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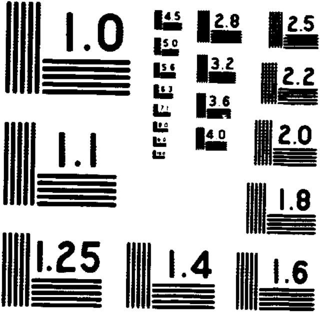

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A CRITICAL REVIEW OF EXPERIMENTAL ACCOMPLISHMENTS IN THE FIELD OF FILAMENT-REINFORCED METAL MATRIX COMPOSITES

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FOREWORD

The extraordinary potential of composites as a new type of material which can be tailored in properties to meet the requirements of a wide variety of high performance applications has created an enormous interest and generated a substantial volume of research activity in the area of continuous fiber reinforced metals. The demonstration that the predicted potential of such materials can be achieved has resulted in a rapid expansion of experimental effort and has created an avalanche of data which requires critical analysis and review. The emphasis which has been placed on the accelerated transition of this laboratory curiosity into a viable material for aerospace application has prohibited the concurrent detailed contemplation by inidividual researchers of the total volume of data being generated from the various active organizations.

This critical analysis and review of the research and development accomplishments in the metal matrix composites field is intended to consolidate the observations of the work conducted since the Cratchley(1) and Kelly and Davies(2) papers of 1965 in Metallugrical Reviews, cross-correlate the data, and compare the results and conclusions for internal consistency and compliance with the theoretical predictions for composite behavior. The delineation of research and development areas where focused activity can contribute most to the provision of metal-matrix composite materials to satisfy critical NASA needs is the ultimate objective of this work.

The review and analysis covers continuous fiber reinforced metal-matrix composites, their fabrication, properties, and problems.

ABSTRACT

The process of writing the critical review of the experimental accomplishments in filament reinforced metal-matrix composites was initiated during this contract period. The process of reviewing individual papers has continued, as has the cross correlation of results. This bimonthly progress report is composed of the text of the review which is in relatively finished form. The associated figures, graphs, and tables are not reproduced in preliminary form.

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SECTION I

INTRODUCTION

The principles and experimental aspects of fiber reinforced metals were admirably summarized in 1965 by Kelly and Davies(1) and Cratchley(2). The first of those articles provided the basis for thinking about this new class of materials and the second served as a basis for believing in them. Together they constitute the point of departure for subsequent work both in England and in the United States. An enormous expansion in metal matrix composites research and development has occurred in the past four years. Cratchley(2) indicated that a "great deal remained to be accomplished in demonstrating the feasibility of fiber reinforced metals for various applications". He identified the design of components from metal matrix composites as the "one field of fiber reinforcement which had apparently received little or no attention". This critical review will address itself to the experimental progress which has been made toward applicational acceptance of this new class of materials.

The historical development of filament reinforced metal matrix composites can be considered to initiate with the Jech, McDanels and Weeton(3), Sagamore Conference Paper in 1959. The reviews of Macklin(4) and Baskey(5) and the ASM Seminar volume(6) together with the Kelly and Davies(1) and Cratchley(2) papers provide a summary of the developments which occurred prior to 1965. Since that time two books have been published which are solely devoted to metal matrix composites(7,8) and multiple chapters of composite materials books are devoted to filament reinforcements of metals(9-18). A technical journal, Composite Materials, has been created in response to the expanding volume of research being generated. The Defense Metals Information Center prepares a periodic Review of Recent Developments for the topic Fiber-Reinforced Metals and has issued two DMIC Reports(19,20) which summarize the research on many unclassified Government-sponsored fiber reinforced-metal research programs. The preceding references, the Survey of Ceramics Fibers and Fibrous Composite Materials(21), an ASM bibliography(22), a Defense Documentation Center report bibliography(23) and the NASA SCAN report notification service for the topic, Composite Materials, were utilized to identify over three hundred contributions to the technical literature pertaining to filament reinforced metal matrix composites.

The body of the review has been organized into three principal subdivisions in an effort to cope with breadth of content which the accumulated literature provides. The fabrication techniques section is intended to review the types of processes which have evolved for composite preparation, identify the forms of composite which can be generated by each process, define the level of mechanical properties which are representative of the application of specific processes to the various filament-matrix composite systems and identify the current limitations of each process.

The discussion of the mechanical properties of metal matrix composites is contained in the second section of the review. It deals with the character of those properties rather than the absolute values generated by individual experimentalists. And finally the problem areas associated with or identified by the fabricational processes or mechanical properties are segregated for discussion. It is the objective of this final section to provide focus for future work by establishing priorities among the problems and suggesting experimental routes to their solution on the basis of the accumulated observations of the reviewed technical literature.

A historical observation of Thomas, Huffadine and Moore(24) regarding the development of cermets is most pertinent to the kind of development which must be accomplished for the composites field.

"In this particular field (cermets) the initial results rapidly gave rise to the realization that the problem of producing useful cermet combinations was more complex than had been originally envisaged. In particular, their brittleness and lack of impact strength became apparent. This factor, combined with the high cost and non-uniformity of much of the early material resulted in a waning of interest. The initial over optimism was replaced by undue pessimism. Throughout this phase the major emphasis had been placed upon the fabrication and testing of different metal/ceramic combinations in hope of finding a cermet with the desired properties. The amount of fundamental work done was, by comparison, small, and the net result was the accumulation of a mass of largely uncorrelated and inadequately understood data on very many different materials. Cermets were a new class of substances with distinct characteristics and neither the basic mechanism of bonding, the effect of different modes of fabrication, the methods of testing required nor the special design considerations involved were sufficiently appreciated."

The problems of producing useful composites has been identified to be complex and their high cost and nonuniformity are a caution flag relative to potentially waning interest. The initial optimism with regard to the achievement of a giant step forward in weight normalized strength and modulus has been fulfilled. The possibility of the development of undue pessimism can be avoided by the clear definition of the pertinent problem areas and the construction of an organized foundation of basic understanding beneath the technological advancement which has characterized these past four years of innovation. The composites field has its accumulation of uncorrelated and inadequately understood data but an appreciation has evolved for this new class of material which acknowledges the need for focused work on the basic mechanisms of bonding, the effect of various fabrication modes, the methods of testing and the special design considerations which are required.

SECTION II

SUMMARY

A variety of metallurgical processes including hot-pressure bonding, liquid-metal infiltrating, electrodeposition, vapor deposition, plasma spraying, cold press and sinter, extrusion and high energy-rate forming have been employed for the fabrication of filament reinforced metal matrix composites. Of these, by far the greatest emphasis has been placed on hot-pressure bonding techniques. The objective of any composite fabrication technique is to accomplish a specific form of material incorporating the reinforcing filament without breakage, with minimal reaction degradation, at a desired volume percent filament loading and with an interfacial bond which is sufficient to transmit the applied load from the matrix to the filament. Unfortunately the fabrication process development aspects of composite materials technology have not been reported in detail. Very few examples of processing parameter versus mechanical property data can be cited. The generalizations which have been reported are obviously based on such detailed studies but the process development effort has apparently been almost universally considered to be proprietary information.

The process development effort has consistently involved progress along a path of staged objectives:

1. Determine filament-matrix stability.
2. Achieve consolidation of filaments in a matrix.
3. Achieve tensile strength approximating that predicted by the rule of mixtures.
4. Reduce product variability at a high fraction of the rule of mixtures strength.

5. Achieve product scale up in size or flexibility of form.

6. Reduce processing costs.

Stage 3 objectives have been achieved for a multitude of filament-matrix combinations fabricated by a variety of processes and Stage 4 progress has been made for the most advanced composite system, hot-pressed aluminum-boron. The current applications oriented character of composites development centers on concurrent progress toward Stages 4 and 5 objectives. Processing cost reduction is an objective of the future which can only be achieved on the basis of a definable demand for a reliable material in a useful size and form.

Filament matrix stability is important to the development of a fabrication process for composite materials because it defines the degrees of freedom which exist for consolidation with minimal degradation in filament strength properties. The only effective techniques for demonstrating filament stability is the pre- and post-fabrication mechanical testing of incorporated filaments. Optical microscopy, electron microscopy, microprobe analysis, electron diffraction, X-ray diffraction and microhardness scans have been used to identify the onset of gross reaction in a wide variety of filament matrix systems. Sophisticated analytical tools have been of little value in identifying the onset of degradation in filament properties.

The development of a sufficient interfacial bond to accomplish load transfer from the matrix to the filament is an intuitively obvious requirement to achieve rule of mixtures performance in the composite system. However, little has been done to define the magnitude of interfacial tensile or shear strengths, to determine what an adequate bond is or to correlate bond strength with composite mechanical property performance. The fractographic observation of adherent metal skins on filament pullouts, the coordination of pullout lengths with expected critical transfer lengths and the degree of filament fragmentation in the composite mechanical testing operation are qualitative indicators that a good bond is beneficial. However, extended time diffusion reaction which can develop a good bond can also result in filament strength degradation. The composite property optimization process involves the definition of the time-temperature regime which yields stability as measured by filament strength retention and the accomplishment of a well-bonded composite within that stability range.

Table I is a summary of the most prominently investigated filament reinforced composite systems relative to their fabricability in viable form by the various processing techniques.

SECTION III

FABRICATION PROCESS DEVELOPMENT

The need for ever increasing composite tensile strength data has taken precedence over detailed development of basic understanding relative to the various composite fabrication processes for at least three of the last four years. As a result high-strength filament reinforced samples have been fabricated to demonstrate the achievement of the predicted potential for metal matrix composite materials. The recent concentration on applications oriented materials development programs have underlined the need for improved reliability, lower cost and greater flexibility in product size and form as the three imperatives for future use. A comprehensive report on manufacturing methods for composite materials by Glasser and Sump(43) contained the metal matrix composite fabrication process development which preceded 1965 and summary articles by Sutton(44), Harmon(45), Davis(46), Thornton(47), Herzog(48), Alexander(49-51), and Snide(52) record the continued progress in a general fashion.

HOT-PRESSURE BONDING

A variety of metallurgical processes, listed in Table I with applicable references, have been employed for the fabrication of composite materials. Of these, by far the greatest emphasis has been placed on hot-pressure bonding techniques. Included in the hot-pressure bonding category is any static pressure consolidation process carried out at elevated temperature whether it utilizes foil filament stacked arrays as in Figure 1a, sheet preforms as in Figure 1b, wire preforms as in Figure 1c or powder infiltrated spaced filament arrays as in Figure 1d.

The hot-pressure bonding process has been used successfully to fabricate aluminum, magnesium, titanium and nickel matrix composites. Foil filament arrays(11,18,20,21,33) are formed by carefully winding a specific filament spacing onto a foil covered drum and utilizing a cleanly decomposing binder to fix the filaments in place. An alternate technique(20) which accomplishes excellent control over filament spacing is cowinding of a matrix wire between reinforcing filaments. The foil filament array is removed from the drum and cut to the desired mat size for insertion into the hot press dies. A light retaining pressure is applied to the stack and the assembly is brought to pressing temperature with the attendent expulsion of the binder. The hot-pressure bonding step has been conducted in air, inert gas, or vacuum environments in chambers or as provided in a sealed retort.

Tape preforms(23,25,38,42) are handled in a similar fashion to the foil-filament arrays, Figure 1b. Both types of starting materials provide for the easy accomplishment of accurately oriented crossply filament orientations. The exhaustion of a binder phase is not a processing requirement for the matrix bonded preforms, however, the formation of the preform tape is considerably more costly than the preparation of a foil-filament array. Matrix coated filament, Figure 1c, has been successfully utilized(34,35) to form uniaxially oriented filament-matrix arrays. The larger degree of relative motion between filament in the consolidation step and the greater potential for random filament overlaps has resulted in the principal use of this process with the more ductile metal filament reinforced metal matrices. In such systems a considerable deformation of the incorporated filaments can occur especially as attempts are made for very high filament volume percent loading.

The final process of infiltrating spaced filament arrays with a powder metal matrix is the most tedious preparation procedure for the hot pressing of metal matrix composites, Figure 1d. The accomplishment and maintenance of good filament spacing is the tedious step. The resultant uniaxially aligned product can be consolidated by die or hot isostatic pressing and has characteristicly been most successful with metal filament reinforcements. A similar preparation procedure has been utilized for use in extrusion consolidation or exposure to high energy rate forming processes.

The assembled preforms are exposed to a combination of temperature and pressure for a specific time in a controlled environment to yield the fully consolidated composite product. The hot-pressure bonding process has been most extensively utilized to form sheet or plate composite forms. Representative cross sections of hot pressed uniaxial and orthogonal crossply composites are presented in Figures 2a and 2b. The adaptation of the hot-pressure bonding technique to more complex shapes has been successfully demonstrated(53). The desired hot pressed final form is differentially simulated as shown in Figure 3a and pressed in resistance heated conformal dies, Figure 3b, to yield the desired complex shape, Figure 3c.

Optimization of the hot-pressure bonding fabrication process has been carried to the most advanced state for the aluminum-boron system as reflected by compliance of the maximum tensile strength properties as a function of volume percent filament with the strength calculated to be attainable from the proportional contribution of the incorporated filament and matrix(32,51,54,55). Filament strength degradation during an optimized hot-pressure bonding cycle approximates 10%.

LIQUID METAL INFILTRATION

Some of the very first reinforced composites were produced by vacuum infiltration of a tube filled with filaments. Such techniques were and are applicable when the filament is liquid metal stable. The model systems of refractory metals in matrices which exhibit little mutual solubility represent a class of materials which have contributed significantly to the understanding of composite characteristics(27-29,56). However, the extremely reactive nature of the most advanced filament, boron, has minimized the practicality of liquid state composite fabrication. The model systems have yielded reproducible quality composites at high volume percent filament loading with close correspondence to rule of mixtures strength and modulus predictions. Stability is the key to success in the utilization of liquid metal fabrication techniques and only magnesium of the structural metal shows significant stability in contact with boron to be practically fabricated by such techniques. Schuerch(26) achieved compressive strengths approaching 350,000 psi in early liquid magnesium infiltrated boron specimens. Only a few minutes of exposure to liquid aluminum is enough to seriously degrade the filament(34). The documented stability of SiC in aluminum matrices(38,57) and the utility of coated boron (SiC, BN, or Ag) as a more stable reinforcement offers future potential for the expansion of liquid fabrication techniques. It is the relative instability of the available advanced filaments which has dictated that little effort be devoted to sophistication of liquid metal fabrication techniques.

Vacuum infiltration, Figure 4a, of a bundle fiber in a tube is a batch process which is limited in size potential, and susceptible to incomplete or irregular fill. However, the utility of liquid infiltration techniques has been demonstrated by the continuous casting of boron filaments in a magnesium matrix(58) and by the effort to form three filament tapes by the rapid passage of boron through aluminum(34). The process simply involves the passing of a bundle of filaments through a metal bath in such a fashion as to accomplish wetting of the individual fibers as they enter the bath and wiping off the excess as the bundle is drawn through an orifice in the bottom of the crucible, Figure 4b. The filaments can alternately be passed through holes in the bottom of the crucible and consolidated through a constraint of the shape and dimensions of the finished rod, Figure 4c. The simplicity of the process decrees that volume production of rod, tube or structural shapes is possible at little added cost over that of the incorporated raw materials. The microstructure of a 75 v/o boron-magnesium continuously cast rod is shown in Figure 5a while Figure 5b shows the almost perfect hexagonal packing of the most densely packed areas. Complete metal sheathing of even the most closely spaced filaments is evident.

The continuous coating process is capable of yielding any uniform cross section form of uniaxially aligned filament reinforced composite. Rods, tubes and structural shapes are uniaxial forms of material and as such can take full advantage of the maximized composite properties in the axial direction. Strength-to-density values in excess of 2×10^6 inches and modulus-to-density ratios in excess of 500×10^6 inches place this type of material in the position of exhibiting a two to four times advantage over conventional aluminum and titanium alloys.

ELECTRODEPOSITION

The utilization of electrodeposition as a fabrication technique saw early application with reactive filaments because it could be accomplished without elevated-temperature exposure and a concurrently deposited sample of matrix could be obtained for mechanical test(59-63). The electroforming technique involves the electrodeposition of the matrix onto a suitable mandrel while concurrently winding the filament reinforcement and has been discussed by Bonnano(64). A schematic of the composite fabrication process is shown in Figure 6. The technique is applicable to any metal that can be electrodeposited and has the following advantages:

1. It is a room temperature fabrication in process.
2. A fully dense matrix sample can be concurrently deposited.

3. Intimate filament-matrix contact is accomplished at the interface.
4. Any shape which can be made as a surface of revolution can be fabricated.
5. Accurate control can be exercised over filament spacing and thus volume percent loading.

Figure 7 is a schematic of the growth pattern which is characteristic of this fabrication process. Figure 7A indicates the mode of formation of the electrodeposit on the filaments which are wound onto an undercoat of nickel on the winding mandrel. Figure 8 shows a monolayer nickel-boron tape formed in this manner. Figure 7B shows the continuation of the process by winding and coating of a second layer. Multiple layer samples are produced by a repetition of this process until the desired thickness is achieved, Figure 9. Figure 7C shows the location of potential void sites in the composite structure. Type 1 voids occur when deposition on the filament progresses at a rate such that the growth from two adjacent filaments intersects before growth from the undercoat reaches the point of intersection. Type 1 voids can be grown out at wide filament spacings by flooding the mandrel with fresh electrolyte and by the imposition of plate-deplate cycles on the forming operation. Type 2 voids are formed when the surface contour of the overcoat for the first layer does not conform to the filament size and shape. The character of such voids is shown in Figure 10. If the overcoat is thick enough the grooves become rounded and accept the subsequent filament layer with little porosity. However, at high volume percent loadings the crevices occupied by the circular filaments leave triangular voids beneath them. It should be emphasized that even when the geometry of the surface is correct for the acceptance of the filament without void formation, the character of the bond between the filament and the matrix is different at the contact point than on the rest of its circumference. At that point it has simply been laid against the matrix, while elsewhere the matrix has been electrodeposited onto it. Another important consideration in the characterization of electroformed composites is the need for accurate control over filament spacing. Variations in filament spacings result in changes in the surface contour of the electrodeposited overcoat. Wide spacings yield a larger valley and close spacings create a larger hump and the effect of such misspacings is to force greater misspacings upon the subsequent layers. Such misspacings ultimately lead to a greater void formation in higher volume percent multilayered specimens.

This effort characterizing the electroforming process for continuous filament reinforced composites can be summarized as follows:

1. Monolayer filament tapes can be produced with minimal void entrapment to roughly 45 v/o.
2. Multilayer composites can be formed to equivalent volume percent loadings but geometrical considerations combined with the potential for misspacings make void formation a problem to be contended with.
3. A densification process should be considered necessary in conjunction with composites formed by electrodeposition.
4. Monolayer tapes can be used as a raw material for multilayer composite fabrication by hot-pressure bonding.

In addition to monolayer tapes, multilayer circumferentially wound reinforced structures can be formed as illustrated in Figure 11, a 20 filament layer circumferentially wound simulated motor case in the Al-B system. The multilayer circumferentially wound composites have demonstrated a tensile insensitivity to the hoop oriented voids(65) as shown in Table II. Hoop strengths exceeding 200,000 psi with a modulus of 35 million psi generate strength-to-density values in excess of 2.0 x 10^6 inches and modulus-to-density values over 400 x 10^6 inches. Composite strength in the transverse direction is minimal (20-30% of the matrix strength) because of the incorporation of 10-15% voids.

The electrodeposition process is capable of yielding continuous monolayer composite form of a width which is only limited by the engineering ability to collimate thousands of filaments. Circumferentially wound structures of several feet in diameter could be deposited as a simple extension of demonstrated fabrication capabilities.

PLASMA SPRAYING

The plasma spraying technique for composite fabrication has been optimized most completely by Krieder(23,25) in the aluminum matrix system and the feasibility of the process for the formation of tungsten wire reinforced tungsten rocket nozzle configurations has been demonstrated(66).

The process is shown schematically in Figure 12(23). This schematic is equally applicable to the description of chemical vapor deposited and vacuum deposited matrices on which a lesser degree of development has been conducted. A layer of matrix in the form of a foil or as a plasma sprayed layer on the mandrel is overwrapped with a spaced array of reinforcing filaments which are incorporated by the spraying of a subsequent layer of matrix. The operation is conducted in an inert atmosphere or with a protectively shrouded flame. The as-sprayed matrix is not fully dense (12-15 v/o voids) and contains a somewhat higher oxide content (1.5 wt %) than foil type material. Transverse strength is low in the as-aprayed condition and matrix ductility is lower than wrought aluminum. The impact of molten particles of matrix on the filament surface provides ultimate contact at the filament matrix interface, and the immediate quenching of the small particles prohibits extensive reaction degradation in the composite formation process. Boron degradation is approximately 20%(23) in the plasma spraying process while the coefficient of variation is almost doubled.

The preparation of multilayer composites by plasma spraying is characterized by the same sort of void formation and filament misplacement difficulties described for the electrodeposited composites. Also, circumferentially reinforced composites exhibit the same poor transverse properties. Post deposition sintering treatments spherodize voids but do not increase matrix density while hot-pressure bonding of multilayer or stacked monolayer plasma sprayed material results in a fully consolidated composite with transverse properties approximately 1/2 that of the matrix itself.

Further optimization of the plasma spray composite fabrication process(39) has been achieved utilizing the silicon carbide coated boron filament which exhibits significantly less sensitivity to the high oxygen content of the sprayed matrix. Coated boron filament does not exhibit the relatively large fabrication degradation nor does it degrade as rapidly as a function of time at temperature in the plasma sprayed condition. This effort concentrated on the optimization of the plasma spraying process to yield a tape preform for subsequent hot-pressure consolidation.

The monolayer tape material as a preform for subsequent hot-pressure bonding experiments has an operational advantage over organically bonded foil filament arrays in that no binder need be exhausted in the fabrication step. Commercially available steel foil heat treating envelops can be utilized to provide the protective environment for the hot-pressure bonding step. However, the higher cost of the preform fabrication process, the necessity of using a higher price coated filament, and the effect of the higher matrix oxide content on stability and mechanical properties are identified disadvantages.

The excellent as-sprayed mechanical properties qualify this process for the formation of multilayer circumferentially reinforced bodies such as the simulated motor case shown in Figure 11 or as a hoop reinforcement in the intermediate temperature ring sections of jet aircraft engine compressor analogous to the boron reinforced resin rings which have been evaluated by Pratt & Whitney at lower temperatures.

A direct application evaluation for plasma sprayed composites was conducted by Greening(66) where tungsten fiber was incorporated in a tungsten matrix by plasma spray deposition. Figure 13 shows a mandrel with a helically wrapped array of filaments prior to plasma spraying. Operational tests on the fabricated nozzles indicated the feasibility of this high-temperature refractory composite structure for relatively simple fabrication of many complex configurations on conventional equipment at a nominal cost.

CHEMICAL VAPOR DEPOSITION

Chemical vapor deposition as a technique for analogously infiltrating filament arrays have been investigated in a preliminary fashion. Withers(67) has worked with the Al-Be system utilizing the decomposition of aluminum alklys on a heated mandrel wound with beryllium filament. While chemical vapor deposition yield a fully dense deposit of the matrix, the irregular nature of the composite surface after the encapsulation of the first filament layer causes misspacings and contact voids as subsequent layers are added. Chemical vapor deposition has the process attribute of yielding metal deposition on all heated surfaces simultaneously rather than being a line of sight process as in plasma spraying or depositing only on the surface of electrically conducting constituents as in electrodeposition. The chemical vapor deposition process operates at a temperature which is low relative to the metal melting point or the effective hot-pressure bonding temperature and thus offers the potential of nonreactive consolidation. While feasibility has been demonstrated, fabrication process optimization would be required to achieve useful mechanical properties in a filament wound structural application.

The feasibility of forming W-W and W-B composites by chemical vapor deposition techniques has been demonstrated by Greszeuk(68) where the relatively low metal deposition temperature (800-1100°F) made consolidation possible without the severe reactivity that would have accompanied hot-pressure bonding or liquid infiltration procedures. The deposition morphology reported indicates that continuous tape formation by chemical vapor deposition on a spaced array of filaments can be considered feasible.

COLD PRESS AND SINTERING

The process of cold pressing powder-filament arrays and sintering has been utilized principally as a preparation procedure for subsequent extrusion and rolling consolidation. The process has been principally applied to metal filament reinforcement of metal matrices. Baskey(69) has indicated that the long time sintering of cold pressed powder-filament blends at relatively high temperature is an undesirable technique for the accomplishment of full density composites because of excessive degradation of incorporated filaments. Additionally the thermal expansion mismatch in some systems, i.e. W or Mo in Hastelloy X results in the matrix expanding away from the wires during sintering. Thus cold pressing or cold pressing and sintering were used only to achieve compact and coherent billets for subsequent hot pressing, extrusion or rolling. Adamski, et al(70) on the other hand utilized the cold press and sinter technique to form Ag-W composites with mechanical property results which were equivalent to hot pressed samples. However, the number of specimens involved make speculation as to the influence of fabrication variables on properties most hazardous.

The Baskey observation that long-term elevated temperature exposures are required for a high degree of densification would seem to limit the process to filament-matrix combinations where extreme stability is exhibited, i.e. stable reinforcements in the low melting metal matrices.

EXTRUSION AND ROLLING

Refractory metal wires have been consolidated in nickel base superalloys and in titanium alloys by extrusion and rolling techniques(69) and the brittle filament boron has been extruded in an aluminum matrix(71). The Baskey(69) work demonstrates how thorough a fabrication process development program can be when a relatively inexpensive filament is being incorporated by a production type process. The experimental program defines the chemical compatibility of the alloys and filaments of interest and then proceeds to fabricate discontinuous and continuous filament composites from hot or cold pressed billets by extrusion and hot rolling within the defined range of chemical compatibility. It is one of the few detailed investigations of the influence of fabrication variables on the properties of fiber-reinforced metals.

It is clearly demonstrated that randomly oriented discontinuous filament-matrix powder mixtures can be cold or hot pressed into a preform for extrusion to yield uniform cross section rod composites containing aligned fiber reinforcements. The effects of preform treatments and extrusion parameters together with post-extrusion heat treatment upon the broadest possible range of pertinent mechanical properties are examined with a sufficient number of samples to permit the definition of a reliable set of conclusions which define the optimum treatment for particular sets of desirable properties. The results are compared to both the unreinforced matrix similarly treated and to the wrought forms of alloys which are the conventional competitors for the applicational range envisioned for these composite materials.

Initial filament strengths are monitored but the composite is characterized in terms of what processing variations can do to its properties rather than in terms of what theory predicts it should be capable of accomplishing. Improvements are registered in both the nickel base superalloys and in the titanium alloy matrices in tensile and yield strengths and stress rupture properties formed by extrusion of discontinuous refractory metal filament reinforced powder compacts. Continuous filament composites of equivalent volume percent loadings exhibited better performance than the discontinuous ones. Very well bonded composites were achieved in spite of the large disparity in thermal expansion coefficients.

HIGH ENERGY RATE FORMING

The utilization of high energy rate forming as a composite fabrication technique has been surveyed by a number of investigators at the Pacific Northwest Laboratories of Battelle Memorial Institute. Figure 14 is a schematic of pneumatic impaction apparatus utilized to impart pressure pulses of up to 400,000 psi to canned powder filament arrays inserted in the die at elevated temperatures. While the process has admirably demonstrated its capability to accomplish consolidation there has been little process optimization against filament degradation or composite properties. The process feasibility work has indicated that low consolidation temperatures result in excessive filament breakage and it is apparent that higher consolidation temperatures result in serious degradation in filament properties. It is apparent from the mechanical property data on the system which has been studied in the most detail, Ti-SiC(75,76), that the time-temperature exposure associated with the high energy rate forming step is excessive. The optimum HERF temperature of 1100°C for that system is significantly above the 900°C maximum temperature utilized for hot-pressure bonding consolidation of the same system(38,41).

The justification for detailed HERF process optimization in a useful metal matrix system lies in the requirement for scale up in the size of sheet or plate composites which can be reproducibly fabricated. In principal, the use of explosive techniques or HERF to apply the consolidation pressure for large size composites is an attractive one. The serious problems of maintaining a uniform distribution of filaments while minimizing both filament breakage and filament reaction degradation have been identified and serve as the basis for continued exploration of the utility of this composite fabrication technique.

The summarized processes represent the source of various forms of composites. The most advanced yield properties which closely approximate the values predicted by rule of mixtures calculation. The accomplishment of rule of mixtures values is heralded and broad generalizations concerning the effects of processing variables on the attainment of such properties are recited. But the experimental data which document the generalizations are largely invisible.

The accomplishment of high strength and modulus values has experimentally demonstrated the potential for composite materials but the translation of existing processes to useful size, with good reliability and at a cost competitive with more conventional alloy designs is the current challenge to the experimentalist. What then can be derived from the critical evaluation of the reported experimental accomplishment in the area of composite fabrication technology?

Table III is a summary of the types of process development data which has been most informative in the dual task of optimizing composite mechanical properties and understanding the origins of the derived properties. Contained in Table III is the blueprint to the understanding of product variability. In simple terms we must understand the filament reinforcement that is being utilized; we must understand it in terms which are pertinent to its functioning in the composite; we must monitor the effect of various processing variables upon its characteristics;and we must correlate its characteristics with the properties derived from the fabricated composite. On a parallel framework we must understand the load translation process in a functioning composite in terms of the character and strength of interfacial bond which is developed by the range of fabrication parameters which maintain filament properties and accomplish composite consolidation. The model for composite failure outlined by Rosen(77) defines the type of data which is essential to the establishment of consistency in such materials:

"When a fiber break occurs, there are several possibilities for the subsequent behavior of the composite. First, the high interface shear stresses may produce interface failure which could propagate along the length of the fiber reducing the fiber effectiveness over a substantial fiber length. To achieve the potential of the fiber strength, it is necessary to study and determine the fabrication conditions which will yield an interface sufficiently strong to prevent interface shear failure. This can be done by using either a high-strength bond or a ductile matrix which permits redistribution of the shear stresses. In the latter case the length of fiber which is affected by the break will increase as it will take a longer distance to retransmit the stresses back into the fiber at the low stress level of a ductile matrix. With a strong bond, the interface conditions can be overcome as a potential source of failure. Second, the fracture toughness of the matrix must be considered to prevent the propagation of a crack through the matrix and parallel to the filaments. A third possibility is that the initial crack will propagate across the composite resulting in failure. This is influenced in part by the fracture toughness of the matrix, and again, since it is clear that with brittle fibers one can always expect a fracture to occur at a relatively low stress level, it is important that the fracture toughness of the matrix material is sufficient to prevent propagation of this crack across the composite. If these two potential modes of failure are arrested, it will then be possible to continue to increase the applied tensile load and to obtain breaks at other points of imperfection along the fibers"

Thus the ultimate objective of a metal matrix composite development program must be a sufficiently well-bonded system to accomplish repeated filament fracture until a statistical accumulation of fiber fractures occurs in the vicinity of one cross section to provide the opportunity for final catastrophic failure. While the rule of mixtures has been an admirable target for composite fabrication optimization, every major fabrication program has yielded individual composite strength values which exceed rule-of-mixtures calculations. If such strength values are to be attained consistently, the statistical nature of the failure phenomenon must be realized and new objectives must be set which transcend those which would be calculated from a simple rule-of-mixtures calculation.

We know that filament strength goes up with decreasing gauge length(78). We know that the standard deviation on strength goes down with decreasing gauge length(79). We know that filaments do break to relatively short lengths in tested composite specimens(39) and we know that the filament fragment lengths tend to be shorter immediately adjacent to the site of final specimen failure(41). The quantitative representation of these characteristics as a function of fabrication parameters is essential to the basic understanding of composite fracture and minimization of variability in composite specimens.

The work of Lenoe(79) establishes statistically the ultimate tensile strength of the filament utilized in that program as a function of gauge length. The standard deviation on U.T.S. as a function of gauge length could be computed. The thermal degradation of the filament for time-temperature exposures which were pertinent to the fabrication procedure was documented in sufficient detail to again permit calculation of standard deviations on U.T.S. at a single gauge length. Filament extracted from fabricated composite samples were tested in the same fashion. The expansion of this procedure to cover a range of gauge lengths and to document the effect of the matrix digestion solution on heat treated filament would provide the background for the interpretation of digested filament degradation effects as a function of fabrication time, temperature, pressure and environment as reported by Cunningham(33) or the correlation of filament degradation data with composite properties as a function of complete range of processing parameters such as were studied by Baskey(69). The accomplishment of minimum filament degradation in the fabrication process and the utilization of the full strength contribution of the minimally degraded filament requires an adequate bond at the filament-matrix interface. Thus quantification of the measurement of the degree of shear strength developed at the interface is essential. The length of sheathed filament pullouts or the measurement of post tensile test filament fragments in the specimen gauge length are techniques which have been utilized. The consistent minimization of filament strength degradation and the concurrent establishment of an adequate degree of bonding at the load transfer interface will permit the evaluation of composite fabrication processes on the basis of consistent full utilization of reinforcing potential.

Cost is the second deterrent to early application of metal matrix composites on a competitive basis with other structural materials. The $500-$600/lb price for hot-pressure bonded material in multi-thousand pound quantities represents fabrication costs which are a factor of 3 to 4 over the cost of incorporated filament. Process economization is obviously essential. Many metallurgical processes can and will prove capable of yielding viable composite samples but detailed and expensive process development effort must give heavy emphasis to the potential for economical production in a useful size.

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