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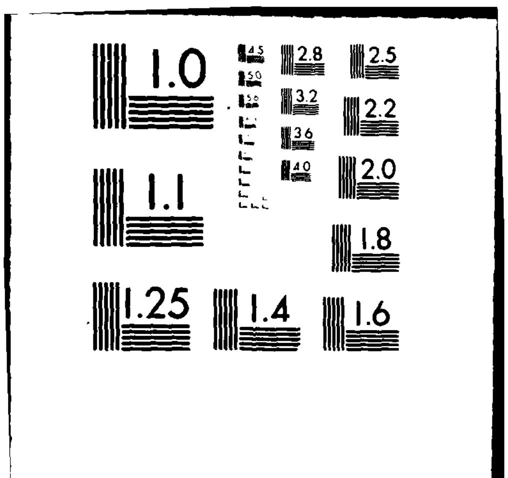

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A FUNDAMENTAL STUDY OF A NEW FABRICATION TECHNIQUE FOR FIBER REINFORCED ALUMINUM MATRIX COMPOSITES.

LEVEL II

FINAL REPORT
3/22/76 TO 8/11/79

(10) R. MEHRABIAN

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U.S. ARMY RESEARCH OFFICE

(15) DAAG29-76-G-0170, DAAG29-78-G-0067

~~DEPARTMENT OF METALLURGY AND MINING ENGINEERING~~
~~DEPARTMENT OF MECHANICAL AND INDUSTRIAL ENGINEERING~~
UNIV~~ERSITY~~ OF ILLINOIS AT URBANA-CHAMPAIGN
~~URBANA, IL 61801~~

(9) Final rept. 22 Mar 76-11 Aug 79,

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO. AD-A086 282	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) A FUNDAMENTAL STUDY OF A NEW FABRICATION TECHNIQUE FOR FIBER REINFORCED ALUMINUM MATRIX COMPOSITES		5. TYPE OF REPORT & PERIOD COVERED Final Report 3/22/76 - 8/11/79
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) R. Mehrabian		8. CONTRACT OR GRANT NUMBER(s) DAAG29-76-G-0170 DAAG29-78-G-0067
9. PERFORMING ORGANIZATION NAME AND ADDRESS Dept. of Metallurgy and Mining Engineering Dept. of Mechanical and Industrial Engineering Univ. of Illinois, Urbana, IL 61801		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS DRXRO-PR-P-13847-MS
11. CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Research Office P. O. Box 12211 Research Triangle Park, NC 27709		12. REPORT DATE April 20, 1980
		13. NUMBER OF PAGES 15
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Distribution Unlimited
Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

N/A

18. SUPPLEMENTARY NOTES

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19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Composite, discontinuous fibers, solidification, Al_2O_3, aluminum alloys, interface interactions

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The feasibility of fabricating fiber-reinforced aluminum alloys by addition of discontinuous fibers to partially solid slurries and completely liquid matrices was investigated. In the first phase of the program, emphasis was placed on the study of interface interactions between polycrystalling Al_2O_3 fibers and Al-2 to 8%Mg, Al-4.5%Cu and Al-4.5%Cu-1 to 2%Mg alloys produced by the slurry process.

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In general, it was observed that the incorporation of fibers could be readily achieved by this technique, and that fibers appeared wetted after a few minutes of contact with the melt. The composites produced exhibited an intimate, void free bond between the constituents. In addition, a region of significantly altered microstructure resulted from accumulation of oxide and/or aluminate particles which either formed within the melt and were attached to the moving fibers, or used the fiber surface as a substrate to grow on. Microscopic examination of this interaction zone and thermodynamic considerations indicated that it consisted of fine α-Al_2O_3, aluminates, oxides of the alloying elements, and probably some intermetallic compounds. For example, it was shown that a stable $MgAl_2O_4$ spinel forms at the interface of Al_2O_3 fibers and Al-Mg alloys. A major shortening of this process was fiber breakage during composite fabrication. The second process developed, in the last fifteen months of the program, avoided fiber damage by incorporating them into gently agitated completely liquid Al-Mg alloys. Examination of fiber - matrix interfaces verified the earlier findings that an intimate bond formed which consists of $MgAl_2O_4$, MgO and fine α-Al_2O_3. A new method was also developed to increase the fiber concentration in the composites and for simultaneous two-dimensional alignment of the fibers. Measured room temperature tensile properties exceeded those predicted by various available theories. For example, the addition of 23 v/o FP Al_2O_3 fibers to an Al-4% Mg alloy matrix increased the planar-random modulus of elasticity and ultimate tensile strength by $\sim$ 50% and $\sim$ 40% respectively. SEM studies of the fracture surfaces showed that failure of the composite seemed to occur through the matrix and not at the interface. Therefore, the interface between the fibers and the matrix was strong enough to permit the transfer of loads.

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I. INTRODUCTION

During the period of this investigation 3/22/76 to 8/11/79, research was focused on (1) the development of new innovative techniques for the production of aluminum alloy - discontinuous Al_2O_3 fiber composites, and (2) the development of a fundamental understanding of the wetting behavior and interface interactions between the fibers and several aluminum alloy matrices. The first fabrication technique studied involved the addition of the Al_2O_3 fibers to agitated, partially solid slurries of Al-Mg, Al-Cu and Al-Cu-Mg alloys. The fibers were incorporated in the alloy and the composites produced exhibited an intimate, void free bond between the constituents. Interface interactions as a function of process variables were studied in detail and the phases present at the matrix-fiber interface were identified. A major difficulty experienced in fabrication of composites by introducing Al_2O_3 fibers into partially solid metal slurries was fiber breakage due to interaction between the brittle fibers and the primary solid particles of the alloy in the slurry. In the last fifteen months of the program a new process for fabrication of the composites was developed. In this case, the matrix alloy is always at a temperature above its liquidus. However, the essential feature of this new process as of the prior one is that induced convection of the melt permits disruption of contamination films or absorbed layers and produces intimate contact between the fiber material and the melt so that interface interactions are facilitated. The temperature and agitation conditions in the two processes are near enough so that the interface reaction layers are substantially the same. However, the new method produces composites with higher fiber fractions and longer fibers. Futher processing of the composites thus produced permits two-dimensional alignment of the fibers with attended improvements in mechanical properties.

II. FABRICATION USING METAL SLURRIES - INTERFACE INTERACTIONS

It is commonly agreed that alumina is an ideal reinforcing material for aluminum since the two are physically and chemically compatible at the projected service temperature of the resultant composities [1]. However, the combination of these two constituents is complicated by the non-wetting characteristics of the system, and either coatings or alloying additions have to be used to promote interactions between the matrix and the filaments.

Interface interactions are important not only in the performance of the composite but also in the design of appropriate fabrication techniques to produce the desired bond. This, in turn, involves the study of surface phenomena like wetting and chemical reactions, and their effect on interfacial bond strength.

Fabrication of Al/Al_2O_3 composites is limited by the non-wetting characteristics of this system, and two different approaches to this problem have been investigated. The first one involves the use of sputtered coatings like Ni(Ti), Ni(Cr), and 1020 carbon steel [2,3] which physically attach to the fiber surface and are wetted by the liquid metal. However, most coatings readily dissolve in molten aluminum and may cause debonding unless the fabrication time is short, on the order of a few minutes. The second approach to enhance wetting calls for the use of alloying additions that can interact chemically with the ceramic material creating a desired new phase at the solid-liquid boundary [4]. Several authors have proposed that elements with greater affinity for oxygen (as measured by the free energy of oxide formation) than the solvent will preferentially concentrate at the interface [5], hence reduce the surface energy (γ_{SL}) as described by the Gibbs absorption equation. However, in the case of Al/Al_2O_3 all the alloying additions that have higher affinity for oxygen than the aluminum also tend to reduce Al_2O_3 and modify the chemical nature of its surface.

It is known that Al_2O_3 readily reacts with many divalent transition metal oxides to form aluminates which are isostructural with the mineral spinel of composition $MgAl_2O_4$. Several investigators [1,6,7] have indicated that spinels or similar oxides may be used to promote interfacial bonding since they have the potential to form strong bonds with both metals and ceramics. If one accepts this point of view, then aluminum alloys containing Mg, Cu, An, Fe become particularly interesting since they could form aluminate compounds under the appropriate conditions. Although some of these reactions have been observed with Al_2O_3 refractory bricks [8-10] only a few additions have been reported to promote sufficient wetting of uncoated Al_2O_3 fibers for successful liquid infiltration [4]. These additions normally involve the use of Li (which forms $Li_2O \cdot 5Al_2O_3$ at the interface) with the consequent problems of preparation and handling of the alloy, and the control of the chemical interaction during the fabrication process to avoid fiber degradation.

In the work described herein fabrication of Al_2O_3 fiber reinforced aluminum alloy composites by incorporation of discontinuous filaments into a partically solidified, vigorously agitated metal slurry was studied. The first phase of this program was mainly concerned with the study of the formation of bonds and related interfacial phenomena during the fabrication process. Two series of experiments were conducted for this purpose. In one series, continuous fibers were immersed into static pools of fully liquid alloys, while in the other discontinuous fibers were incorporated into agitated baths of partially solid metal slurries. The latter approach was used to prepare composites containing from 1 to approximately 15 v/o of fibers with a variety of alloys as matrices. The structure of the composites, and especially the interaction zone, were analyzed using different microscopy techniques. Some composites with more than 10 v/o fiber were extruded and fractured under tension to examine the behavior of the matrix-reinforcement bond. Copper and magnesium were selected as alloy additions because both have been reported to reduce the contact angle between aluminum and Al_2O_3 [12].

Table I lists composites prepared and studied in this portion of the program. The details of the findings from this work have been published [13]. A summary of the findings is listed below.

1. Homogeneous dispersions of polycrystalline Al_2O_3 fibers* were obtained by adding them to agitated, partially-solid slurries of Al-Mg, Al-Cu and Al-Cu-Mg alloys. The fibers appeared wetted and bonded to the matrix in the final composite product. It is postulated that the fibers added to the metal slurries were kept dispersed by the agitation and the moving primary solid particles until they developed some sort of interaction with the matrix. Incorporation and wetting were thus readily achieved even in alloy systems that normally exhibit a non-wetting behavior.

2. Attempts to incorporate the fibers into static baths of the alloys above their liquidus temperatures were unsuccessful.

3. Microscopic examination of the composites prepared revealed the existence of a significantly altered microstructure around the Al_2O_3 fibers which consists of a fine multiphase material. Features common to all the structures were the existence of an intimate bond, the absence of voids at the fiber boundary and the presence of fine polycrystalline α-Al_2O_3 in the interaction zone. The average maximum thickness of this "apparent interaction zone" depends on residence time and alloy composition. No changes in the dimensions and optical appearance of the interaction zone were observed after heat treatment of the composites.

4. Interactions between Al_2O_3 fibers and Al-Mg alloys resulted in the formation of a Mg-rich region around the fibers which was retained during heat treatment. This is attributed to the presence of $MgAl_2O_4$ and MgO at the fiber boundary in addition to α-Al_2O_3. Changes in appearance of the interaction zone were observed for different magnesium contents in the alloy.

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*These are Dupont's Type I Al_2O_3 FP fibers. The properties of the fibers are listed in Table II.

5. Interactions in the Al-Cu system produced a distinctive accumulation of copper around the fibers. The copper occurs in the form of discrete particles of a Cu-rich phase which disappears with heat treatment. It is suggested that $CuAl_2O_4$ may be present along with α-Al_2O_3 and possibly $CuAl_2$ in the interaction zone.

6. Addition of small amounts of magnesium to the Al-Cu alloy significantly reduced the extent of interaction observed. Both magnesium and copper enrichments around the fiber were detected in this case, but as opposed to the Al-Cu matrix, the Cu-rich phase was still present at the fiber boundary after heat treatment. Experimental observations indicate that $MgAl_2O_4$, α-Al_2O_3 and possibly $CuAl_2O_4$ coexist in the interaction zone.

7. The results of this study suggest that a compound of the aluminate type may form on the fiber surface and provide the required bond with the surroundings. This reaction will be enhanced in the presence of oxygen which is introduced by the vigorous agitation during the fabrication step.

8. Examination of fracture surfaces of the composites revealed that in general the failure was not at the interface but rather by plastic flow of the matrix around the fibers.

III. NEW COMPOSITE FABRICATION TECHNIQUE

As previously noted, a major difficulty experienced in fabrication of composites by introducing the Al_2O_3 fibers into partially solid metal slurries was fiber breakage due to interaction between the brittle fibers and the primary solid particles of the alloy in the slurry. To avoid this difficulty, a new composite fabrication technique was developed in this program in which uncoated discontinuous fibers are introduced and successfully incorporated in baths of aluminum alloys. In this process, the alloy is kept above its liquidus temperature while the discontinuous fibers are introduced and entrapped in the melt via mechanical agitation of the latter. The discontinuous fibers are thus forced into intimate contact with the melt which invariably results in chemical interaction between the two - e.g. formation $MgAl_2O_4$. This technique avoids fiber damage. Details of the apparatus and procedure used are described elsewhere [14].

The composites produced contained discontinuous fibers which were randomly oriented in three-dimensions. A method was developed to form a disc-shaped part which also aligned the fibers and increased their volume fraction in the composites [14]. Table III gives the size and volume percent of the fibers in each composite product produced in this investigation. The fiber length and volume fraction were determined by dissolving away the matrix alloy with a 30% hydrochloric acid solution. From the data given in Table III, it is evident that some fiber damage occurred during the compositing and the compression procedure. During the compositing, the fibers were reduced to approximately one-half of their size before addition to the melt. Subsequent forming into disc-shaped parts further reduced the fiber lengths by a factor of two.

Close examination of the interfaces were carried out in cooperation with another research team [15]. Auger and electron diffraction studies of the Al_2O_3 fibers isolated from a composite verified the earlier conclusions [13] that $MgAl_2O_4$ spinel formed at the interface when the fibers were introduced into agitated Al-Mg melts. The formation of this spinel is important because a strong bond is required to permit the transfer of load from the matrix to the fiber.

Tensile specimens from the disc-shaped composite product and the matrix alloy, which was shaped under identical conditions, were machined and subsequently tested at the Textile Fibers Department, Pioneering Research Laboratory of DuPont. The results are given in Table IV. As expected, the modulus of elasticity and the ultimate tensile strength of the composites improved with increasing fiber content. The stress vs. strain curve for an Al-4% Mg composite containing 23 v/o Al_2O_3 fibers is shown in Figure 1. The modulus of elasticity and the ultimate tensile strength of this composite were 100 GPa and 254 MPa.

In order to achieve strengthening in a discontinuous fiber composite, the average aspect ratio of the fibers and the total volume percent of the fibers in the composite must exceed critical values. Using the analysis of Kelly and Tyson [16] and the data in Table IV, critical values of $\sim$14:1 for the aspect ratio and $\sim$13 volume percent of fiber content in the composite are calculated.

The experimental results of the tensile tests are plotted in Figure 2. The dotted lines show the predicted behavior of the modulus of elasticity and the ultimate tensile strength with increasing fiber content, based on the theory of Kelly et al [16] with the assumption that the average fiber length is equal to the critical length. This theory treats the case of uniaxial discontinuous fiber composites, whereas Christensen and Waals [17] consider randomly oriented fiber composites containing continuous fibers. The solid line shows the behavior of the modulus of elasticity with increasing fiber content according to Christensen and Waals. The experimentally determined maximum values of the modulus are in good agreement with those predicted by this theory.

The fracture surfaces of the tensile specimens were examined in order to determine the nature of failure of the composite. Many fibers exhibited clean fracture surfaces and this was probably due to failure under shear or bending stresses. There was very little fiber pullout, and dimpling and undulations were noted in the ductile matrix. This indicated that the failure was probably through the metal matrix and not at the interface.

In summary, this new composite fabrication technique permits successful incorporation of discontinuous Al_2O_3 fibers into Al-Mg matrices. Furthermore, two-dimensional alignment of the fibers in a composite containing $\sim$23 v/o of FP Al_2O_3 fibers increased the planar-random modulus of elasticity and ultimate tensile strength by $\sim$50% and $\sim$40%, respectively.

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Table I

Summary of Composites Fabricated in the Liquid-Solid Range

Run	Matrix Alloy[1]	Fiber[2] Content (Vol.%)	Addition Temp. (°K)	Addition Time (min.)	Total Residence Time[3] (min.)
1[4]	Al-4.5%Cu	1	925	5	60
2	Al-4.5%Cu	1	910	2	50
3	Al-4.5%Cu-0.8%Mg	2	900	60	75-135
4	Al-8%Mg	1	873	5	60
5	Al-4%Mg	1	903	5	90
6	Al-2%Mg	1	913	5	145
7	Al-4.5%Cu-2%Mg	1	890	3	90
8	Al-2%Mg	12	910	35	12-40
9	Al-4.5%Cu-2%Mg	14	900	28	20-55
10	Al-4.5%Cu-2%Mg	14	900	20	10-30

Notes:

[1] Degassed with N_2 except in runs 2-4. Compositions given in weight %.

[2] Initial fiber length was 3 mm except for run 9 where 6 mm fibers were used.

[3] Total time includes the remelting which took 15 to 20 min. on the average.

[4] Run 1 was made in the fully liquid state under agitation.

TABLE II

Properties of Type I FP Al_2O_3 Fiber [4]

Tensile Strength	1.4 GPa
Modulus of Elasticity	380 GPa
Density	3.95 g/cm^3
Melting Point	2318 K
Filament Diameter (round cross-section)	15-25 µm
No. of Filaments in Continuous Yarn	210

TABLE III

Measured Volume Percent and Size of Fibers in the Al-4% Mg Composites Produced

Composite No.	As-Cast Ingot v/o	As-Cast Ingot ℓ/d	Disc-Shaped Part v/o	Disc-Shaped Part ℓ/d
1	5	48	14	25
2	7	42	19	28
3	9	35	21	26
4	10	39	23	21

Average length to diameter ratio, ℓ/d, of the fibers before addition was $\sim$ 100.

TABLE IV

Tensile Properties of the
Planar-Random Fiber Composites

Ingot No.	v/o Fibers	UTS (MPa)	Modulus (GPa)
Matrix Alloy	0	188	63
1	14	205	87
1	14	206	84
1	14	196	88
2	19	—	99
2	19	—	98
3	21	194	88
3	21	257	104
3	21	193	97
4	23	243	92
4	23	243	95
4	23	254	100
4	23	263	105

The yield strength of the matrix alloy is $\sim$ 110 MPa

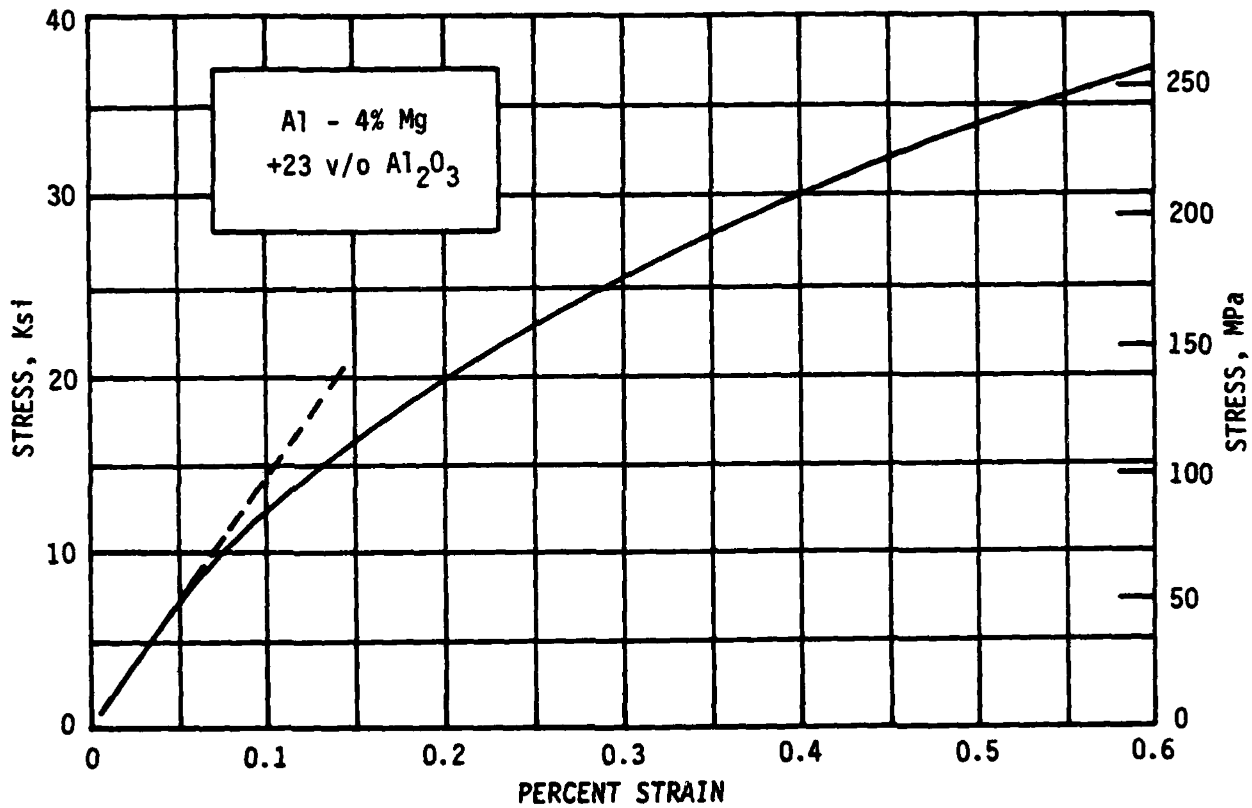

Figure 1. Stress vs. Strain curve for an Al-4% Mg planar-random fiber composite part containing 23 v/o Al_2O_3 fibers.

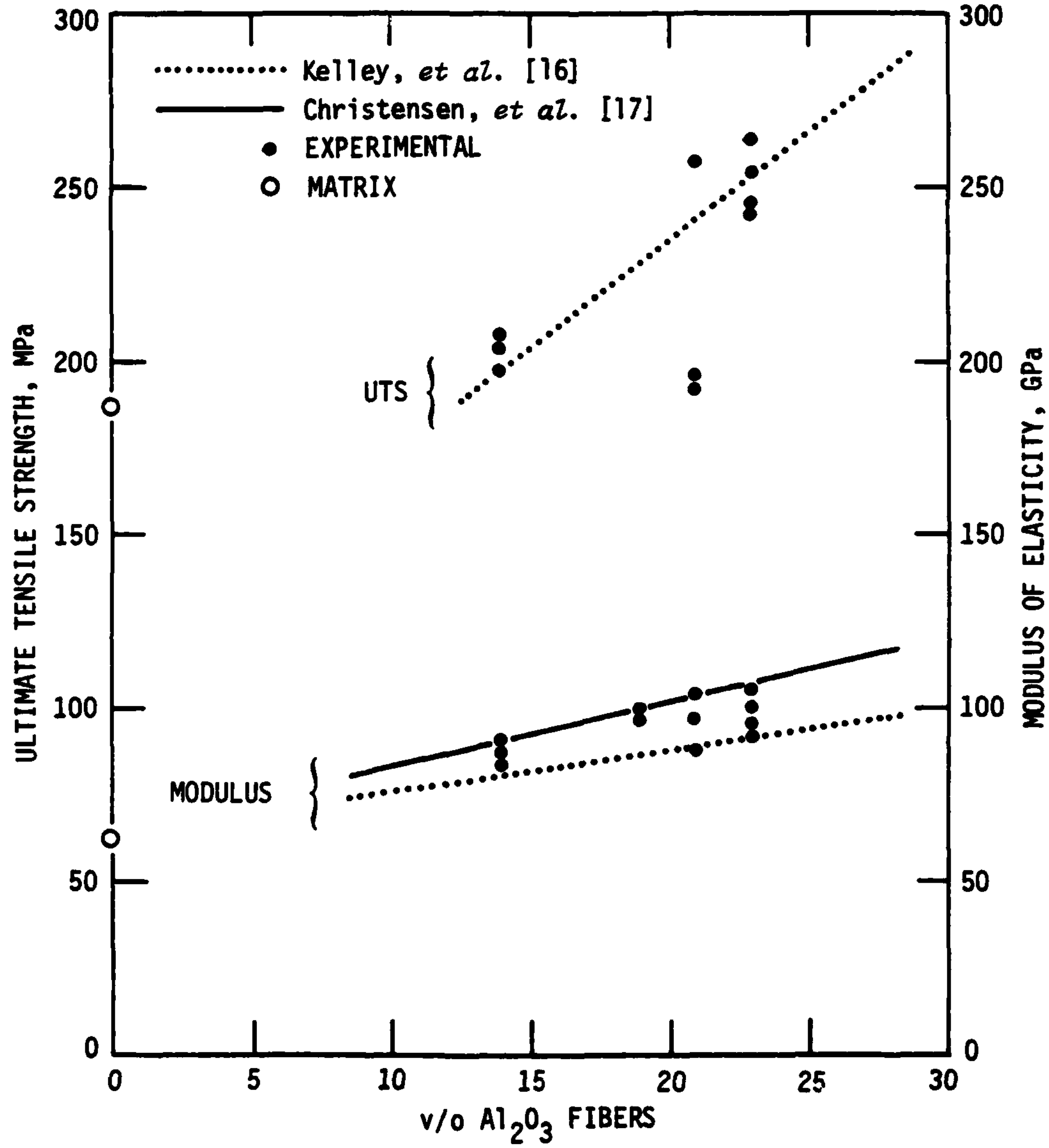

Figure 2. Calculated and experimentally measured moduli and ultimate tensile strengths of planar-random fiber composite parts.

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