Advanced Composites for Structures

George P. Peterson*
Wright-Patterson Air Force Base, Ohio

The Air Force Advanced Development program on high-modulus filaments and composites involves a comprehensive and integrated effort; it involves not only materials development but simultaneous and heavy emphasis on design and fabrication. It includes, as an essential ingredient, the conclusive demonstration of the value of the technology in the product form, which are major advanced composite structural components. The attack on the most rapid and efficient technique for development of the technology is organized along three vertically integrated major lines. These are: reinforcement development, composite materials development, and component development. The brain center of the program is component development, where (by actual design, fabrication, and test of composite materials hardware) direction and emphasis will be given to the filament and composite development efforts. The program areas of primary interest, although concentrating initially on continuous boron and glass filaments with organic matrices, will include other reinforcement materials, reinforcement forms, metallic and inorganic matrices, etc. as dictated by their applicability and importance to the highest payoff composite structural components. The total advanced development technical program, including status and progress, is briefly discussed; and the perspective with related exploratory and manufacturing development activities is treated.

A REVIEW of the status of the development of structural composite materials would indicate that massive strides still must be taken to develop and realize the full potential of these materials in primary aerospace vehicle structures. Certainly, there has been substantial progress over the past 20 years in projecting structural composites into several key Air Force end item applications. This progress has been, to a great degree, spearheaded by the usage of glass reinforced organic composites. These composites have excellent, and even improving, strength-to-weight ratios that are a factor of great importance to the aerospace structural designer. Some of the applications currently utilizing glass components include radome structures, helicopter rotor blades, and high performance rocket motor cases. These applications, however, do not constitute the large volume Air Force structural materials market. Additionally, the progress, although substantial, has been painfully slow; and in many of the major United States hardware development concerns, there is a lack of confidence in extending the usage spectrum of these anisotropic materials. There are several primary reasons for this attitude which are definitely interrelated. Glass reinforced composites have a low modulus of elasticity (and a relatively low modulus-to-weight ratio), and the glass compositions known or being researched, at present, offer little hope for massive improvements. This point is demonstrated graphically when the modulus of glass composites is compared to homogeneous materials such as titanium, steel, and, certainly, beryllium. Analysis of the major Air Force structural applications has indicated a definite requirement for high modulus in addition to high strength-to-weight characteristics. This lack of an essential materials characteristic has resulted in a less than enthusiastic drive for a sophistication in the essential design and fabrication technology areas. These latter two areas are absolutely essential to projecting structural composites into any usable end item applications. An example of their impact is the advent of the filament winding fabrication techniques and isotensoid design concepts which made practical the operational usage of glass reinforced rocket motor cases. It is safe to say that without filament, winding Minute Man or Polaris would not be using structural composite material rocket motor cases today. There is no doubt that any meaningful development of composite materials for structures must include concurrent and strong efforts to develop new design concepts and fabrication procedures which are tailored to meet the needs of the structural components.

It was in this environment that the Air Force Materials Laboratory in 1961 initiated substantial and concerted exploratory development efforts to obtain filamentary materials with high modulus in addition to high strength and low density. This attack was primarily oriented along materials development lines and was almost immediately successful in the form of continuous chemical vapor deposition boron filaments with high strength (approaching that of glass filaments), the density of glass filaments, and a modulus of elasticity of 60,000,000 psi. Initial attempts to put these filaments into organic composites were successful. The data obtained reflected the high modulus, high strength, and low density of the boron reinforcements. This breakthrough in the materials area provided the impetus for numerous paper analyses on the potential impact of such advanced composite materials on a broad spectrum of primary aerospace structural components. The analyses indicated that massive improvements in regard to high performance lightweight structures were theoretically possible in all application areas studied. These have included airborne structures, aircraft engines, space and re-entry vehicles, rocket propulsion hardware, and helicopter rotor blades and propellers. The analyses were conducted by Air Force teams, various industry hardware development concerns, and special ad hoc activities. A general summary of the results indicates that weight reductions from 10–50% are possible depending on the particular application involved and the assumptions made. Additionally, there are benefits beyond weight savings which are possible to the structural designer and weapons system designer because of the combined high strength, high modulus, and low density characteristics of advanced composites. These benefits center largely on a relief of constraints on the designer which force him to make severe design compromises or leave him with impossible solutions to meet system operational requirements. One example is high-strength, thin-wall rocket motor cases that can withstand the sequential and/or combined loadings involved in high chamber pressure operation, rapid launch accelera-

Presented at the AIAA/RAeS/JSASS Aircraft Design and Technology Meeting, November 15–18, 1965, Los Angeles, Calif.; revision received April 29, 1966.

* Chief, Advanced Filaments and Composites Division, Air Force Materials Laboratory.

tions, and the quick turns necessary for rapid maneuvering operations and that can still maintain the light weight of filament wound structures. It must be pointed out that the analyses conducted had a minimum of design data to draw on and that in the materials, design, and fabrication areas many assumptions and extrapolations were made. Technological advances to convert these assumptions to practical reality clearly form the basis for efforts to project advanced composite materials into actual structural applications. It would be naive to expect realization of the full potential in even a single application area. This seldom occurs, but, certainly, the overwhelming potential of these materials warrants comprehensive effort to rapidly develop and exploit the technology. At any point in time, the scorecard on the success of technological development activity will be the assessment of the performance of actual structural component hardware. It was apparent from the studies, however, that even marginal development success could have dramatic impact on aerospace structures. They also defined the major development areas to be attacked and categorized them into three primary areas: materials, design, and fabrication.

The primary motivating force behind the current Air Force advanced development program is to project improved structural composite components into actual operational applications. This program rests on a base of continuing exploratory development and manufacturing technology efforts to develop new reinforcements, matrix materials, fabrication techniques, and design concepts. It is from these areas that the advances, which will result in continuous improvement in the performance of advanced composite structures, will appear first. Prime examples are boron filaments that exploded out of exploratory development and, presently with glass filaments, form the initial reinforcement basis for the advanced development program. Other reinforcements and materials are being investigated vigorously, and these will be interjected into advanced composite structures as they are developed and required. Some of the most promising of these will be discussed later. The advanced development program essentially provides the bridge from exploratory development to actual systems applications. The program concept is based heavily on an aggressive and dynamic component development function. It is the brain center of the entire program. All activity within the program will be vectored to support the development of actual advanced composite componentry. With this approach, it rapidly becomes evident that materials development is only part of the picture. Design and fabrication technological progress are vital factors. Their progress must be geared and oriented to the available materials technology, and all three technologies must be clearly responsive to the needs of the structural components. Analysis has indicated that different components will require different materials, design, and fabrication solutions. There will be no one over-all solution for all applications areas. Indeed, the most satisfactory solutions even within an application area probably will vary to a considerable extent. The simultaneous attack of the materials, design, and fabrication problems will be highlighted by design, fabrication, and test of actual structural components. These components will be graphic demonstrations of the technological progress, the major problems still to be solved, and ultimately the value and and payoff of the technology. They also will serve continuously and most rapidly to build hardware-oriented industry experience and confidence levels in the use and value of the technology in the form of the products they market.

A review of the organization of the program effort indicates that there are three primary elements of the program:

1) The component development area includes component demonstration, component-oriented technological building block solutions, and the development of new structural design concepts for components. The component demonstration area will involve a concentrated effort to develop and demonstrate conclusively the value of the technology for major structural components. This area will involve the development of materials, design, and fabrication technology for the particular hardware item of interest. The area also will reflect and utilize the materials, design, and fabrication advances made in other portions of the program. The components selected for full development will reflect the highest payoff areas and those that will represent most graphically the technological progress achieved. It will not be practical or feasible to demonstrate fully, concurrently, the technology in all application areas of potential interest. Many of these areas will have substantial problems that must be resolved prior to a full demonstration phase. In these areas, the individual problems will be attacked to obtain building block solutions that will then provide the basis for downstream full-scale component development. The area of new design concept generation is one of the most essential activities. New design concepts that are tailored specially to the available materials capabilities, available fabrication procedures, and the functional requirements of the component must be developed. Advances in the design area will aid in a realization of the full potential of the materials and will minimize some of the disadvantages of both the materials and fabrication processes. One of the major areas, for example, involves multidirectional loads. It is painfully clear that structures having complex loading situations impose severe limitations on structural composites. These multidirectional loading situations may not have been a critical factor with homogeneous materials. They are of great consequence to advanced composite materials, because they reduce the extermely high strength and modulus characteristics available in unidirectional composite structures. Every step that the designer can take either to reach or approach unidirectional structures is a vital one in regard to improved performance. We must force the designers in this country to exercise their ingenuity and to help themselves to the design of vastly improved structures. The directional characteristics of composites are a tremendous advantage only if design concepts will support and make possible the use of the most effective composite structures. The attack in this area will be to project a strong component orientation to the mechanics and micromechanics efforts. This will be highlighted by a substantial effort to close the loop and utilize experimental and theoretical mechanics efforts to form the basis for the evolution of design advances for these components.

2) The filament development area primarily attacks a basic materials development problem and initially reflects a heavy concentration of effort on development of improved processes for the preparation of boron filaments. There will be a continuing attack on development of low cost, high production rate, high quality filaments. Chemical vapor deposition, direct melt spinning, and other techniques such as corona discharge and dip coating are processes that will continue to receive attention. It must be stressed, however, that boron filaments in continuous form are only one type of reinforcement material. New reinforcement materials such as carbon, silicon carbide, boron carbide, and others presently are being researched actively; and scaled up process development activity with the most advanced of these materials is projected for the near future. The impact of these materials on structural component development efforts will be determined by the needs of the component and the success in research efforts advancing these materials to the point where they can be projected realistically into the composite engineering and component development activities. Additionally, the picture is the same for new reinforcement forms that might include short filaments, whiskers, ribbons, flakes, etc. These reinforcement forms, although not presently in as advanced a development stage as continuous filaments, may well (via research advances) have substantial impact on downstream advanced composite structural components. The filament development activity is also the source of all advanced filament supply for the entire program effort.

Initially, this primarily involves boron filaments prepared using vapor deposition techniques. Downstream, other processes and other materials and reinforcement forms may be utilized. Sufficient filament to conduct all advanced development component and composite activities, along with supporting the needs of exploratory development and manufacturing technology activities, must be supplied in a timely manner. Additionally, filament will be generated to furnish industry to stimulate and support their in-house and Internal Research & Development efforts. This selective furnishing of materials will help to build and maintain a broad base of industry interest, participation, and responsiveness.

3) The composite materials development area is the next step beyond the filament development area and must lay the groundwork for bridging the technological gaps leading to successful component development. It primarily involves materials development, composite materials design, and the development of fabrication procedures for advanced composites. A key element of these activities involves matrix material and filament treatment development. This is necessary to realize the full potential of composite materials performance for the widest range of environmental and load bearing conditions. These conditions will be defined so that they will be responsive to the requirements of the particular structural component items being developed in the program. The early emphasis will be on extending the capability of organic-type matrix materials to utilize past experience and processing and fabrication advantages. Immediate goals are a better match of filament and matrix properties, improved shear strength, and higher temperature capability. Metallic matrix composites, which are in an early stage of development, show great promise and offer increased temperature, shear, and attachment capabilities. Metals of interest include aluminum, magnesium, titanium, and nickel among others. Filament surface treatments to improve adhesion between high-modulus filaments and organic matrices and diffusion barrier coatings for metal matrices are another area closely oriented to matrix material development. Guidance for determining the reinforcement and matrix materials required will be obtained from micromechanics efforts aimed at clarifying the specific role that composite constituents play in determining ultimate composite performance.

Fabrication techniques are a major factor in determination of the final cost and have a pronounced effect upon the performance and integrity of the structural component. Fabrication procedures oriented to the requirements of the particular components of interest must be developed. These procedures should provide for minimum constraint on the materials and design solutions and must be simple, reliable, low in cost, and reproducible. Initial emphasis will be placed on efforts leading to revolutionary new techniques for organic matrix composites. These techniques must be capable of handling boron and glass filament initially and new filaments such as carbon and silicon carbide in the future. Preimpregnated and collimated reinforcement packages must be developed. These should be capable of accommodating several filament materials in selected volumes and orientations (including three-dimensional orientations). The guidance for this development must be the compatibility with the fabrication process, which is responsive in turn to the requirements of the end item component. Metallic matrix composite fabrication procedures and reinforcement packages will be an area of increasing activity as appropriate materials and fabrication fundamentals and concepts are defined more clearly. The over-all purpose of the entire fabrication activity is to predict and develop the advanced fabrication techniques so that the component development efforts will have available proven and constantly improving fabrication concepts.

The engineering data efforts are oriented toward development of the most meaningful design and engineering data for the structural designer. A prerequisite is the development of valid test procedures which convey the information necessary for the design of component structures. The data base generated will reflect closely the materials configurations and properties of greatest interest to the component structural designer. This base of data will be supplemented by specialized tests to determine characteristics such as thermophysical, electrical and environmental (such as rain erosion) properties. Effort is planned for development of nondestructive test procedures and techniques for effective collection, collation, analysis, and finally dissemination of all the data generated to the aerospace industrial community.

There has been substantial technological progress in the advanced composites area over the past year. There is no technological plateau and there has been a continuous, increasing stream of technological advances. A brief assessment of some of the highlights of the advances made would touch on the three primary program areas: filament development, composite development, and component development areas.

In the area of filament development, there are presently onstream at Texaco Experiment Inc., a bank of 10 chemical vapor deposition units. These units are running on a three shift per day basis and currently are preparing filament at an 85 lb/mo. rate. This filament utilizes a $\frac{1}{2}$-mil-tungsten core as a substrate and is 4 mils in diameter. The filament is averaging 325,000-psi tensile strength and 60,000,000-psi modulus of elasticity. The strength distributions, which have been the major characteristic showing a wide variation, have densified increasingly in the 300,000-to 500,000-psi category. Additionally, the lower strength filament quality and handleability have been upgraded greatly by a hot nitric acid etching process that will be discussed later in more detail. Practically all filament reflect a 60,000,000-psi modulus of elasticity. The increased volume preparation of high modulus filament and the improved processing procedures and equipment, along with the vital onstream experience factor, have all contributed to a major reduction in the present cost of boron filaments. As-formed filament materials cost, in the approximately 1,000-lb quantity to be prepared over the next year, is currently estimated at \$850/lb. This is a substantial reduction from the \$6,000/lb in the initial laboratory units poundage quantities prepared and the \$3,000/lb cost of the filament obtained from initial operation of the 10 units. It can be projected realistically that the completed development of new crossflow units designed to increase deposition rates from the present 0.03 mils/sec to 0.30 mils/sec will reduce the cost of chemical vapor deposition boron filaments to \$250/lb, even with the expensive tungsten substrate. These units differ from the present process primarily in the direction of the reactant gas flow from "parallel to the moving filament" to "perpendicular to the moving filament". This process is in the final development stages, and initial units should be in operation within the next year. Additionally, combined crossflow-low-pressure units utilizing boron hydride as the source gas are in an earlier stage of development and should reduce costs to \$100 to \$150/lb. This process has been demonstrated in the laboratory and could achieve deposition rates of 1.0 mil/sec. It utilizes the hydride reactant gas and a tungsten substrate. A further advance that could replace tungsten with a coated silica or glass filament substrate has the potential to reduce the cost to \$20 to \$30/lb even with low yearly (10,000 lb) filament preparation requirements. All of these processes have been demonstrated and have progressed from the laboratory and are under development at this time. It is anticipated that substantial progress will be made on chemical vapor deposition boron filaments in regard to quality, reproducibility, reliability, and cost over the next several years. There has been substantial progress in the development of boron filaments which can be prepared by a melt spinning process. This development is in a relatively early stage, and the process is essentially similar to that used for glass filaments. The boron filament materials prepared would be substrateless. There is great potential, if successful, for very high production rates, low costs, and good reproducibility. At the present time, a continuous jet has been established and short length substrateless filaments obtained. This initial success, based on past experience utilizing this technique for processing other molten materials into filaments, should mean that continuous boron filaments will be obtained in the laboratory in the immediate future. There is considerable development work ahead and principal problems to be resolved, beyond the processing procedure itself, involve the filament properties and raw materials costs. There is potential for this process eventually to provide boron fialments at a price of $5 to $10/lb. Silicon carbide filaments, which are continuous and have fairly good strength and modulus characteristics, have been prepared by chemical vapor deposition techniques. These filaments have a tensile strength of 400,000 psi, a modulus of elasticity of 50,000,000 psi, and a density of 1.04 lb/cu. in., and are presently available in experimental quantities. These filaments are of particular interest as a possible reinforcement for metal matrix composites, because they are less reactive than boron and have excellent high-temperature characteristics. Progress has been made in carbon filament process development, and in the immediate future it is hoped that continuous filaments with good intermediate characteristics may be available. These filaments have a very attractive low density, and there is great promise for the future availability of very high strength and modulus continuous carbon filaments.

In the area of composite development, there has been substantial progress in the treament, collimation, and preimpregnation processes to develop a usable tape material. In the treatment area, a recently completed study has indicated significantly higher filament properties that were translated into 30–40% higher composite properties by using a simple hot nitric acid boron filament etching technique. This process removes the surface imperfections and increases the quality of all as-formed filaments. It has great value especially in upgrading the performance of the lower grade filaments that have more surface flaws. The etch process results in an approximately 15% by weight boron filament removal. Additionally, a post-etch nitrogen treatment for boron filaments has been developed which has increased the composite wet interlaminar shear strength by a factor of 2. Boron filaments that have the nitrided surface also have greatly increased strength retentions at high temperature after exposure at temperature. These filaments, which lost 30% of their strength after an hour at 1000°F, showed no perceptible loss in strength after they were nitrided. This could be very significant for use with high-temperature resistant organic matrices and also for use with metallic matrices. All present boron filament material is now being etched and nitrided routinely. A laboratory collimation and impregnation unit for the formation of uniformly controlled pre-impregnated (prepreg) tape material has been developed. This unit is capable of handling 828/1031 commercially available epoxy resins and produces a 30-end, $\frac{1}{8}$-in.-wide tape that has good shelf life and laminating characteristics. This unit is being ultiized as the basis for an initial development unit to set up a continuing source for prepreg materials to all program activities. This development unit will increase the quantity processing capability from 1 lb/shift for the laboratory unit to 5 lb/shift. The development unit also will possess the capability to prepare wider bands of prepreg (on the order of up to 3 in.). Recent flat panel specimens prepared using such improved materials and improved fabrication techniques have reflected excellent composite properties. Examples are unidirectional compressive strengths of 350,000 psi with a 40,000,000-psi modulus and bidirectional compressive strengths of 170,000 psi, with a modulus of 20–25,000,000 psi. These laminates had densities of 0.075 lb/cu. inch and utilized commercially available matrix materials and no finish on the filaments. There also has been initial but widespread evidence of success reinforcing metals with boron filaments. These first generation efforts have utilized different fabrication techniques and different metals such as aluminum, titanium, nickel, and magnesium. Illustrative of the success and the potential of the concept of reinforcement of metals to upgrade their performance is the aluminum-boron composite area. Tensile strengths of up to 150,000 psi with a modulus of 30,000,000 psi have been achieved with 50% by volume boron reinforcement.

The component development area has been restricted to minimal activity in the past because of limitations in filament material availability and technological gaps existent primarily in the filament and composite areas. The past year has seen the evolution of some first generation evidences of advanced composite componentry. This activity was largely the result of either independent industry or cooperative Air Force industry efforts. The most illustrative these efforts utilizing organic matrices have produced boron and boron/glass stator vanes, boron compressor blades, and boron pressure vessels. These pressure vessels are of considerable interest since they have immediate application potential for life support and propulsion components. The boron reinforced organic matirx pressure vessels have high tensile strengths that enable, along with glass reinforced pressure vessels, considerable weight reductions from titanium or strainless steel structures. The filament wound vessels, whether boron or glass reinforced, require liner materials because of chemical compatibility and/or leakage considerations. The strain compatibility of boron composites with the metallic liner materials necessary, however, make it far superior to glass for usage from a cycling standpoint. Boron reinforced pressure vessels appear to be a most promising possible solution from a consideration of all critical factors. These industry efforts have great value and strongly complement the Air Force component development activity. In this area, however, the great strides and major advances are projected from Air Force sponsored programs. Along these lines, initial component development programs have been undertaken in five major structural application areas. These include aircraft wing and tail structures, re-entry vehicle structures, aircraft engines, and helicopter rotor blade applications. These initial efforts are for a period of seven months and will serve to answer some of the critical needs of the entire program. A primary purpose is to define the most urgent reinforcement materials, fabrication, and composite materials engineering problems. This will enable a rapid orientation and the establishment of priorities for the filament development and composite materials development programs to support most effectively the component development requirements. This component guidance is needed vitally since without specific inputs the most efficient materials development efforts are not possible. The component programs also will provide for their own initial advances in materials, design, and fabrication oriented specifically to their particular needs. It is realized that the short span of these programs will not enable great technological advances. Incremental and initial advances are anticipated, however, and these will be reflected in seven month structural demonstration items. These items will provide a reference plane for the status of the technology. They will also, more importantly, serve to highlight and emphasize the major problem areas still to be resolved. The future direction of the component development programs will be to demonstrate conclusively the advances made and the value of the technology to major structural components. The key milestone on the development timetable is a conclusive three year major structural assembly demonstration. This demonstration should be of such significance and magnitude to enable and support complete dynamic and flight testing immediately thereafter. An example might be a complete aircraft tail section or an entire rotor blade assembly. The seven month demonstration items will fit the three year development schedule and will be the first milestone in the over-all component development program within each area. The results of the seven month programs will be reviewed closely and continually to determine the over-all progress, the ability to meet realistically the program milestones, the payoff for composites in each structural component area, and the ability of the contractor materials/design/fabrication team to function effectively and efficiently to solve the problems facing component development. Based on this assessment, follow-on programs in the component demonstration area will be undertaken. Follow-on area(s), vigorously attacked on a component demonstration basis, also will be affected considerably in terms of the area selected, the nature and orientation of the program, and also the contractor activities involved, by the continued noncontractual Air Force-industry cooperative efforts in the various component areas. It can be assured that there will be a continuing drive to insure that the advanced development program will be flexible enough to be rapidly responsive to the most practical, realizable, and highest payoff development areas and the most progressive, capable, and efficient industry activities participate in the program.

In summary, an assessment of the present situation would indicate that the time is ripe for a concerted drive to develop and demonstrate the technology. There is available an initial high-modulus filament material in a form that makes the undertaking of this effort feasible at this time. There has been sufficient initial substantiation of the filament and composite performance to insure that we are not dealing with an artifact. There are sufficient quantities of high modulus material and a constantly improving cost picture to make practical a total development program that must include the design, fabrication, and test of full-scale structural components. There are, in the laboratories of the country today, research activities that show great promise in the immediate future for new reinforcement materials with both improved properties and lower cost potential characteristics. Indications have been good that metal matrix composities, which will relieve some of the current disadvantages of organic composites, are practical; and their rapid development is, and will continue to be, pursued vigorously. The experience with continuous glass filament composites in terms of design and fabrication, although lacking in a total sophistication, provide a firm enough basis for initial component efforts. There is at least a defined starting point for development of the vitally needed new design concepts and fabrication procedures. The initiation of component development activity has provided the program with a balance, guidance, and evaluation factor, which is so necessary for an efficient and successful full development, demonstration, and exploitation of an advanced composite structural technology.

The Air Force has committed itself to a concerted effort that will project the technology into the next generation of aerospace systems. An ever increasing, stimulated, interested, and responsive industrial community is eager and prepared to join in and undertake its crucial role in this effort. The problems to be faced are many and complex. The potential impact of the technology, however, is tremendous and far-reaching. The future is bright!