were, however, observed, leading to coarse particle dispersions in some parts of the sample. Froyen et al.<sup>14</sup> developed several TEXUS and Spacelab experiments to investigate the complex mechanisms involved in the fabrication of composite materials. They specifically studied interfacial phenomena such as wetting and compound formation, as well as diffusion between the metallic matrix and the dispersed phase. Barbieri et al.<sup>15</sup> focused on the control of bubbles in such materials. Al, Cu, and Ag were used as matrix, with SiC or Al<sub>2</sub>O<sub>3</sub> particles as reinforcing element. They found that exudation of large particles (100 microns in diameter) occurs already during the melting of compacted samples as a consequence of the interfacial reaction between liquid matrix and particles. This is independent of gravity; exudation of finer particles is, however, limited. Agglomeration of small particles may be due to collision of particles by Brownian motion or residual convective flow, leading to the formation of a skeleton of particles held together by interfacial tension or by diffusion reactions (sintering). The volume fraction remains a determining parameter. For most of the Al<sub>2</sub>O<sub>3</sub> and SiC reinforced aluminum samples processed in space, improved uniformity was found for both the macrohardness and the adhesion between the SiC and Al matrix.

## CRYSTAL GROWTH

Much of the promise of microgravity research for large-scale applications has centered on providing the most beneficial conditions for exceptionally pure or perfect crystals. Modern semiconductor materials can cost anywhere from 30 to 40 times the price of silicon (e.g., for gallium arsenide) to upwards of \$500,000 per kilogram for a HgCdTe single crystal.<sup>16</sup> When one recalls that the quality of these materials is far inferior to the best silicon produced today (especially for ternary compounds), space processing for high-performance crystals becomes more attractive.

## Semiconductors

The production yield of integrated circuits and optoelectronic devices is, to a certain extent, a direct result of the quality of the starting materials. Although there are numerous processing steps in the fabrication of such devices, any one of which can affect the overall yield, it is generally accepted that as the process engineering is improved, the starting material will eventually become the yield-limiting step. The quality of the material is a subjective interpretation of quantifiable material properties, which may include the extent of single crystallinity, the number and distribution of dislocations and other defects, and the micro- and macrodistribution (segregation) of added impurities (dopants) (fig. 2). Each of these issues has been, and is currently being, addressed by theoretical investigations, terrestrial experimentation, and microgravity research in LEO.



Figure 2. Mercury iodide space crystal which showed a seven-fold increase in electron mobilities.

The first objective of such microgravity research is to establish quiescent profiles for crystal growth, namely the production of diffusion-controlled conditions relatively free from convection in the melt or vapor phase of a growth method. Carruthers<sup>17</sup> among many others have examined in detail the stability and types of gravity and nongravity related convection, which can disrupt or otherwise determine crystal growth. As early as 1978, an independent R&D review of microgravity crystal growth experiments<sup>18</sup> concluded that space-based processing of germanium, a semiconductor material, provided a firm foundation for the growth of electronic materials in space.

Yoel<sup>19</sup> reviewed the results of 11 flight experiments on germanium-based crystals formed by both chemical and physical vapor transport methods and concluded that the results were internally consistent, that bigger and more microhomogeneous crystals were observed in every case, and that in some cases crystals 100 times larger were achievable in space compared to Earth.<sup>20</sup> Independently, at least 10 long-term studies on the Soviet *Mir* space station<sup>21</sup> have found that along with other semiconducting materials, crystallizing gallium antimonide (GaSb) in microgravity differs appreciably from terrestrial references and has produced "more perfect structures and striation-free crystals." Weidemeier<sup>22</sup> has reviewed 13 different experiments encompassing the spectrum of chemical (CVT) and physical-vapor transport (PVT) and crystal growth of diverse materials (IV-VI compounds) ranging from GeSe-GeI<sub>4</sub> (CVT and PVT) to the GeSe-Xenon (PVT) systems performed during the *Skylab* and Apollo-Soyuz (GeSe-GeI<sub>4</sub>), and during the STS-7 and D-1 (GeSe-Xenon) flights (fig. 3). These results demonstrated "a considerable improvement of the surface and bulk chemical and structural microhomogeneity of space-grown GeSe crystals relative to ground controls. The space crystals are much larger and several grew in the middle of the ampoule without direct wall contact. In the GeSe-Xenon system, space crystals are in very close agreement with theoretically predicted data for diffusion controlled transport."



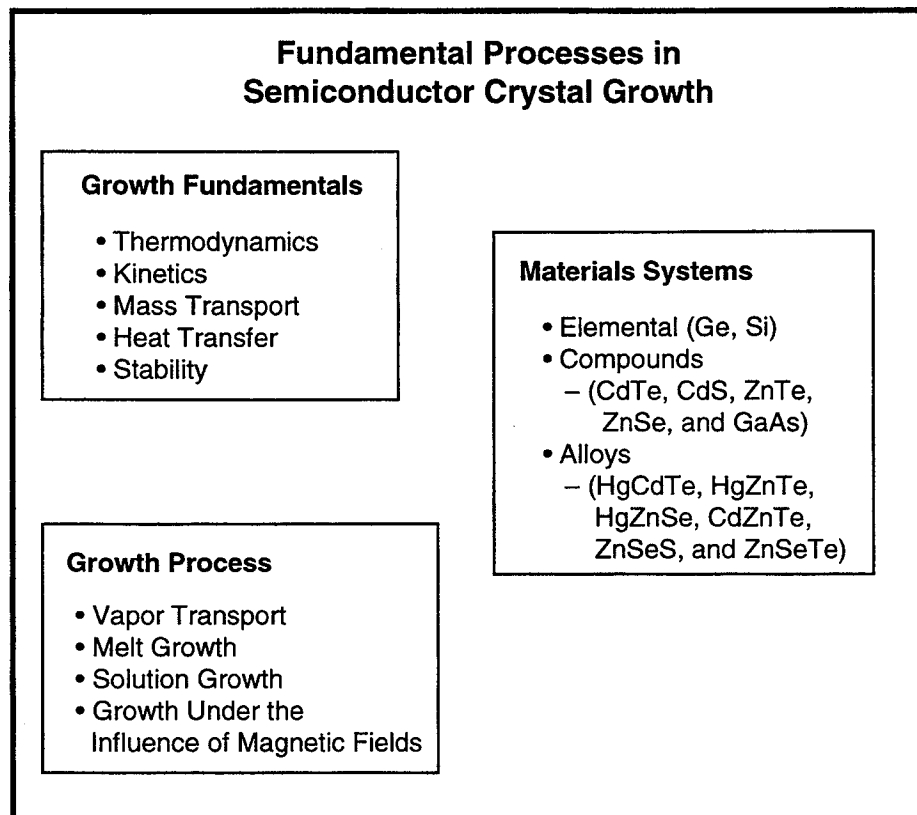


Figure 3. Fundamental processes in semiconductor crystal growth.

In 1993, the technologically important (II-VI) compounds, such Hg-Cd-Te, demonstrated nearly defect-free crystal surfaces, with at least a 1,000-fold reduction in crystalline defects compared to Earth-grown references.<sup>23</sup> Two space-grown crystals used in devices for infrared energy detection showed infrared radiation transmission levels approaching the theoretical maximum. These crystals have traditionally been considered good microgravity candidates because of their extreme sensitivity to very minute fluid disturbances. Wiedemeier<sup>24</sup> independently studied the Hg-Cd-Te system, and found mirror-smooth surfaces (under scanning electron microscopy (SEM)) on the space-crystals, but a wavy step terrace surface with 20- to 200-micron step heights on Earth.

Independent projections for electronic materials have estimated a long-term, space-based economic contribution of between \$6 billion annually (Rockwell International) and \$31 billion annually.<sup>25</sup> The milestones for near-term growth of semiconductor crystals include: (1) production of defined, chemically homogeneous standards of silicon for resistivity and chemical analyses; (2) high resistivity, homogeneous, defect-free silicon, with diameters up to 15 cm for high-voltage, high-current power rectification; (3) large dislocation-free, homogeneously doped silicon for infrared-detector arrays or very large sensor integration (VLSI); (4) intrinsic and extrinsic (indium-doped) Si-Ge alloy crystals with chemical homogeneity for infrared (IR) detectors; (5) highly perfect III-V (e.g., GaAs) substrates for heterostructures; (6) ternary semiconductor compounds (e.g.,  $Cd_{1-x}Zn_xTe$ ,  $Hg_{1-x}Cd_xTe$ ,  $PbSnTe$ ) for R&D; and (7) quaternary semiconductor compounds such as GaInAsP, InPAsSb, GaPAsSb, and AlGaInSb for R&D.

## Computer Equipment Overview

By 1997, more component parts will be put on a silicon chip than on the whole space shuttle—about a billion. Computing power continues to double every year. The number of chip components doubles every 18 months. By 2012, there will be far more computers than Americans, about a billion sold. From 1973, when the first space semiconductor crystals were grown on *Skylab*, the number of calculations possible per second has increased by a factor of 1,000, from fewer than 250,00 calculations per second in 1973, to 250 million calculations per second in 1995.<sup>26</sup> Each day, stocks, currency, and bonds traded on worldwide electronic markets amount to an estimated three trillion dollars, twice the annual U.S. budget.

Industry shipments of equipment by the U.S. computer industry will be more than \$66 billion in 1994, an increase of 6 percent in current dollars, following 8-percent growth in 1993. Imports will rise faster than exports, resulting in a higher computer trade deficit of about \$17 billion in 1994. U.S. manufacturers will continue to restructure their operations and reduce employment, while they face significant challenges in the complex “infotainment” market during the next 5 years. Total industry employment in the United States equals 200,000 workers. By 2010, the current \$600 billion spent each year on information—telecommunications, computers, and video products—will reach trillions of dollars.

R&D has always been critical to this industry’s ability to maintain its technological leadership. Data compiled by *Business Week* in its annual R&D scoreboard survey shows that a combined sample of 87 U.S. computer firms raised their R&D spending by 4 percent, to \$14.2 billion, in 1992. This growth was low relative to the 7-percent average increase for all U.S. industries. However, the computer equipment industry surpassed all other U.S. electronics industries and industrial sectors in absolute spending, and only lagged behind health care in the percentage of sales devoted to R&D. Three U.S. computer firms—IBM, DEC, and Hewlett-Packard—ranked among the top 10 R&D spenders in the United States. The electronics and computer companies had combined worldwide revenues totaling more than \$123 billion in 1991, devoted 12 percent of their revenues to R&D, employed more than 400,000 Americans directly, and created 2 million more jobs indirectly in such fields as software design, computer programming, and services. Roughly 80 percent of their R&D jobs and 60 percent of their production workers remain in the United States. According to the Semiconductor Industry Association, from 1980 to 1992, U.S. companies spent an average 12 percent of annual revenue on R&D and 14 percent of annual revenue on capital equipment and facilities, well above the average of all U.S. industry.

## Anticipated Microgravity Market

Between 1973 and 1995, a number of melt-growth experiments were performed in spacecraft and on sounding rockets. Favorite materials were semiconductors. The reasons for this choice, apart from technological interest, seemed to be the existence of well-established, reproducible growth procedures and characterization techniques as well as good knowledge of defect structures and dopant segregation behavior. The interest in space processing of crystals has been from the beginning the supposition that in a convectionless, quiescent nutrient solution or gas, where heat and mass transport does not occur except through diffusion, better crystals will grow. Modest improvements in structural perfection have been reported, such as reduction in dislocation density or in the number of twins and grain boundaries.

## Decreased Dislocation Densities

On Salyut 6, Kashimov et al.<sup>27</sup> recrystallized undoped and tellurim-doped ( $7 \times 10^{17}/\text{cc}$ ) bars of indium antimonide in quartz ampoules at 0.19 mm/min. The dislocation density in the undoped samples was 100 times smaller than in the terrestrial counterparts, and 20 times smaller in the doped samples. The crystals grew partially wall-free. Markov<sup>28</sup> performed a similar experiment with gallium-doped ( $1 \times 10^{17}$  cc) germanium, confirmed the hoped-for diffusion-driven growth without convective mixing and found a 100-times decrease in dislocation density.

The general finding was that in a variety of semiconductor materials, more quiescent growth of crystals was observed. Witt et al.<sup>29</sup> found in tellurium-doped ( $1 \times 10^{18}$  /cc) crystals grown on *Skylab*, an axial dopant concentration that corresponded to "no mixing" as sought or, in other words, an effective segregation coefficient near unity. Witt et al. regrew a gallium-doped ( $1 \times 10^{19}$ /cc) germanium crystal on the Apollo-Soyuz Test Program mission and found the same space-based profile of diffusion (no mixing) as sought, compared to a terrestrial reference sample that showed convective mixing. Kashimov reported a similar result for a Salyut experiment.

It has been put forward that this no-mixing growth scenario is beneficial in some cases for more uniform crystal growth. Yue and Voltmer<sup>30</sup> resolidified about 35 mm of a gallium-doped ( $8 \times 10^{16}$ /cc) germanium melt at a rate of 5 microns/s on *Skylab* and found a 600-percent decrease in axial macrosegregation of the gallium dopant. Rodot and Totterau<sup>31</sup> reported no striations showing the microscopic inhomogeneity in their silver-doped, melt-grown Spacelab crystals of lead telluride, in contrast to Earth-grown samples. Walter solidified a seeded, gallium-doped germanium melt during a short rocket flight (6 min) and could distinguish the many striations during the strong acceleration phases from the no striations observed in the microgravity phase of the flight.

### **Reduction in Twins and Grain Boundaries**

On *Skylab*, Yee et al.<sup>32</sup> resolidified melts containing InSb and GaSb in the molar ratios 1:1, 3:7, and 1:9. In the polycrystalline ingots, twinning was 70 percent less and the number of ground boundaries was 18 percent less than in terrestrial samples.

### **Software Development**

Fuzzy logic, a technology used in development of artificial intelligence applications, received a great deal of attention in the 1990's. According to Hoover industry profiles, NASA is probably the most active Government organization in the field, with programs in intelligent computer-aided teaching, real-time, vehicle-health maintenance, and space shuttle docking. The potential commercial applications of fuzzy logic are abundant, as the Japanese have shown in more than 100 different product areas, from washing machines and video cameras to elevators and subway trains. Fuzzy logic and neural network revenues will grow at an annual compound rate of 65 percent over the next decade, according to Market Intelligence Research Corp. According to Frost & Sullivan Market Intelligence Research Corp., the combined worldwide market for the combined technologies of neural networks and fuzzy systems by 1998 will be nearly \$10 billion.

Fuzzy logic allows computers to emulate the human reasoning process, which makes decisions based on vague or incomplete data, by assigning values of degree to all the elements of a set. According to Cognizer Almanac, the 1991 global market estimate for fuzzy logic was \$150 million, almost half of which was for training and custom applications. Cognizer predicted that the total market would be \$3.5 billion in 1995. Based on fuzzy-set theory, fuzzy logic recognizes that statements are not necessarily only "true" or "false," but also can be "very unlikely" or "more or less certain."

The use of fuzzy logic in products reduces time-to-market, lowers development costs, and improves product performance. Many U.S. firms have begun to incorporate this technology into their manufacturing processes and products. Ford Motor Co. is currently working on an antilock-braking system that uses fuzzy logic. Motorola's Advanced Microcontroller Division states that within 4 years, half of their microcontrollers will incorporate fuzzy logic.

The incorporation of fuzzy logic into U.S. products and processes is important to U.S. competitiveness. Companies that incorporate fuzzy-based technologies into their operations achieve cost savings through shortened waiting time and reduced energy consumption. In addition, the market for consumer goods with this technology is lucrative and growing. Japan continued to lead in commercial applications

of fuzzy logic technology. In 1990, Japanese company revenues from fuzzy-logic products reached \$1.5 billion. Revenues from such products were less for U.S. firms, but are expected to grow as more companies like Saturn and Ford incorporate the technology into their products.

Fuzzy logic is primarily a software technology and, as a result, major revenues will come from development tools and support services. Fuzzy-control applications are the most successful area for fuzzy-systems development, and many companies are developing hybrid tools with both neural networks and knowledge-based systems. The learning capabilities of neural networks are also important in developing fuzzy rules for programming microcontroller chips.

### **Plasma Processing**

Semiconductor makers are counting on improvements in a technique called plasma processing, in which a partially ionized gas is used to initiate the chemical etching reactions, to drive the process. Such research, says Rebecca Gale, a manager in semiconductor R&D at Texas Instruments (TI), will be "extremely key" over the next decade. As a result, thousands of new technical and research jobs are likely to open in this area. Plasma processing began putting its mark on the computer industry in the early 1980's. Plasma-based etch tools, or reactors, (as opposed to masks) cut much straighter, finer features on many chips at once by exposing masked semiconductor wafers to a partially ionized gas that includes a reactive component, such as fluorine. "Plasmas, I feel, were responsible for ushering in the personal computer revolution," says David Rusic, at the University of Illinois, Urbana-Champaign. Roughly 20 semiconductor fabs, or factories, should be opening each year over the next 5 years. Each fab will create about 1,500 jobs, of which 30 to 40 percent will be technical.<sup>33</sup>

### **Plasma Physics and Heat Transfer on Semiconductor Chips**

The needs for heat dissipation and thermal transfer are driving semiconductor chip density; a ten-fold increase in heat loss efficiency is required by the year 2000. According to estimates by industry representatives, multilayer boards for computers and workstations must be able to handle 800 to 1,200 input/output (I/O) semiconductors by the year 2000. This forecast is based on ongoing increases in chip functions due to shrinking line widths on silicon substrates. Accordingly, heat dissipation needs will increase from 3 W at 200 I/O's to 30 W at an 800 I/O count. Therefore, considerations about heat dissipation will dictate the multilayer board structure and the type of semiconductor used in the end product. According to industry data, the world market for printed circuit boards (PCB's) combining rigid boards and flex circuits was \$18.2 billion in 1992 (\$17.1 billion for rigid boards and \$1.1 billion for flex circuits). Japan and the United States are the largest PCB producers.

## **METALS, ALLOYS, AND COMPOSITES**

For InSb-NiSb, two space experiments showed a thinning of the structure by about 15 percent less distance between the fibers under microgravity in comparison to terrestrially solidified samples<sup>34</sup> (fig. 4). Morphological analyses (of MnBi-Bi eutectics formed by MnBi fibers in a Bi matrix) showed statistically smaller inter-rod spacing and rod diameter with respect to ground samples grown under identical conditions.<sup>35</sup> For binary refractory metals of chromium disulfide CrS<sub>2</sub>, solidified from a low-melting zinc Zn solution, Gurin et al.<sup>36</sup> found a 1.5 to 2 times increase in the sizes of isometric crystals aboard the *Mir* space station, as well as new face forms and a compositional change in crystals obtained. In Al-Cu samples aboard the D-1 mission, the results were considered "very significant." Low-concentration copper alloys (1 percent) with aluminum showed excellent agreement with a prediction of no-mixing and a interdendritic spacing about 5 times larger in space compared to Earth references.<sup>37</sup>

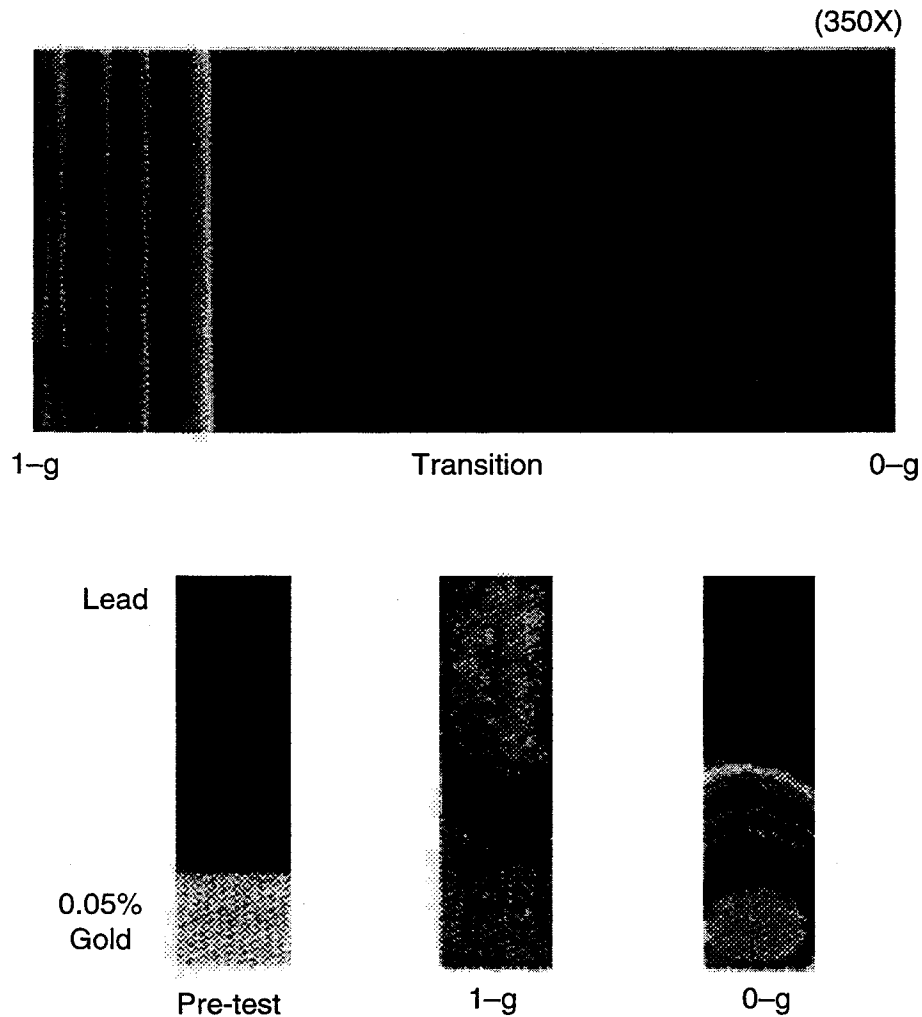


Figure 4. Solidified samples.

The competitiveness of a large volume Earth manufacturing process suggests that the application of space research is primarily for improving the basic understanding of how metals and alloys segregate in gravity and how to improve existing process engineering steps. In 1994, industrial shipments of most metals were expected to increase in the range of 2 to 4 percent, except titanium, which was expected to decline slightly. Shipments by the major metals industries increased moderately in 1993. The principal factor influencing the boost in shipments was the increase in automotive vehicle production. This was a boon to all the industries, except titanium which is heavily dependent upon the flagging aerospace industry. Prices for steel mill products increased, while prices for all nonferrous metals declined as a result of weak demand and mounting inventories worldwide. The large inventories are principally a result of export surges by the countries of the former Soviet Union.

## GLASSES AND CERAMICS

The U.S. production of manufactured ceramic products exceeds \$10 billion annually, which range in scope from solid-state electronics, optical waveguide fibers to more traditional products like window glass.<sup>38</sup> The high-performance market for ceramics is estimated to be \$1 billion per year.<sup>39</sup> The flat glass industry is a \$2 to 3 billion industry with more than 14,000 U.S. employees. The major user of flat glass products is the construction industry, which consumes approximately 57 percent of the output

of the glass industry. The other major consumer is the automotive industry, which accounts for roughly 25 percent. The remainder is taken by producers of "specialty products," including mirrors, solar panels, and advertising signs. The significant microgravity market is the relatively lower volume specialty markets for ultrapure glass and ceramics in premium optics (lasers), modern communications technology (photonic conductors), or electronics applications.

For such reasons, together with the common uses for which ordinary silicate glasses are used, special glasses find increasing applications. They play an essential role in experiments on energy production by laser fusion (SHIVA-NOVA) and they are the starting point for the production of glass-ceramics by controlled crystallization of suitable glasses. Glasses thus appear as a whole a class of materials with increasingly diversified and sophisticated applications. Microgravity should, for example, permit the production of glasses of new critical compositions and of higher purity in containerless processing conditions.

Metallic as well as nonmetallic glasses are of great scientific and industrial interest. Glasses can be formed by a variety of techniques, such as cooling from the liquid state, condensation from vapor, formation from gels, electrogalvanic deposition, cloud discharge, and many others. Of these methods, cooling from the liquid state is by far the most important and most widely used.

Glass formation by cooling liquids requires the achievement of sufficient undercooling to a characteristic temperature, the glass transition temperature  $T_g$ , while avoiding the nucleation of crystallites. Microgravity conditions offer unique advantages for the technology of nonmetallic glasses as well. They are linked to the possibility of containerless melting and processing due to the absence of contamination by the container walls and enhanced control of nucleation and crystallization. In principle, this permits the preparation of glasses of extreme purity and the extension of the range of vitrification to new compositions. However, levitation and positioning techniques are required, which can be operated at the high temperatures necessary for processing; furthermore, the fining problem (removal of gas bubbles) needs to be solved. There is also the difficulty of melt homogenization due to the absence of gravity-driven convection. The gel technique could be one of the means for preparation of gas-free starting materials.

Reactions in high-temperature glass technology are rate-limited by the superposition of both diffusion and convection processes; the latter contribution is especially difficult to assess when the viscosities of the liquid reactants are low. Under reduced gravity, reaction processes can be studied under purely diffusion-controlled conditions, for the examination of the basic underlying kinetic processes in mass transport, corrosion, bubble dissolution, etc. This possibility is indeed of great interest since well-defined experiments cannot be conducted on the ground.

On the *Mir* space station, Regel et al.<sup>40</sup> formed a metallic (semiconducting) glass alloy of  $\text{Te}_{80}\text{Si}_{20}$  and found a more electrically uniform sample produced in space. In the whole temperature range (77 to 300 K), a five-fold reduction in electrical resistivity was observed in microgravity glasses compared to Earth references, owing to "a microgravity glass forming process which tended to the ideal."

One of the simplest techniques by which to perform containerless experiments under microgravity is the use of drop tubes or drop towers. This technique has been utilized recently for nucleation studies in undercooled Pd-Si droplets of 50 to 370 microns in size. Depending on the droplet diameter, the droplets solidified partly in a glassy state during the free-fall.<sup>41</sup> The results suggest heterogeneous surface nucleation to be dominant, which can be related to oxidation at the surface. Similar experiments have also been conducted with Pd-Si, whose glass-forming ability was improved by adding 6-at. percent Cu. The free-fall experiments were conducted in the 32-m drop tube at NASA's Marshall Space Flight Center.<sup>42</sup> Spheres of glassy metals of 1.5-mm diameter have been produced in an amorphous state. The same drop tube was also used to undercool  $\text{Nb}_3\text{Ge}$  droplets with diameters up to 4 mm. Undercoolings up to 500 K have been achieved, leading to bulk metastable  $\text{Nb}_3\text{Ge}$  alloys with metastable A15 structure.<sup>43</sup>

Interesting work was done by Topol et al.,<sup>44</sup> who prepared tiny glass spherules (100 microns in diameter) by laser spin melting and free-fall cooling. New glasses with high contents of  $\text{Nb}_2\text{O}_3$ ,  $\text{Ta}_2\text{O}_5$ ,  $\text{Ga}_2\text{O}_3$ ,  $\text{La}_2\text{O}_3$ ,  $\text{Y}_2\text{O}_3$ ,  $\text{Sm}_2\text{O}_3$ , and  $\text{Gd}_2\text{O}_3$  were obtained. Such tiny glass shells are needed to contain fuel for inertially confined fusion experiments. Although the equipment used did not truly provide free-fall conditions, a high degree of surface smoothness and concentricity could be achieved.

Two types of experiments have been carried out thus far: (1) experiments dealing with glass formation and (2) experiments dealing with kinetic processes. Using a single-axis acoustic levitation an experiment was carried out on SPAR VI in 1979. The sample had a composition of 39.3 mol.-percent  $\text{Ga}_2\text{O}_3$  – 35.7 percent  $\text{CaO}$  – 25 percent  $\text{SiO}_2$ , and even though the levitation process was poorly controlled, a nearly spherical sample with some bubbles inside was obtained.<sup>45</sup>

The Ge-Sb-S chalcogenide system was investigated in the INTER KOS-MOS program.<sup>46</sup> The samples were processed in crucibles. A  $\text{Ge}_{25}\text{Sb}_{20}\text{S}_{55}$  glass, prepared in the KRISTALL furnace on board Soyuz-Salyut, which was cooled rapidly to room temperature, was much more homogeneous than a similar sample processed under 1-g conditions. This was verified by SEM and infrared transmission as well as optical investigation of the microstructure after recrystallization on the ground.

A similar experiment was conducted<sup>47</sup> during the D1-Spacelab mission in October/November 1985. Samples of  $\text{Li}_2\text{O-SiO}_2$  and  $\text{Na}_2\text{O-B}_2\text{O}_3\text{-SiO}_2$  were remelted and solidified in containers in the isothermal heating facility (IHF). The temperature-time profiles show the result: the space-processed sample is much more homogeneous. Measurements with a Christiansen-Shelyubskii filter show that the flight sample has a much more narrow distribution of the refractive index than the ground sample, which indicates that the melting in space yielded much more homogeneous glasses even when a crucible is used.

## POLYMERS

Polymeric materials were originally and incorrectly assumed to be too viscous and high volume to benefit from microgravity. However, the variety of lower molecular weight organics, polymers, and composites, and the premium optoelectronic applications that have appeared, now make it a promising area, particularly for solution polymerizations. Important applications, such as nonlinear optics, computing, switching, and communications, are now major technologies. Broadly, this category (SIC 2821) groups together various petroleum-derived monomeric and polymeric materials, whether used singly or in combination, to make a wide variety of molded plastic shapes. Production of plastics follows a well-defined sequence: three primary materials (petroleum, natural gas, and coal) are broken down by refining and fractionation processes into various light-to-heavy petrochemical feedstocks. These materials, also known as light, middle, and heavy oils, are then reacted with others to make more complex intermediates. These can be further reacted with accelerating agents to yield low molecular weight monomers and the heavier, more complex polymers.

Total output of U.S. plastic materials producers in 1992 reached an estimated 66.6 billion pounds. Profit margins that had eroded in 1990 and 1991 were partially offset in 1992 as prices stabilized. In volume terms, demand in 1992 was highest for the low- and high-density polyethylenes, polypropylene, polyvinyl chloride, polystyrene, and acrylonitrile butadiene styrene. The fastest growing market segments in 1992, however, were the engineering resins, high-density polyethylene, polyvinyl chloride, and the polyolefins. Quantum, Union Carbide, Dow Chemical, Shin-Etsu, Formosa Plastics, Himont, Amoco, Exxon, FINA, Huntsman Chemical, Occidental Petroleum, and Bayer are among the largest producers of plastic materials. Although of interest to building space structures with low-weight, high-strength ratios, the specialty uses of polymers are most suitable for microgravity materials research.

Most applications of lasers and fiber optics depend on molecules and crystals that show nonlinear optical activity. This can involve doubling or tripling the frequency of laser light. A laser of standard type then can produce light with a wavelength better suited for its intended use. Another type of activity rotates the plane of polarization of light in response to an applied electric field (the Pockels effect) to modulate or switch an optical signal. As an example, in preparing polymeric materials with optoelectronic applications, the thrust has been the search for polarizable molecules with significant nonlinear optical activity. These have an electron donor at one end and an electron acceptor at the other, separated by a bridge. A bridge is an organic or polymeric structure containing both single and double carbon-to-carbon bonds.

Polymer thin films with fewer defects and more uniform thickness, which would provide superior optical devices, might be prepared by electrochemical polymerization in microgravity due to the elimination of solutal convection. This is indicated by the work of Owen<sup>48</sup> and Riley et al.,<sup>49</sup> who used the laser shadowgraph/Schlieren technique to observe the concentration gradients in a 1-molar  $\text{CoSO}_4$  cell during electrodeposition experiments on a KC-135 aircraft in parabolic flight. At identical times into the electrodeposition experiments, comparisons were made of the ground-based and low-gravity processes. Shadowgraphs showed the absence of "plumes" at the electrode surface in low gravity. The feasibility of using electrochemical polymerization to prepare third-order nonlinear optical (NLO) polymer thin films for use in devices was demonstrated by Dorsinville et al.<sup>50</sup> These researchers used this process to prepare films of polythiophene and a homologous series of thiophene-based polymers that had  $X(3)$  values that are among the largest and fastest for polymers. The maximum measured value for the series was  $11 \times 10^{-9}$  esu at 532 nm, which is comparable to measured values obtained for polydiacetylenes.<sup>51</sup>

Organic thin films of phthalocyanines prepared by vapor deposition processes are excellent candidates for the development of nonlinear optical devices because these materials have two-dimensional planar p-conjugated systems and excellent stability against heat, chemicals, and photo-irradiation. Ho et al.<sup>52</sup> grew thin films of chloro-gallium (GaPc-Cl) and fluoro-aluminum (AlPc-F) phthalocyanines by vapor deposition onto fused silica optical flats at 150 °C and  $10^{-6}$  Torr. The thickness of the GaPc-Cl and AlPc-F were 1.2 and 0.8 microns and the values for  $X(3)$  were  $5 \times 10^{-11}$  and  $2.5 \times 10^{-11}$  esu, respectively. The research of Debe et al.<sup>53</sup> indicates that better quality thin films for use in NLO devices might be obtained by closed cell physical vapor transport (PVT) in microgravity. In the PVT process, the source material is sublimed in an inert gas and allowed to convect or diffuse down a thermal gradient and to ultimately condense at a crystal or thin film growth interface (fig. 5). The advantage of thin film growth in microgravity is that it provides the opportunity to eliminate buoyancy-driven convection.

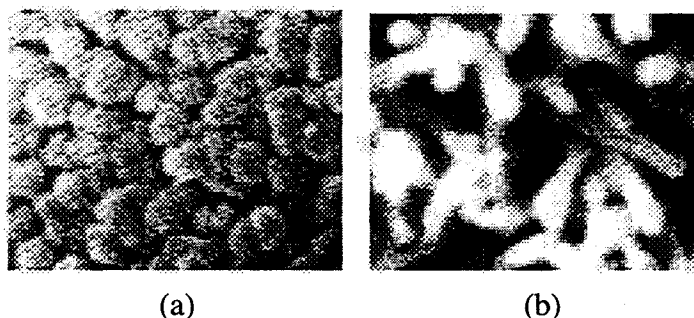


Figure 5. Thin film morphology of (a) space and (b) ground samples of copper phthalocyanine ( $\times 30,000$ ).

Debe reported the results of experiments in which copper phthalocyanines (CuPc) were epitaxially deposited onto highly oriented seed films of metal free phthalocyanine (H2Pc) contained on a 1.4-cm diameter solid copper disc. Analysis involving the use of external reflection-absorption IR spectroscopy, grazing incidence x-ray diffraction, and visible near IR reflection-absorption spectroscopy



reveal that the microgravity-grown films are more highly uniaxially oriented than Earth-grown films, and consisted prominently of crystalline domains of a previously unknown polymorphic form of CuPc. In addition, SEM analysis revealed that there was a distinctly different microstructure in the center of the space-grown films and that the circular perimeters of the microgravity-grown films had a microstructure much like that of the ground-control films.

## SUPERCONDUCTORS

Superconductivity, a phenomena that occurs in certain metals or alloys, ceramics, and carbon-cluster compounds called fullerenes, is characterized by a vanishing electrical resistance at a specific temperature and the expulsion of magnetic fields (the Meissner effect). In microelectronics, the ultimate performance levels of superconductors—high speed, high sensitivity, a high degree of accuracy, low power consumption, and low dispersion—are unmatched by any devices based on other materials. Superconductors are considered one of the critical future technologies in defense and commercial applications.

In response to the important discovery in 1986 of high-transition temperature (30 K) superconductors, governments dramatically increased their funding of superconductivity R&D. Currently, the U.S. Government is spending \$246 million; Japan, \$149 million; Germany, \$45 million; the United Kingdom, \$20 to \$25 million; France, \$22 million; and the European Commission, \$20 to \$30 million. Since 1987, significant progress has been achieved in discovering new compounds with higher transition temperature, increasing the current carrying capacity of thin films and wires, the development of prototype devices, and the demonstration of hybrid superconductor-semiconductor subsystems.

The second International Superconductivity Summit (ISIS) took place in May 1993 in Hakone, Japan. ISIS is a multilateral cooperative effort by the Council on Superconductivity for American Competitiveness (CSAC), the International Superconductivity Technology Center (ISTEC) in Japan, and a consortium of European companies determined to use superconductivity (CONNECTUS). An ISIS survey of 70 companies involved in superconductivity projected that the current market of \$1.5 billion for the use of superconductors would increase to \$8 to \$12 billion by the year 2000, \$60 to \$90 billion by 2010, and \$150 to \$200 billion by 2020.

The \$78 million for R&D was divided as follows: about 38 percent in enabling technologies (i.e., materials, film, wire and tape processing, and cryogenics); 27 percent in components and devices (e.g., magnets, superconducting quantum interference devices (SQUID's), analog-to-digital converters, and interconnects); and 35 percent in systems and applications.

### Microgravity Objectives

Type II superconductors are materials of interest for storing electrical energy. However, such materials should preferably be ductile so that wires can be made. Heye and Klemm<sup>54</sup> have carried out a sounding rocket experiment with the objective to obtain a fine particle dispersion. They specifically wanted to produce a type II superconductor in which dispersed particles act as flux pinning sites. The samples consisted of Pb with 10 percent Ag and BaO particles. The fine dispersion of Ag in Pb obtained was sufficiently uniform to exhibit type II superconducting properties.

Superconductivity research is now reaching a sufficient level of theoretical understanding to predict the behavior of particular phases. This is the case for magnetic properties, where the coercivity can be related to the atomic arrangement and the crystallographic structure. Often complicated phase diagrams including peritectic reactions and reactive materials are involved. The preparation of these crystals is difficult; it depends on the control of composition, the homogeneity of the initial liquid, and one has to avoid contamination from the crucible, which also activate heterogeneous nucleation. These

parameters could eventually be better controlled in a microgravity environment, where sedimentation, contact with a crucible, and variations in liquid composition can be avoided or better controlled.

On this basis, Pierre et al.<sup>55</sup> have successfully prepared large single crystals of  $\text{CeMg}_3$  during a sounding rocket flight, which they could not obtain on the ground. Another example is the MnBi experiments performed during TEXUS and STS flights.<sup>56</sup> Samples consisting of 95 percent MnBi have been produced. They had the expected coercivity, which proves that difficult materials can eventually be produced in space.

It seems unlikely that commercially competitive dispersions could ever be made in microgravity by freezing a metal with solid precipitations. These processes could not be competitive with processes such as spray deposition of metals and ceramics or processes such as rheo-casting, where ceramic particles can be stirred into a semi-solid metal. In these Earth-bound processes, dispersions are being made at little more than materials cost. Rather the potential benefit for experiments in space would appear to be in the field of a better scientific understanding of processes such as particle pushing, ripening of a solid surrounded by a liquid, and the coarsening of a two-phase liquid. The better understanding of these phenomena could lead to improved materials manufactured on Earth.

### **Technology Developments**

In 1993, there occurred the discovery of a new class of high-temperature cuprate superconductors, with transition temperatures around 140 K. This class of superconductors based on mercury has achieved a slightly higher transition temperature than the thallium-based superconductors, which had a previous record high of 125 K. Research continues on the basic properties of films, enhanced deposition rates, and lower deposition temperatures. High-temperature film processing technology has reached the stage where the growth of yttrium compounds now is standard procedure, and the processing of thallium-based compounds is well-developed.

Multilayer technology is commercialized at the 2-cm level, and 5-cm circuit processing and 10-cm film growth are being developed. Interconnects of 2 microns have been demonstrated. High-temperature, integrated circuit technology has progressed to the point where several Josephson-type junctions (a device consisting of an extremely thin insulating barrier sandwiched between two superconductors that is capable of extremely fast switching) are in use for development. Several SNS junctions (superconductor-normal metal-superconductor tunnel junctions) also have been developed. Three-terminal devices, which have the advantage of high-frequency and high-output voltage and can be used as potential interfaces between superconductors and semiconductors, are under active development. A 32-bit yttrium shift register, a basic component in computer circuits, has been constructed. Multichip modules are being developed for demonstration in avionic systems.

In the microwave area, a number of components (delay lines, filters, antennas, modulator-demodulators, resonators) have been developed and scheduled for testing in space. Detection coils 10 times more sensitive than conventional coils have been developed for magnetic resonance applications. In general, the state of technology in high-temperature superconductors has advanced to the point where developments in film processing and devices are capable of being introduced into some commercial markets. Manufacturers are seeking approval from the U.S. Food and Drug Administration for high-temperature SQUID's for better magnetic resonance imaging (MRI) and brain and heart diagnosis equipment.

### **Industry Sector Results of Market Survey**

If progress and funding continue at the present rate, researchers predict that within the next 5 years significant progress will be made in both low-temperature and high-temperature superconductors. A 5-year scenario for the former would include: (1) demonstration of an analog-to-digital converter (an

electronic device that translates an analog signal from a sensor, for example, to a set of corresponding discrete signals intelligible to a computer), combined with a shift register (devices used in computer and data processing systems as storage or delay elements); (2) demonstration of high-speed crossbar switches, hybrid complementary metal oxide semiconductor (CMOS), and Josephson technology; (3) faster logic circuits; and (4) improved refrigeration. A 5-year scenario for high-temperature superconductors would include: (1) development of multilayer, planarized integrated circuits; (2) improved SNS Josephson junctions; (3) development of various three-terminal devices, hybrid CMOS, and Josephson junctions; and (4) solutions to the design of individual circuits.

At the second ISIS, industry experts from the United States, Japan, and Europe stated that while additional R&D and manufacturing scale-up activities are required to achieve full commercialization of high-temperature superconductor (HTS) technology, it is also clear that commercialization will occur in the near-term; it is no longer a question if HTS technology will be commercialized, but when. Companies and governments that invest aggressively in HTS technology development will enjoy the benefits of participating in a major new industrial sector by the turn of the century. An ISIS survey of about 70 companies in the superconductor industry indicates that the major commercial market for superconductors, mainly low temperature, is now the medical and scientific area, where superconducting magnets are extensively used in MRI and spectroscopy. The survey indicated that, by the next century, applications in electronics, energy, and other areas outside of the medical and scientific field will increase to 70 percent of the projected \$8 to \$12 billion market. Within the next 30 years, electronic applications are projected to increase steadily at the expense of other markets and will eventually be the largest application.

## ZEOLITES

Zeolites are a class of crystalline aluminosilicate materials that form the backbone of the chemical process industry worldwide. They are used primarily as adsorbents and catalysts, and they support, to a significant extent, the positive balance of trade realized by the chemical industry in the United States (around \$19 billion in 1991).<sup>57</sup> Since their introduction as "petroleum cracking catalysts" in the early 1960's, they have saved the equivalent of 60 percent of the total oil production from Alaska's North Slope. Thus, the performance of zeolite catalysts has economic ramifications. It is estimated that a 1-percent increase in the yield of a gasoline fraction per barrel of oil would represent a savings of 22 million barrels of crude oil per year, representing a reduction of \$400 million in the U.S. balance of payments.<sup>58</sup>

Flight results on zeolite growth in microgravity (fig. 6) have revealed that "larger, more defect-free zeolite crystals can be grown in high-yield in space."<sup>59</sup> The size increase for the chemical formulations flown, zeolite A and zeolite X, varied between 10 to 50 percent. Characterization of the flight samples versus their ground references, indicated that the lattice defect concentration is reduced when these crystals are produced in space. The result of these experiments produced the first perfect zeolite crystal with the theoretical limit of a ratio between silica and aluminum (Si/Al) near one.

In 1950, the zeolite catalyst market was a \$50 million/year commercial enterprise that has grown 1,000 fold to a billion dollars or more by 1990. The acceleration of that growth has been exponential, with a doubling of the sales from 1950 to 1970, a tripling from 1970 to 1980, and a 5 to 6 fold increase from 1970 to 1990. For catalysis, zeolites provide a large internal and external surface area for carrying out chemical reactions. In energy industries, zeolites are used to crack or reduce the molecular weight of large molecular weight hydrocarbon fractions to refine petroleum to gasoline. This use for zeolites constitutes a 29-percent portion of its total market, with specialty markets (such as research devices for quantum dot and integrated circuit design) representing 10 percent, use as adsorbents in ion exchange over 50 percent, and the remainder broadly classified as natural products for the chemical industry.

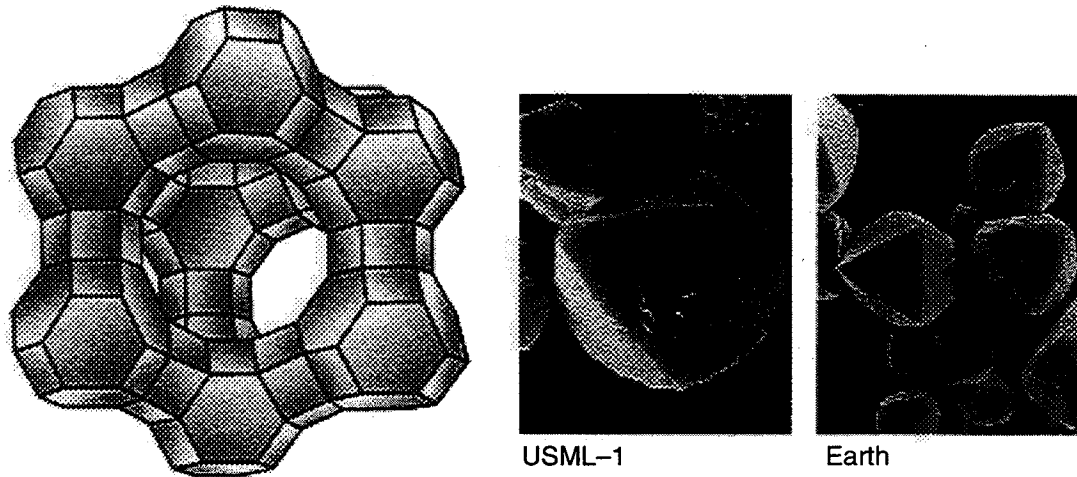


Figure 6. Zeolite growth in space.

The annual U.S. consumption for catalysts is in controlling automobile exhausts (\$300 to \$600 million per year), in petroleum refining (\$168 million/year), in the chemical industry (\$95 million per year), and in the synthetic natural gas (SNG) industry (\$30 million/year). Within the petroleum refining industry, zeolites feature in catalytic cracking of heavy hydrocarbons in fluid beds (226 million pounds of catalysts per year or \$56.5 million per year) and in moving beds (27.2 million pounds of catalysts per year or \$9.5 million per year). Alumina-silica zeolites are the method of choice for fluid bed processing.

In engineering a catalyst, the correct formulation is a compromise between designs that allow fluid flow through the pores of the catalyst, chemical activity based on composition, and available surface area for reaction and mechanical stability. To produce desirable catalysts, the manufacturing depends on the chosen reaction, the reactor design, process conditions, and economics of construction. A high fluid flow rate translates into increased volume for production, but fluid should not distribute nonuniformly across the fluidized bed; it should have a low pressure drop and high mechanical strength. If mechanical strength is low, the catalyst can break up from the weight of fluid and catalyst above it and produce channeling and unreacted throughput. For maximum chemical activity of the catalytic pellet, it should principally have a porous composition with high specific surface area. The final stability of the pellet will depend on its resistance to poisoning, fouling, and sintering.

The second major use for zeolites is in the production of optoelectronic devices and quantum dots. Resulting devices include very fast transistors, tiny solid-state lasers, and advanced solar cells. Such fabrication techniques permit zeolites to serve as scaffolding to support novel semiconducting structures. Quantum wells, known since the 1970's, attract electrons and confine them in two-dimensional sheets. Quantum wires are the electronic analog of a single mode optical fiber. Quantum dots, 10 to 20 nanometers in diameter, confine electrons in zero dimensions as an effective cluster of atoms with quantized electron energy states. Several research groups are currently working toward incorporation of arrays of quantum dots and wires in optical wave guides that would be suitable for use as a laser or optical amplifier. Such devices should exhibit greatly improved efficiency, with a broad range of applications.

## PROTEIN CRYSTAL GROWTH

Since 1984, protein crystal growth experiments have been performed on more than 20 space shuttle missions. These experiments have crystallized proteins using vapor diffusion, liquid diffusion, and temperature-induced crystallization techniques. In a number of cases reported by a diverse group of

university, industrial and government investigators, the proteins grown in microgravity (>20 percent) may be larger, display more uniform morphologies, and yield x-ray diffraction data to significantly (>25 percent in some cases) higher resolution than the best crystals of these proteins grown on Earth.

Of all the imaging techniques, x-ray crystallography can usually provide the most complete picture of a protein's structure. It does so by revealing the density and location in space of a protein's electron cloud. This provides information on where the different atoms of a protein might be located, because different atoms have different electron densities. The technique's biggest limitation is that the protein must be crystallized. A case in point of protein crystallization in nature is the formation of human cataracts. The human eye lens has a protein content in excess of 35 percent, far exceeding even the leanest muscles with protein contents about 17 percent. Cataracts affect millions of people and engage a medical costs in excess of \$5 billion annually. However, unlike cataract formation, protein crystals formed for determining the enzymatic structure has been sought for providing new therapeutics and a basic understanding for biology and medicine.

Solving the three-dimensional structure of proteins by crystallization and x-ray diffraction analysis has proved to be a valuable tool in the fundamental understanding of hormone action, disease prevention and most recently in the design of therapeutics. There exist more than 300,000 proteins in the human body, of which the three-dimensional shape and structure has been so far solved for fewer than 1 percent. Examples include the blood protein, hemoglobin, and the potential cancer therapeutic, alpha-interferon. The size of this undertaking can be illustrated by referring to the current rate of space experiments. At a rate of six shuttle flights per year dedicated to crystallizing 1,000 different proteins on each flight, this large biological research area would theoretically not have to duplicate a crystallization experiment until the year 2030. This 45-year undertaking would only exhaust the 300,000 human proteins well into the next century and does not assume any reflight or limitations in protein availability from the molecular biology community. In other words, this field, if a significant space-related advantage is demonstrated, will keep an active research community busy for years to come.

A few case studies illustrate the progress to date. Among the greater than 33 proteins ranging from insulin to HIV reverse transcriptase, microgravity effects beyond the best Earth-grown crystals were observed to give larger crystals (45.4 percent of the cases), new crystal morphologies (18 percent), at least a 10-percent increase in diffraction intensity (58 percent), less thermal motion (27.2 percent), an x-ray diffraction resolution improvement on the order of 0.0 to 0.3 Å (42.4 percent), 0.3 to 0.5 Å (9.9 percent), and 0.5 to 1.0 Å (9.9 percent). In the improvement of diffraction resolution, a 1 Å improvement can mean the three-dimensional structure can be determined and atomic positions in the macromolecule can be resolved.

While the study of protein structure has traditionally focused on research areas, the applied aspects of designing therapies or diagnostics is now robust. In 1993, the value of shipments of the pharmaceutical industry reached \$69 billion, of which pharmaceutical preparations accounted for 78.4 percent, medicinals and botanicals 10.2 percent, diagnostics 7.6 percent and biologicals 3.8 percent. Industry shipments increased by 1.9 percent in constant dollars to \$48.2 billion. Exports amounted to more than \$7.2 billion in 1993, and imports increased to \$6.7 billion.

As an example of the level of visibility for microgravity protein crystal growth, the startup pharmaceutical company, Vertex, has recently acknowledged NASA in the most bottomline fashion, namely to its stockholders and investors. Their 1995 report reads: "Vertex was founded in 1989 by former Merck employee Joshua Boger, who wanted to design pharmaceuticals at the atomic level but felt his team could not reach its potential at Merck. (Mass screening, the method of traditional chemistry, is the way most drugs have been discovered.) The next year Vertex created a 50/50 joint venture with Chugai, which gave the fledgling company \$30 million in exchange for a cut of future profits associated with developing immunosuppressive compounds. The company's efforts to develop drugs atom by atom are chronicled in "The Billion Dollar Molecule." Vertex kits have ridden the space shuttle in order to form flawless crystals (possible only in zero gravity) from which to blueprint proteins for research efforts. Despite no actual products under its belt, Vertex has made some promising discoveries. Its HIV protease

inhibitors continue to show great promise in fighting AIDS. In 1993, the company acquired an exclusive license for two compounds to potentially treat sickle cell disease and beta thalassemia. Vertex is pursuing the development of a drug to counter multidrug resistance in certain cancers. In 1994, the company announced progress in determining how the body's inflammatory response is activated."

The incentive for pursuing protein structure determination is a better understanding of disease progression. The social costs of American illness and diseases of all kinds are estimated to total more than \$900 billion annually. The advent of new molecular diagnostics and therapies for HIV, cancer screening, and heart disease all remain targets for molecular design of enzymes and proteins. Of this total, the costs associated with a few examples of major illnesses has been assessed as follows. Cancer related illnesses cost the United States \$104 billion/year (according to the American Cancer Society) and among the many protein crystals targeting the various associated ailments are epidermal growth factor, apocrustacyanin C, interferon a-2b, etc. The social costs of diabetes equals \$92 billion/year (according to the American Diabetes Association), and on USML-2 an artificial sweetener called Thaumatin was crystallized in space. The illness of alcohol-related ailments affects more than 18.5 million Americans (according to the AA) and a liver transplant costs more than a quarter million dollars. On USML-2, the enzyme responsible for metabolizing alcohol in the liver, alcohol dehydrogenase, was crystallized in space. A number of HIV-related proteins, including HIV-protease and HIV-reverse transcriptase (RT) have flown in space, with the first RT samples showing an improved internal ordering in the space-grown crystals. HIV is projected to afflict more than 40 million people worldwide with large, long-term care costs. Because of the increasing proportion of the population above 60 years and the long-term nature of care, Alzheimer's disease is often considered one of the major challenges for the next decade. The social costs of Alzheimer's by the year 2015 are projected to reach \$750 billion per year by the American Alzheimer's Foundation. While still very much in the research stages, a number of proteins involved in cell death, such as CcdB protein, are part of ongoing flight experiments, most recently on USML-2 in 1995.

For the future, the U.S. pharmaceutical industry is adjusting to changing market conditions and remains the leader in world industry sector competitiveness and innovation. R&D investment has doubled every 5 years since 1970. In 1993 the industry invested more than \$12.6 billion in R&D, a 14.5 percent increase over 1992. R&D expenditures now represent 16.7 percent of total sales. This is more than double the amount of R&D investment in any other high-technology industry. Recent discoveries, such as a drug that eases the acute pain of migraine headaches and products used to treat Alzheimer's disease, reinforce the industry's belief that R&D investment assures continued growth and success.

U.S. manufacturers account for nearly half of the major pharmaceuticals marketed worldwide. While consistently maintaining a positive trade balance, the industry faces increasing international competition. To maintain competitiveness, the industry must overcome international obstacles such as price controls, illegal use of patents and copyrights, and foreign regulations on marketing and R&D.

## AEROGEL

"Practically all biochemical processes which occur in living beings are proceeding in medium with a sol-gel balance of all components. If fragments of gel phase depend on gravity, it can be an additional way for gravity to influence living beings, including humans."<sup>60</sup> In fact, "gel formation can display very high gravitational dependence. . . The structure of gel matrices obtained on Earth and in orbital conditions has been found to be different. . . space processing of gels could be quite advantageous. . . microgravity conditions can allow gels with a more uniform or prescribed structure."<sup>61</sup>

Beyond this fundamental interest, the commercial gel and sol market is growing rapidly. As *Chemical Engineering Progress* reported,<sup>62</sup> the short-term applications for aerogels feature its remarkable insulation properties (windows, refrigerators, etc.). As early as the 1950's, Monsanto Co.

maintained a large silica aerogel business under the trade name Santocel, which was used as thickening agents, silicone rubber reinforcements, and thermal insulation. However, in the long-term, aerogels “will dominate catalyst manufacture because of their massive surface area, which can serve as host to many chemical reactions.”

According to the “Technology to Watch” section of *Fortune Magazine*,<sup>63</sup> the overall market for the aerogel industry worldwide is projected to include more than 800 potential product lines ranging from surfboards to space satellite components.

The area of biggest growth in the aerogel market is in the area of invisible window insulators. Currently, the market distribution is shared by fewer than five participants, with Aerojet’s Sacramento facility considered a market leader. Other commercial participants include United States, Swedish, and German pilot plants.

For many years, the primary application for silica aerogel has generally been as a transparent or high performance insulator (fig. 7). As *Chemical Engineering Progress* described, “the holy grail of aerogel applications has been developing invisible insulation for use between window panes.”<sup>64</sup> It is our belief that microgravity production of a factor better (R-10 or better) insulating and transparent windows—and its accompanying intellectual property—can develop into a substantial market for residential and commercial applications. The excellent thermal performance and transparent nature of silica aerogel make it an obvious choice for superinsulating windows, skylights, solar collector covers, and specialty windows.

Aerogel is transparent because its microstructure is very small compared to the wavelength of light. However, all but the clearest aerogels scatter some light at the blue end of the spectrum, giving them a slightly hazy appearance. The scattering can be thought of as arising from the large holes or pores that have a lower index of refraction than the average of the aerogel, i.e., index of refraction 1.00 versus 1.02. Thus, research on aerogel preparation to improve its clarity currently is focused on minimizing the number and size of the large pore population in the aerogel (fig. 8).

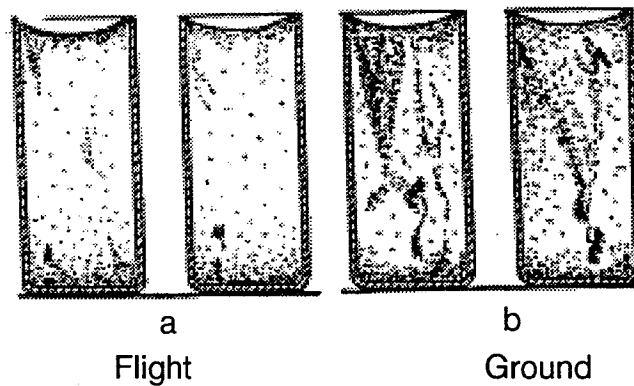


Figure 7. Microheterogeneities.

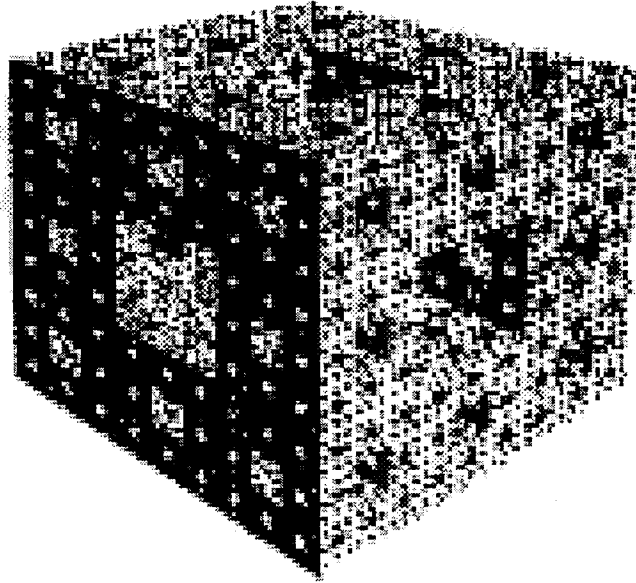


Figure 8. Aerogel.

Although discovered over 60 years ago, aerogels are just becoming commercialized. Within the last decade, a variety of applications have been proposed for aerogels: as superinsulating windows, solar collector covers, and as insulation for refrigerators, water heaters, and pipes. Aerogels might also be used as catalysts for gas-phase reactors, ultra-filters, battery electrodes, acoustic devices, and even as safe insecticides. The "state-of-the-art"/condition of the industry today is such that many metal oxide aerogels can be manufactured by reacting a metal alkoxide with water to form an alcogel, a colloidal suspension of metal oxide particles in alcohol that link together to make an alcogel (gel permeated by alcohol). The alcogel is then supercritically dried to produce aerogel. A development at Lawrence Berkeley Laboratory in Berkeley, CA, permits reduction of the temperature and pressure required for supercritical drying by substituting, under pressure, liquid carbon dioxide for the alcohol in the gel and then supercritically drying the aerogel with carbon dioxide. The process results in reducing the temperature and pressure required to dry aerogels and providing a safe method of manufacture.

### **Microgravity Objectives**

To perform parallel observations on gel growth in both ground and microgravity (aircraft based or long-term space based) experiments, the following objectives are identified: (1) determine the time evolution of silica particle aggregate size and geometry during gellation experiments of various durations by means of multi-angle light scattering; (2) visualization of convective flows during gellation by Schlieren or Mach-Zehnder interferometer; (3) determine the effects of microgravity on pore size distribution in supercritically dried gels (aerogels) by angle-scanning laser light scattering, nitrogen adsorption, gas pressure dependent thermal conductivity measurements, and high resolution electron microscopy; (4) control growth of gels with uniform distribution of particles added in long-term microgravity experiments in order to confer specific properties to the gel; and (5) improve the properties of aerogel materials through better understanding of the condensation and aggregation of colloidal systems.



## **Product Aims**

While silica aerogel is relatively clear, its transparency needs further improvement to become acceptable for window and optical applications. The cause of distortion seen when viewing scenes through aerogel arises from variations in the average refractive index. This feature is frozen into the gel at the time of gellation. These index variations arise from convective flow caused by the heat released during hydrolysis of the alkoxide. These convective forces evidently affect the concentration of the ester precursors of the sol before gellation. Understanding the origin and reducing the magnitude of these index variations are the second set of issues to be studied under microgravity conditions. Another issue to be addressed is the role of diffusion on the condensation phase of the sol-gel process.

“It is evident from the space-processed material that the process of formation of three-dimensional gel is very sensitive to gravity. This sensitivity is displayed both at the macro- and micro-scale structure. . . Weightlessness gives the opportunity to modify the process of formation of three-dimensional gels.”<sup>65</sup>

Aerogel commercial processing in space is undertaken to reach a quiescent environment for more uniform gelations. On Earth, solutal flows are thought to play a role in gel formation as early as the aggregation stage when clusters of molecules of less than critical size are formed. Sol-gel processing depends critically on the formation of aggregates of sol particles. The contact between particles after they collide is maintained by Van der Waals forces and becomes irreversible when chemical bonds form between the particles. One main goal for the Aerojet effort, therefore, is to find space-based methods to reduce the number of large pores in gel structure, which are responsible for most of the light scattering in aerogel, thus diminishing the optical qualities of existing terrestrial products. Another goal is to understand the formation of larger-scale heat-driven convective structures of varying density that are frozen into the material at gellation.

## **Present Commercial Partners**

A variety of silica and organically based products are currently in the commercialization and evaluation stage with:

- Maytag Refrigeration Products, Newton, IA, makers of home appliances, and Glacier Bay Co., Belmont, CA, manufacturers of marine refrigerators, will evaluate environmentally safe aerogel insulation.
- General Motors Cadillac Division, Detroit, MI, will evaluate aerogels of thermal and acoustic insulation in door panels, car ceilings, and under the hood.
- Benteler Industries, Detroit, MI, will look at aerogels for use in the exhaust manifolds it manufactures. It hopes they will improve catalytic converter efficiency.
- Boeing Commercial Aircraft, Seattle, WA, will evaluate aerogel use for aircraft applications. Silica aerogels have been certified as fireproof.

## **Unique Microgravity Features for Aerogel**

The microgravity processing elements to be evaluated are: (1) that microgravity affects formation of highly uniform density of aggregates by reducing convection flows; (2) that the reduction of gravitational forces greatly reduces the sedimentation of growing aggregates of silica, thus reducing the creation of large pores during gellation; (3) that heat-driven convective forces that cause large-scale density variations can be reduced by understanding and controlling their origin; and (4) that the

microgravity environment can be used to uniformly distribute particles (e.g., metals) in the sol to give the gel better structural properties or impart specific catalytic properties.

These four processing elements have been tested in a limited fashion on a number of Russian flights as early as 1991. Technical results were preliminary but postflight analysis revealed a number of promising results related to a more uniform pore distribution in polyacrilimide gels. No current transparent aerogel exists terrestrially and microgravity provides the most promising avenue for producing the first truly invisible insulators.

### **Results of Flat Glass and Insulation Market Survey**

The aerogel industry has long targeted the marketing of invisible insulation, taking advantage of the need for energy-efficient window panes and the strong insulating properties of a high internal surface area product like aerogel. Two microgravity products and their corresponding markets are summarized below in premium flat glass industries and specialty insulators.

#### **Premium Construction Materials—Flat Glass**

The flat glass industry is a \$2 to \$3 billion industry with more than 14,000 U.S. employees. A 1- to 5-percent market penetration for aerogel insulating panels sandwiched between two glass window panes amounts to several tens of millions of dollars. The flat glass industry is made up of companies (including PPG Industries and Pilkington PLC) that make "float glass" (unfabricated flat glass) and various products made from it, including window glass, cathedral glass, picture glass, laminated glass, motor vehicle windshields and windows, skylight glass, and tempered glass. Because of the extremely high cost of constructing a plant to make float glass, it is produced by only six companies. One of these is a "captive plant" producing only for its own consumption. However, the relatively low capital requirements for fabricating float glass enable many firms to enter this segment of the industry.

The major user of flat glass products is the construction industry, which consumes approximately 57 percent of the output of the glass industry. The other major consumer is the automotive industry, which accounts for roughly 25 percent. The remainder is taken by producers of "specialty products," including mirrors, solar panels, and advertising signs.

Between 1987 and 1992, shipments of unfabricated float glass reached historically high levels, averaging 4.4 billion square feet annually. This compares with an average annual output of only 3.1 billion square feet for the previous 6 years (1981 to 1986). Overall, the industry (including fabricators of float glass) has not displayed dynamic growth, but float glass production continues to increase. Shipments rose from 4.3 billion square feet in 1991 to 4.6 billion square feet in 1992. The major reason for this high level of shipments is that about 22 percent of output is exported and the export market for flat glass products has been very strong in recent years (U.S. Department of Commerce: Bureau of the Census; International Trade Administration (ITA). Estimates and forecasts by ITA.).

Prices of flat glass and flat glass products fell each year from 1988 until 1992. However, the decline was very small—less than 1 percent from 1991 to 1992, compared with 2 to 6 percent in previous years. During the first part of 1993, prices rose slightly (2 percent) compared with 1992. Nevertheless, it is expected that prices will remain about the same, with possible minor downward adjustments, as manufacturers engage in strong price competition to increase gross sales and retain market share.

## **International Competitiveness**

Beginning in 1992, the flat glass industry shifted its emphasis from the U.S. market to foreign markets. No longer a totally U.S.-based industry, today only three of the five U.S. float glass producers are U.S.-owned. The other two are owned outright by foreign interests (AFG Industries and Libbey-Owens-Ford).

U.S. companies are expanding significantly into foreign markets, usually by establishing plants. For example, in April 1993, Guardian Industries Corp., which operates facilities in South America, Western and Eastern Europe, and other parts of Asia, opened its newest foreign float glass plant in Thailand. Also, PPG Industries recently joined a Japanese flat glass producer and three other partners to establish a major float glass plant in China.

Foreign trade plays an important role, and a favorable balance of trade has developed for U.S. producers. Beginning in 1990, an unfavorable balance of trade over the previous decade was reversed as the value of exports exceeded the value of imports by \$168 million. Exports in 1990 totaled \$665 million, compared with only \$497 million in 1989. Both years represent considerable gains over 1988 and 1987, when exports were \$416 million and \$356 million, respectively.

The trend toward larger export volume continues, with an increase to \$692 million in 1991 and \$723 million in 1992. The export pace during the first part of 1993 was exceeding that of 1992 by 18 percent. Major export markets from 1989 to 1992 were Canada (49 percent), Mexico (10 percent), and Japan (9 percent).

Imports may have reached a peak of \$535 million in 1992, up from earlier levels in 1990 to 1992. Import performance in 1990 to 1992 reflects foreign exchange rates, a great deal of the decline is attributable to the recession in the U.S. construction industry.

The leading countries exporting to the United States from 1989 through 1992 were Canada (36 percent), Mexico (22 percent), Japan (12 percent), and Germany (5 percent).

## **Near-Term Prospects for Aerogel in Flat Glass**

For all products, the industry will continue to work on developing new products to increase sales in the construction and motor vehicle markets. Further advances are expected in two important competing products: switchable glass, in which the opacity is changed by electronic and other means; and in energy-conserving low-emissivity glass.

Foreign trade will continue to be a major marketing focus both for producers of float glass and manufacturers of flat glass products. After nearly doubling export sales from 1989 to 1993, it appears likely that the U.S. industry will continue to emphasize exports as part of its overall marketing plans.

## **Specialty Insulation Market**

Aerogel has a five-fold advantage over other insulating materials including foams, beads, and certain vacuum dewars. The existing aerogel market in refrigerants is the high-quality end for premium applications, particularly where chiller space is limited such as in yacht refrigerators. A general market survey is summarized below including historical data and recent legislative mandates including the national interest in energy conservation and environmental issues related to existing chlorofluorocarbons (CFC's).

## **Household Consumer Durables—Refrigerators**

Product shipments of appliances increased 3 percent in real terms in 1993, to a record \$17.7 billion in current dollars. The appliance industry is dominated by five major corporations that produce complete lines of basic, major household appliances: Whirlpool, General Electric, White Consolidated Industries (Electrolux), Maytag, and Raytheon, in that order. State-of-the-art major appliances of high quality are offered at low prices because of intense competition among well-capitalized companies, high volume production, heavy capital investment, and a market open to foreign producers. Imports constitute more than 50 percent of the domestic market in several categories of small appliances, because of the high labor content of these appliances. The major U.S. producers are now moving to become global manufacturers. Most have plants in western Europe, and some have begun to expand into Central and Eastern Europe and China. U.S. production employs 107,000 workers, 80 percent of which are production workers (U.S. Department of Commerce: Bureau of the Census; International Trade Administration (ITA). Estimates and forecasts by ITA).

## **National Commercial Interests in Better Insulation**

The National Appliance Energy Conservation Act of 1987 set national efficiency standards for several categories of major household appliances, including refrigerators. The standards for refrigerators became effective in 1993, and the others were scheduled to take effect in May 1994. The 1994 standards can be met through several changes including more efficient motors and better insulation. In September 1993, DOE also published advance notice of a new round of standards rule-making for refrigerators, as well as for furnaces and central air conditioners. The new standards would not become effective before 1998.

## **Environmental Profile for Insulation Improvements**

Energy efficiency and CFC's remain major issues for the appliance industry. For more than 50 years, CFC's have been used as coolants in refrigerators and freezers, as well as in the production of foam insulation. However, because CFC's allegedly damage the Earth's protective layer of atmospheric ozone, the United States has pledged to halt CFC production by 1996 under the Montreal Protocol of 1987. In addition, DuPont, a major U.S. producer, has stated that it would end CFC production by the end of 1994. This accelerated phaseout places more pressure on manufacturers to finish testing substitutes, solve problems in material compatibility and toxicity, and meet energy-efficiency requirements. The use of an alternative foaming agent, HCFC-141b, is likely to be only a temporary substitute, since U.S. production is expected to be phased out by 2003.

Some appliance manufacturers in Europe are already offering CFC-free refrigerators and freezers, most using the mentioned substitutes. Early 1993 models were priced about 7 percent higher than those with CFC's, however. A U.S. manufacturer may soon use high-insulation vacuum panels in place of foam insulation. The latest versions of aerogel panels can reach insulation factors of R-32 or upwards, far higher than the R-8 achieved with foam produced with CFC-11 or the R-7.5 with HCFC-141b. If the use of these aerogel-vacuum panels proves to be technologically feasible and cost-competitive, it could result in a substantial increase in a refrigerator's interior space while substantially contributing to energy efficiency. The panels might also be used in other appliances, such as water heaters.

## **International Competitiveness and U.S. Marketing Strategies**

Appliance imports and exports increased at nearly the same rate in 1993, imports about 7 percent to \$4.1 billion and exports 6 percent to \$2.5 billion. The leading suppliers of appliances to the United States were Mexico, China, Japan, South Korea, and Taiwan, in that order (*Dealerscope Merchandising Magazine*, North American Publishing Co., 401 North Broad St., Philadelphia, PA 19108). The leading

markets for U.S. appliances are Canada, Mexico, Japan, Germany, and Saudi Arabia, in that order. Exports to Canada have more than doubled in the past four years as tariffs declined under the United States-Canada Free Trade Agreement, under which all Canadian tariffs on U.S. appliances will be eliminated by 1998. Likewise, U.S. appliance exports to Mexico have nearly doubled in the past four years because of major tariff reductions by Mexico and increasing shipments of parts to U.S.-affiliated appliance factories in Mexico.

In the areas of specialty insulators for refrigerators and flat panes, a microgravity advantage in production of 1 to 5 percent represents a new market in the tens of millions of dollars. To investigate and market whatever niche can be defined for a more transparent product will be developed within these scenarios.

## FUNDAMENTAL SCIENCE

A number of investigations address the next generation of microgravity experiments. A few representative examples will be included: (1) laser cooling of single atoms, and (2) lambda point measurement for the heat capacity of liquid helium.

### Atom Trapping in Microgravity

Ramsey won the 1989 Nobel Prize for related breakthroughs in atomic trapping. Atoms are electrically neutral; hence, unlike ions, one cannot trap them by using electrical fields. The simplest trap involves three sets of laser beams, oriented respectively to define x, y, and z axes and intersecting within a small region of space. These lasers are tuned to just below the frequency of a strong atomic spectral line. An atom in motion, in whatever direction, will absorb photons more effectively and, hence, will experience a drag force that acts to slow it down. The atom then behaves as if it were held within a viscous fluid, sometimes called "the optical molasses." As temperatures move toward absolute zero and all thermal motion begins to cease, gravity becomes a dominant influence.<sup>67</sup>

In the absence of frequent atomic collisions and some heat source (either as heated walls or high velocity atomic neighbors), a lone atom undergoes free-fall at  $9.8 \text{ m s}^{-2}$ . With this acceleration, an ultra-cold atom (cooled to 2.5 micro K) has a thermal velocity of only  $12 \text{ mm s}^{-1}$ . Nevertheless, after 1 s in a unit gravity field, this atom has dropped 5 m and its velocity has increased by  $\sim 10^3$ . For typical observation chambers ( $\sim 1 \text{ cm}^3$ ), a 5-m dropping distance effectively limits trapping times to milliseconds or less.

Recent experiments in reduced gravity have confirmed this low transit time effect. Lounis and co-workers (*Acad. Sci. Paris*, 316 (1993) 739) note that "for free particles, the Earth's gravity imposes a severe limit on this atomic observation time and on the usefulness of the lowest achieved temperatures." Their observations indicated that in reduced gravity ( $a = 10^{-2} g$ ), atomic confinement times can now extend to 6,000 times their unit gravity counterparts. This finding has opened microgravity research to include the atomic-scale physics that now underpins quantum mechanics and relativity theories.

The great precision of these experiments makes possible a new generation of time and frequency standards with considerable improvements in accuracy. The present time standard is an atomic clock that relies on cesium atoms. It keeps time with an error of one part in 1,013, corresponding to a few-thousandths of a second over a human lifetime. Beyond this accuracy, the resolution of microgravity atomic clocks may reach a thousand- to a million-fold improvement. This advance makes it possible to determine if the constants of physics may be changing slowly with time. These constants include the speed of light, the charge of the electron, and Planck's constant in quantum mechanics. As an example, by atomic trapping experiments that confine atoms featuring two different physical principles (nuclear

versus electromagnetic forces), the two very precise clocks would begin to “tick” at different rates if, for example, the strength of the electromagnetic force changed relative to the fundamental nuclear force.

By using atomic interferometry based on these principles, Chu et al.<sup>68</sup> has shown that a gravity sensing accelerometer can be designed with an accuracy of 3 parts in  $10^8$ . This atomic standard for gravity measurements has already received attention as a sensor for detecting geological anomalies in local gravity associated with large petroleum reserves. These improvements have, therefore, been suggested for mapping the distribution of natural resources from gravity changes.

### **Lambda Point for Liquid Helium**

The 1993 lambda point experiment<sup>69</sup> involved the best measurement of the heat capacity of liquid helium as it changes phase from the superfluid to the normal fluid phase to within  $10^{-9}$  K of the transition temperature. The objective of the experiment was to rigorously test the range of validity of Wilson’s Nobel Prize renormalization group theory as it pertains to critical phase transitions. Hydrostatic pressure effects, caused by gravity, result in increased smearing of the data as the critical point is approached. During flight, in the absence of hydrostatic pressure effects, over a ten-fold improvement in resolution could be achieved. Overall, the analysis of the results show the potential for yielding by far the most definitive test of renormalization group theory that has ever been performed. In space, the measurable lambda point may be 10,000 times better defined than the best current Earth measurement.

### **ASPECTS UNRELATED TO TECHNOLOGY**

Based on these new opportunities, surveys of public opinion continue to point to a large constituency for exploration without perceived technological spinoffs. The independent polling by Yankelovich Partners (1994) found that “three-quarters of those surveyed favor a continued human presence in space and believe that we should embark on joint space ventures with other nations. The majority say expand current U.S. space activities and more than half favor a return to the Moon and manned voyages to Mars.”

A number of factors have been pointed to as underlying this popular support: international, educational, and the comparatively minor costs of space exploration relative to other Federal outlays. These three issues in turn will be described briefly.

The first agenda is international cooperation and competition. Increasingly, the presence of international cooperation and economic competition has encouraged each nation’s commitment and financial participation. The *International Space Station* is multinational, both encouraging a sharing of resources and results, while at the same time creating an environment of friendly competition.

The second agenda is educational. Space exploration has always been a focus for capturing students’ imagination and a high-profile arena for encouraging their education. Therefore, in addition to the space program’s role in fostering international cooperation, the growing sentiment that American education standards are not consistent with global standards—particularly in science—has driven a reevaluation of curriculum in science and mathematics. The direct consequences of incomplete curriculum in our schools is born out in the declining intelligence scores of America’s youth: (1) from 1963 to 1980, the proportion of all 17-year-olds scoring 700-plus verbal and math SAT’s fell by almost half; (2) when compared with their peers in 14 other countries, American 13-year-olds rank next to last in math and just one notch better than that in science; (3) among the 100 fifth graders who scored highest on international educational tests, 88 percent were Japanese and 1 percent was American. Despite this, Japan spends 45 percent less on grade schools than the United States.

The saving grace internationally for the American educational system has been that whatever deficits might survive grade school, the strength of the American university system has provided sufficient coverage. However, as Norm Augustine pointed out, this sense of making up lost ground later has diminished: (1) since 1986, the United States has graduated 14,000 fewer engineers, or a 19 percent drop, compared to previous graduations; and (2) a U.S. scientist starts at half the salary for a starting lawyer and two-thirds less than for a starting business school graduate, but with a longer educational investment in science graduate school. While the role of the space program in redeeming these deficiencies remains abstract, the exploration agenda bears a direct relation to student awareness of opportunities in science and engineering.

The final issue driving public support of the space program can be identified as the comparatively low costs in the Federal budget—less than 1 percent of total outlays since 1977. A commonly repeated theme is that the space program has little or no effect on present budget concerns (fig. 9). The Health and Human Services Agency, for example, spends the entire NASA budget every 7 days; the Defense Department spends the same every 18 days. If entirely ellipsed from the accounting sheet, NASA would represent less than 1 day's difference in calculating the interest alone on the Federal deficit; no progress would be made on the deficit principal at all. In other words, the difference in a leap year and a non-leap year in the interest would equal the entire space program's budget. Willard Rockwell<sup>70</sup> put this comparative argument succinctly: "It's amazing when you realize that our space budget today is less than one-third of what we spend on farm subsidies alone. . . less than a third."

An interesting feature of the public support for space exploration is that when the public is surveyed about spending, the NASA budget is considered equivalent in the public's perception to the entire Defense Department budget, of which in reality the space program is a mere fraction. Without entering into the political, regional, and fiscal complexities of how budget priorities are set, it is worth considering an illustrative case in point. In the 1996 budget, the Pentagon turned down a Congressional proposal to purchase \$10 billion worth of B-2 bombers, saying that the DOD neither wanted to nor could maintain and operate the expensive planes. However, Congress approved the purchase despite Pentagon recommendations, thus essentially buying "graveyard" bombers. The cost of these planes, recommended against by the Pentagon itself, would amount to nearly the entire U.S. expenditure on the space program. The remarkable part of the comparison, however, is not the complex issue of budget priorities, but rather despite all public sense that NASA and DOD spend dollar for dollar on an equivalent basis, nearly 75 percent of those surveyed continue to endorse an enlargement of the space program beyond its present support.

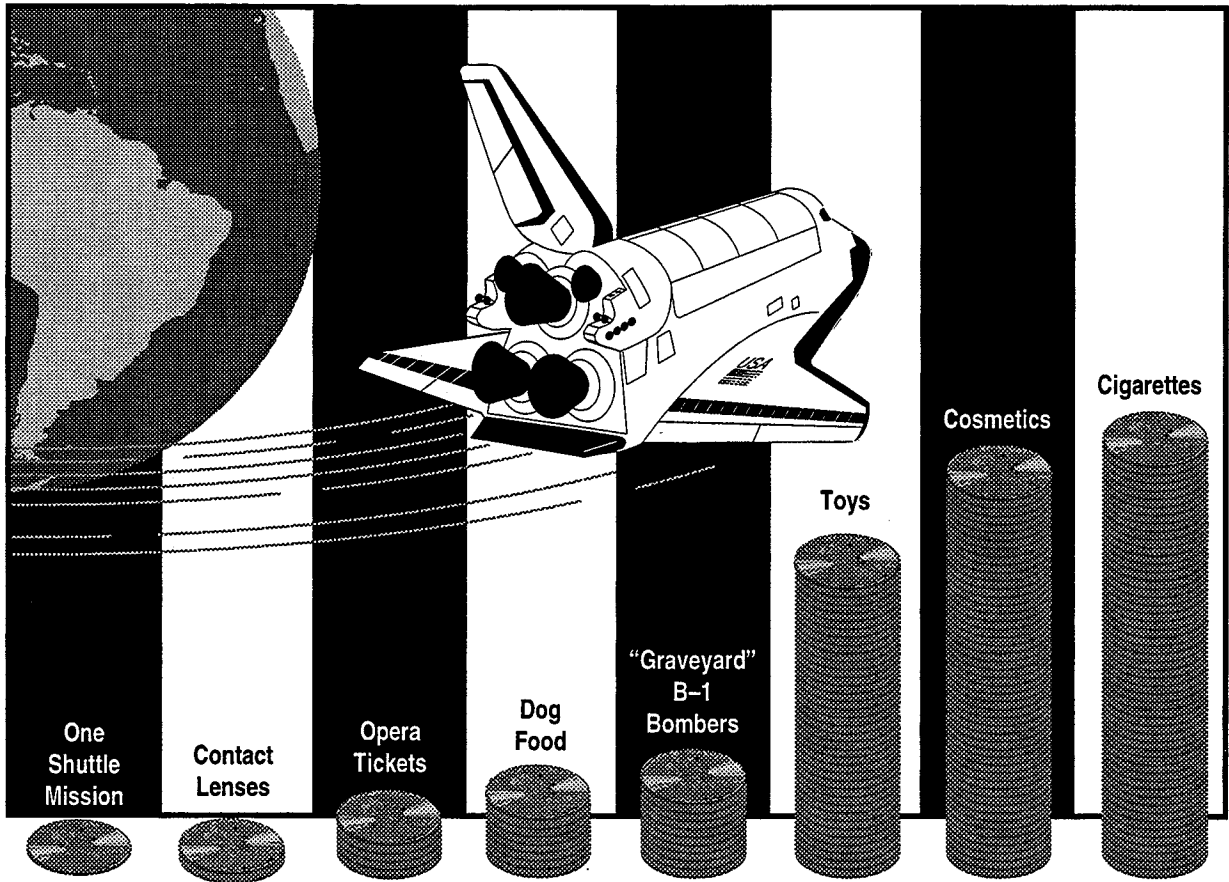


Figure 9. Counting pennies in the consumer spending market.



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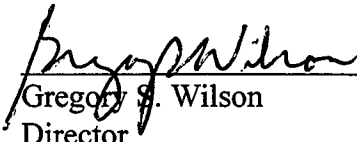
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**APPROVAL**

**TECHNOLOGY THRESHOLDS FOR MICROGRAVITY: STATUS AND PROSPECTS**

By: D. Noever

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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