

# POWDER FABRICATION OF ALUMINUM ALLOYS

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FOREWORD

This report was prepared by the Kaiser Aluminum and Chemical Corporation under USAF Contract No. AF 33(616)-2296. The contract was initiated under Project No. 7351, "Metallic Materials", Task No. 73513, "Aluminum Alloys", formerly RDO No. 615-14, "Aluminum Alloys", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Lt J. D. Wood acting as project engineer.

This report covers work conducted from December 1953 to December 1954.

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## ABSTRACT

A fabrication method, combining powder metallurgy techniques and conventional extrusion, was developed for the purpose of alloying aluminum with refractory compounds and other unusual constituents. Additions of  $B_4C$ ,  $\alpha-Al_2O_3$ , TiC, SiC,  $ZrO_2$ , WC, TlAl,  $MnAl_6$ ,  $FeAl_3$ , Mo, Cr, Si and Cu were made to a base of commercial atomized aluminum powders, and the resulting alloy properties were determined.

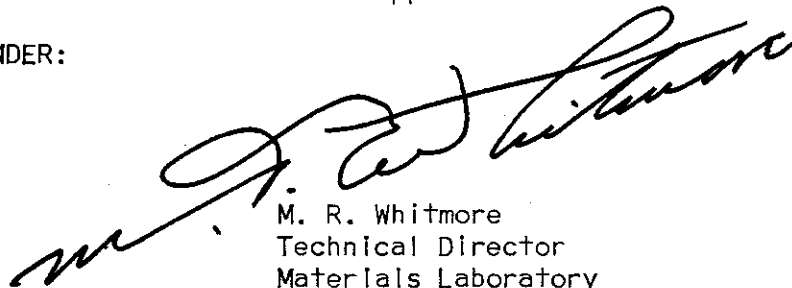
Outstanding improvements in Young's modulus were achieved by means of the SiC and  $B_4C$  additions; however, accompanying tensile properties were only mediocre.

Current attempts to improve these tensile properties by utilizing pre-alloyed powders as bases for the refractory additions are still in preliminary stages of study.

## PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



M. R. Whitmore  
Technical Director  
Materials Laboratory  
Directorate of Research

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## I. INTRODUCTION

Aluminum alloys have been the chief structural materials for aircraft construction. However, future aircraft and air weapons will require light weight structural materials having greater strength, higher modulus, and better elevated temperature properties than the aluminum alloys which have been developed and used heretofore, so that aircraft designers can provide better performance and overcome the problems of higher stresses and aerodynamic heating at faster-than-sound air speeds.

The possibilities for obtaining appreciable improvements in the above properties with modified aluminum alloys of conventional types have appeared to be somewhat meager. However, the development of Al-Al<sub>2</sub>O<sub>3</sub> alloys and recent studies of the effects of additions of the nitrides of aluminum, chromium, magnesium, iron, vanadium, titanium and zirconium to aluminum by powder metallurgy techniques<sup>(1)</sup> have suggested that unconventional alloying additions may offer great possibilities for producing significant property improvements. The present project was undertaken to survey these possibilities in considerable detail.

The specific objectives of this investigation were:

- (1) to explore the potentialities for developing improved aluminum alloys through additions of unusual alloying constituents, and
- (2) to determine the practicability of introducing these additions and of fabricating improved aluminum-base structural materials by means of powder metallurgy techniques in combination with other conventional metal working procedures.

The properties in which improvements were specifically sought included:

- (1) higher Young's modulus,
- (2) greater strength at room temperature and at elevated temperature, especially in the neighborhood of 600°F, and
- (3) increased strength-to-weight ratio.

An initial comprehensive literature survey disclosed no attractive possibility whatsoever for increasing the Young's modulus of aluminum alloys by means of solid solution alloying effects, nor by means of controlled crystallographic textures to take advantage of natural crystalline anisotropy. However, clear evidence of excellent possibilities for producing a higher modulus was found in the work of Dudzinski<sup>(1)</sup> and of others<sup>(2,3,4)</sup>, based upon the principle of producing a polyphase alloy structure by introducing into the aluminum matrix secondary phases which individually possess high moduli. Theoretical reasoning suggested that the incremental improvement in modulus which might result from this type of alloying procedure should

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be approximately proportional to the volume-fraction of high modulus constituent added and to the difference in modulus values between that of the added phase and the aluminum matrix.

It seemed likely, moreover, that this type of alloying procedure might also produce improved elevated temperature properties, especially if a uniformly ultra-fine dispersion of the added phases in the aluminum matrix could be achieved. For example, the outstanding elevated temperature properties of S.A.P. apparently can be accounted for on the basis of the extremely fine dispersion of the refractory  $Al_2O_3$  secondary phase<sup>(5)</sup>

## II. EXPERIMENTAL DETAILS

### A. General Plan of Experiments

Since the main objective of the project was to develop structural alloys suitable for application as extrusions, forgings, sheet, bar, etc., the experimental powder alloys were all prepared and studied in wrought form, extrusion being selected specifically for this purpose as one convenient typical method of metal working on which initial studies might reasonably be concentrated.

The initial literature survey had indicated that at least three major problems could be anticipated in fabricating aluminum powder alloys, namely:

- (1) difficulties from the aluminum powders seizing and galling the walls of the compacting dies,
- (2) tendency for extrusion of the very plastic aluminum particles at clearances between moving and stationary parts of the compacting dies during consolidation operations, and
- (3) lack of satisfactory bonding between particles because of inadequate puncturing of the oxide shells originally present on the surfaces of the powder particles.

Consequently, it became necessary to devote considerable attention, as one of the major items of study, to investigation of the effects of variations in fabricating practices and to the development of suitable general fabrication procedures to produce powder alloy specimens of consistently sound metallurgical quality. The fabrication variables which were studied, at least partially, included:

- (1) powder compositions,
- (2) nominal particle size and shape, and particle size distribution

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of the component powders,

- (3) cold compacting pressure,
- (4) die lubrication,
- (5) sintering time and temperature,
- (6) extrusion die design (e.g. flat-faced vs. bell-shaped entries), and
- (7) post-extrusion heat treatment and/or working.

To provide a base of reference, initial studies were carried out using unalloyed aluminum powder. The procedures thereby developed were then modified as necessary to permit satisfactory fabrication of the various powder alloys.

The alloying additions selected for the initial series of powder alloys were chosen primarily on the basis of their theoretical promise for increasing the Young's modulus without substantially raising the alloy density. For this purpose, the specific modulus (i.e. modulus-to-specific gravity ratio) was utilized as a criterion of merit for constituent selection, because any addition constituent having a higher specific modulus than that of aluminum itself (i.e. in excess of  $3.7 \times 10^6$  psi) would, in theory, be expected to produce alloys of higher specific modulus than aluminum, in proportion to the volume-fraction of constituent added, as long as the addition is chemically stable with respect to the aluminum matrix. Based therefore upon such data regarding the specific modulus of elements, inter-metallic compounds, and refractory materials as could be found in the technical literature, and upon such additional considerations as stability, melting point, hardness, and possible commercial feasibility, the constituents listed in Table I were selected for evaluation as powder additions in a matrix produced from commercial, atomized, unalloyed aluminum powder.

Tests showed that the tensile properties of the powder alloy specimens fabricated from mixtures of these constituents with unalloyed aluminum powder were not greatly superior to those exhibited by specimens fabricated from aluminum powder without additions\*. The obvious next step was to attempt to strengthen the matrix of the powder alloys. The experimental approach employed for this purpose was to substitute known conventional alloys for the matrix of unalloyed aluminum in the powder alloys. Particular attention was given to using those alloys which exhibit the best elevated temperature properties. This alloying of the matrix could, at least in theory, be carried out by either of two alternative procedures. The necessary elements could be added as separate powder additions before compacting, and an intermediate heat treatment of sufficient duration to permit

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\* However, all additions produced some increase in modulus; and several, as will be described later, produced very significant improvements.

TABLE I

SPECIFIC MODULI OF CONSTITUENTS SELECTED  
AS ADDITIONS TO ALUMINUM POWDER ALLOYS

<u>Constituent</u>	<u>Young's Modulus (psi x 10<sup>-6</sup>)</u>	<u>Specific Gravity</u>	<u>Specific Modulus (psi x 10<sup>-6</sup>)</u>
B <sub>4</sub> C	65	2.5	26
Be	37	1.82	20
αAl <sub>2</sub> O <sub>3</sub>	52	3.7	14
TiC	45.8	4.93	9.3
SiC	21	2.73	7.7
VC	39	5.36	7.3
Si	16	2.33	6.9
ZrO <sub>2</sub>	36	5.49	6.6
WC	102.8	15.7	6.6
Mg <sub>2</sub> Si	12	1.88	6.4
TiAl	20.5	3.8	5.4
Cr	36	7.19	5.0
Mo	50	10.2	4.9
MnAl <sub>6</sub>	unknown	3.09	unknown*
TiAl <sub>3</sub>	unknown	3.31	unknown*
FeAl <sub>3</sub>	unknown	3.81	unknown*
CrAl <sub>7</sub>	unknown	unknown	unknown*

\* MnAl<sub>6</sub>, TiAl<sub>3</sub>, FeAl<sub>3</sub> and CrAl<sub>7</sub> were selected on the basis of the secondary considerations, for general exploratory purposes, even though modulus data were not available for them.



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diffusion and alloying in the solid state could be used during fabrication. Alloy matrices could also be made by preparing pre-alloyed powders for direct incorporation in the powder alloys. The latter method appeared to be the more desirable alternative. Unfortunately, pre-alloyed aluminum powders are not available commercially, and considerable time was necessarily expended in having them prepared. As a result, this last-described portion of the experimental program could not be completed during the contract period.

## B. Equipment and Testing Methods

A cylindrical, hardened steel die of approximately 5/8 in. diameter was used to cold compact the blended powder mixtures. These compacted billets were then extruded with a small extrusion apparatus mounted between the hydraulic platens of a 60,000 lb tensile testing machine. General views of this extrusion equipment are shown in Figure 1. The compacted billet, as well as the billet container, extrusion die, dummy block and ram are all enclosed within an electrical resistance furnace so that these tools are maintained, during extrusion, at a constant temperature which is controlled within  $\pm 3^{\circ}\text{F}$  by means of a thermocouple permanently located in the container wall. In conjunction with a die giving an extrusion ratio of 12.8:1, extrusion speeds up to approximately 2.5 ft/min can be obtained. A detailed view of the extrusion tools and a typical extruded specimen of 0.175 in. diameter are shown in Figure 2.

Crushing, milling, sieving, and powder blending equipment were all of conventional design. Heat treatment of the powder alloy specimens was carried out in electric, forced circulating air ovens.

Young's modulus was determined by a dynamic testing procedure patterned after the method of Roberts and Nortcliffe<sup>(6)</sup>. Specimen rods were suspended, as "free-free" or floating beams, from supports placed near the nodal positions for the fundamental mode of transverse vibration, and the resonant frequency for this type of vibration was measured. The Young's modulus was then calculated directly from this measured frequency to an estimated precision of less than 1%. In a few cases, the dynamic measurement of the modulus was checked by a static method, using a Martens extensometer.

Tensile tests at room temperature were carried out on a 5000 lb Model PTE-3 screw-actuated tensile machine, using spherically seated Templin Vee-grips. The specimens were tested with as-extruded surfaces, without machining.

For short time tensile tests at 600°F, the specimens were prepared with a reduced section approximately 1-1/4 in. long x 0.125 in. diameter. A chromel-alumel thermocouple was wired to the specimen with asbestos tape. Specimens were brought to temperature with a Marshall vertical furnace and controller, held at temperature 15 minutes, then tensile tested using a free-running crosshead separation rate of 0.16 in./min. No attempt was

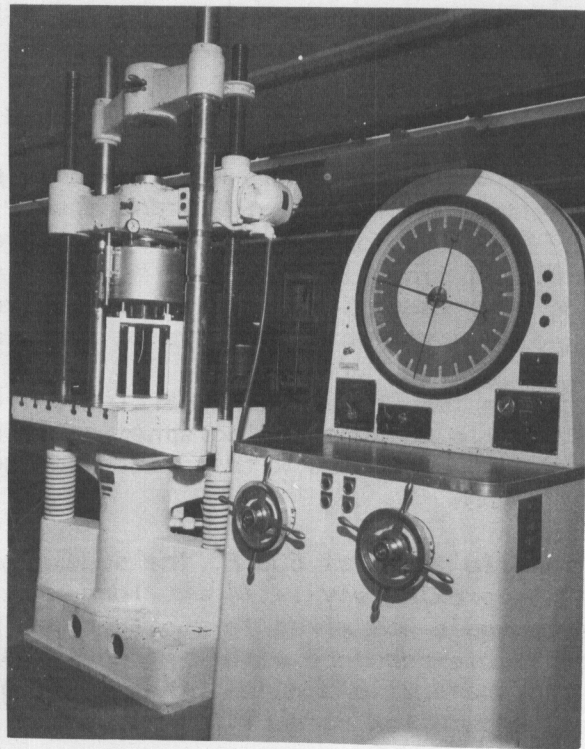
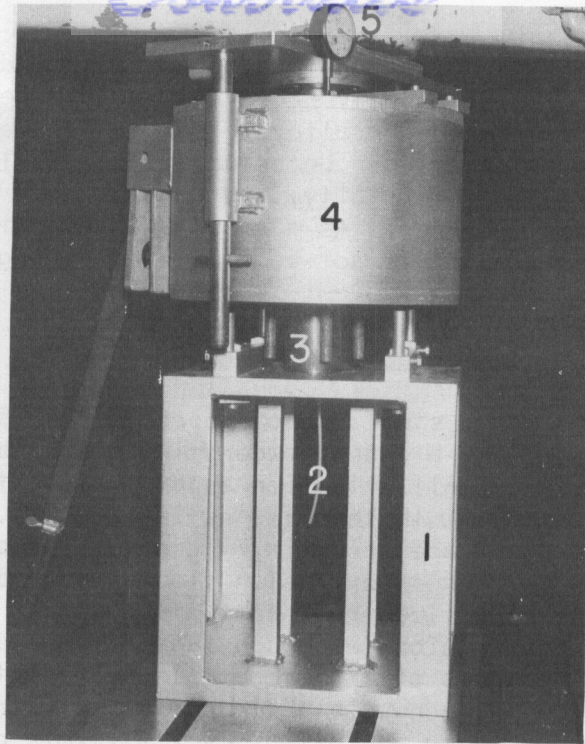


Fig. 1 Two views of extrusion press mounted in tensile testing machine: (1) support stand, (2) extrusion, (3) die bolster, (4) furnace shell, (5) dial gauge to measure extrusion rate.

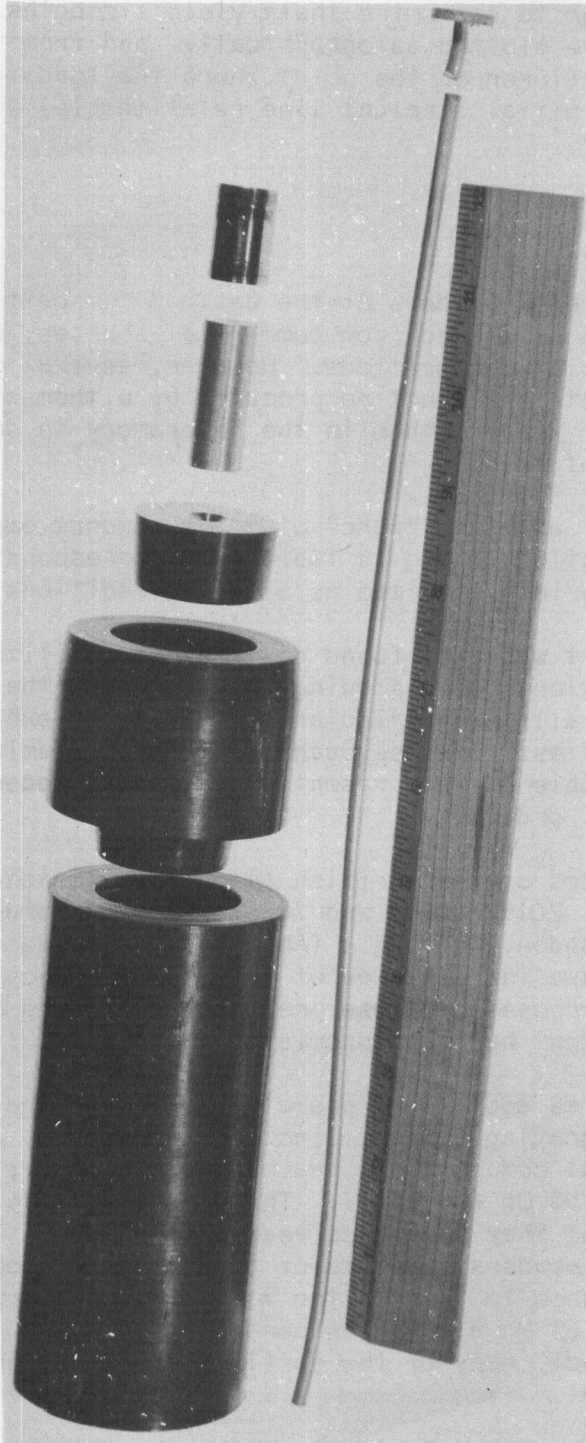


Fig. 2 Exploded view of extrusion tools. A typical extruded specimen with sheared discard butt is shown just above the ruler.

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made to record the stress-strain curves of these small specimens autographically in order to determine their yield strengths. However, load vs. time curves were plotted autographically and from these an apparent proportional limit (taken as the point where the load-time curve first departed from the initial straight line relationship) was obtained in each case.

## C. Materials

Whenever possible, powders of the desired compositions, particle shapes and mesh sizes were purchased from commercial sources, either as standard products or as special preparations. However, several of the desired addition constituents could not be procured by either of these methods, and it was necessary to prepare them in the laboratory in small quantities suitable for the present tests.

The commercial atomized "pure" aluminum powders employed in the studies had the characteristics listed in Table II. Corresponding data for the powders of the materials utilized as alloying additions are given in Table III.

In addition, it was also found desirable to utilize pre-alloyed atomized powders in compositions corresponding to several of the high strength conventional aluminum alloys, particularly those which exhibit the best elevated temperature properties. Because such pre-alloyed aluminum powders are not commercially available at the present time, it was necessary to have them prepared on special orders.

Atomized powders of four British alloys, corresponding approximately to the alloys 2014, 2018, 5056, and 7075, have been ordered from Powder Metallurgy Ltd., London, England. (An order for powder of R.R.57 alloy was not accepted, because the patentee of this alloy composition refused to sell it for atomizing purposes). These pre-alloyed powders have not yet been received, but shipment has been promised in December, 1954.

Meanwhile, three additional pre-alloyed powders have been prepared by the Metals Disintegrating Company, Inc., Elizabeth, N. J., in one standard and two experimental compositions, namely: 2024 alloy; Al + 6% Mg + 0.3% Zr; and Al + 6% Mg + 0.5% Cr + 0.1% Ti. These powders were obtained only very recently, and as yet they have received little study. The metallographic structure of these powders consists of an extremely fine chill cast structure, comparable in fineness to that of the metallized powders described below. The powders of the 6% Mg alloys, in particular, appear to exhibit considerable gas porosity; in fact, many of the particles seem to consist only of hollow, thin shells of metal.

While the pre-alloyed powders were being prepared commercially, an attempt was also made to prepare pre-alloyed powders in the laboratory by a spray method using a Metco (Metallizing Engineering Company, Inc.) Type 2E metallizing gun for the purpose. The metallized powders tended to consist of excessively elongated particles. However, after considerable

TABLE II  
CHARACTERISTICS OF COMMERCIAL ATOMIZED ALUMINUM POWDERS

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Laboratory Designation	Supplier	Manufacturer's Designation	Manufacturer's Data		Laboratory Sieve Analysis					
			Min- mum Purity	Con- trol Mesh	Fineness Index	+100	+200	-200	+325	-325
A	Reynolds Metals Co.	DE-198 #200	99.3%	-200			0	5	11	84
B	"	DE-199 #400	99.3	-325			0	0	0	100
C	Metals Disintegrating Co.	MD-13	99.0*	-20	30%-325		39	19	11	31
D	"	" MD-33	99.0*	-30	35%-325		14	24	19	43
E	"	" MD-44	99.0*	-40	40%-325		25	25	13	37
F	"	" MD-101	99.0*	-100	75%-325		0	9	11	80
G	"	" MD-105	98.0*	-100	99%-325		0	0	0	100
H	"	" MD-201	98.5*	-200	85%-325		0	0	10	90
J	**	--	--	--	--		100	0	0	0
K	**	--	--	--	--		0	100	0	0
L	**	--	--	--	--		0	0	0	100

\* Includes Al<sub>2</sub>O<sub>3</sub> content.

\*\* Laboratory sieve fractions prepared from powder "E".

TABLE III  
CHARACTERISTICS OF POWDER ALLOYING CONSTITUENTS

Constituent	Supplier	Description or Mfr's Designation	Purity	Chief Impurity	Particle Size	
					Mesh	Microns (a)
B <sub>4</sub> C	Norton Co.	"Norbide"	99%	- - -	-320	
αAl <sub>2</sub> O <sub>3</sub>	Linde Air Products	Type A	99.9%	- - -	--	<0.3
αAl <sub>2</sub> O <sub>3</sub>	Laboratory Preparation (b)	- -	99.5%	about 25% γAl <sub>2</sub> O <sub>3</sub>	-65	35-200
TiC	A. D. Mackay, Inc.	- -	95%	C, Fe	--	7-20
Si	National Radiator Corp.	G-120	96%	SiO <sub>2</sub>	-200(c)	15-70
SiC	Buehler, Ltd.	No. 400 grit	--	- - -	--	20-40
ZrO <sub>2</sub>	Metal Alloys Corp.	- -	--	- - -	--	9-14
WC	A. D. Mackay, Inc.	Carbide Tool Grade	--	- - -	--	4-6
TiAl	Blackwell Metallurgical Works, Ltd. (d)	Hardener Alloy (pulverized in laboratory)	--	TiAl <sub>3</sub> , Fe, Si	-150(c)	18-90
Cr	Chas. Hardy, Inc.	Electrolytic Grade	99%	- - -	-300	15-33
Mo	Chas. Hardy, Inc.	Hydrogen Reduced	99.75%	- - -	-200	5-13
MnAl <sub>6</sub>	Laboratory Preparation (e)	- -	--	Al, MnAl <sub>4</sub>	-200(c)	8-65
FeAl <sub>3</sub>	" " (f)	- -	--	- - -	-200(c)	11-70

TABLE III  
CHARACTERISTICS OF POWDER ALLOYING CONSTITUENTS  
(CONTINUED)

Constituent	Supplier	Description or Mfr's Designation	Purity	Chief Impurity	Particle Size	
					Mesh	Microns(a)
CrAl <sub>7</sub>	Laboratory Preparation	(g) --	--	--	--	--
Mg <sub>2</sub> Si	"	(h) --	--	--	--	--
Cu	Metals Disintegrating Co.	MD-410 Electrolytic	99%	--	-40	--
Cu	Metals Disintegrating Co.	MD-210 Electrolytic	98.5%	--	-200	10-20

- Notes:
- (a) Estimated microscopically.
  - (b) Bayer Process alumina, calcined 30 minutes at 1000° C.
  - (c) Laboratory sieve fraction.
  - (d) A high purity stoichiometric TiAl alloy (Ti + 36% Al) has been prepared in the laboratory by arc-melting under vacuum to replace this impure alloy in future studies.
  - (e) A binary alloy analyzing 23.78% Mn was prepared by melting high purity components at 1800°F. This melt was slowly cooled to 1300°F and held at 1300°F for 42 hours to permit the peritectic conversion to MnAl<sub>6</sub> to occur. After complete solidification, the alloy was pulverized and screened to -200 mesh. Metallographic and x-ray diffraction analysis indicated that the peritectic reaction was not actually completed in this alloy, and that it contained some unreacted MnAl<sub>4</sub> and Al.
  - (f) An alloy analyzing 40.8% Fe was prepared by melting high purity components at a temperature somewhat above 2000°F, and then chill cast, pulverized and screened to -200 mesh.
  - (g) CrAl<sub>7</sub> was recovered as a residue from an electrolytically leached chill-cast Al-4% Cr binary alloy, using dilute HCl.
  - (h) Mg<sub>2</sub>Si was prepared by leaching the excess magnesium from a slowly cooled Mg-30% Si binary melt, using saturated NH<sub>4</sub>Cl solution.

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experimentation with the operating conditions employed during spraying, reasonably spheroidal-shaped particles were produced in the size range of -100/+325 mesh. In general, it was found desirable to remove an erratic portion of coarse, sprayed fragments by sieving to approximately -100 mesh, and similarly to eliminate fines smaller than -325 mesh which tended to be especially elongated.

The best powders were produced under the following spray conditions:

Oxygen	30 cu ft/hr
Acetylene	34 cu ft/hr
Air blast pressure	60 lb
Wire size	1/8 in. diameter
Wire feed rate	6 ft/min

The metal spray was directed vertically down a bottom-vented collecting stack into a pan of water, approximately 7 to 10 ft distant from the spray nozzle, from which the powders were recovered and dried.

Within the limited range of particle sizes aforementioned, the metallized powders appeared to be a satisfactory product; Figure 3, for example, illustrates the extremely fine chill cast microstructure that was typical of these powders. However, the complexity of the method (which includes the necessity for first casting the desired alloy and drawing it into wire) and the poor recoveries of final product makes it relatively impractical, even as a laboratory method for preparing special powder compositions, except as an essentially last resort.

### III. EXPERIMENTAL RESULTS AND DISCUSSION

#### A. Development of Powder Alloy Fabrication Techniques

1. "Pure" aluminum powders. An attempt was made to avoid using lubricants during cold compacting. However, serious galling of the compacting die was encountered in cold compacting all grades of "pure" aluminum powders (Table II) at compacting pressures sufficiently high to achieve relatively well-consolidated billets, while green densities of only 65 to 70% of theoretical density were obtainable at compacting pressures sufficiently low to hold the rate of build-up of galled metal on die parts within reasonably tolerable limits. This galling problem was greatly alleviated by lightly coating the die parts before each compacting operation with "Dag" (Acheson Colloids Corp.) colloidal graphite dispersed in alcohol. Although



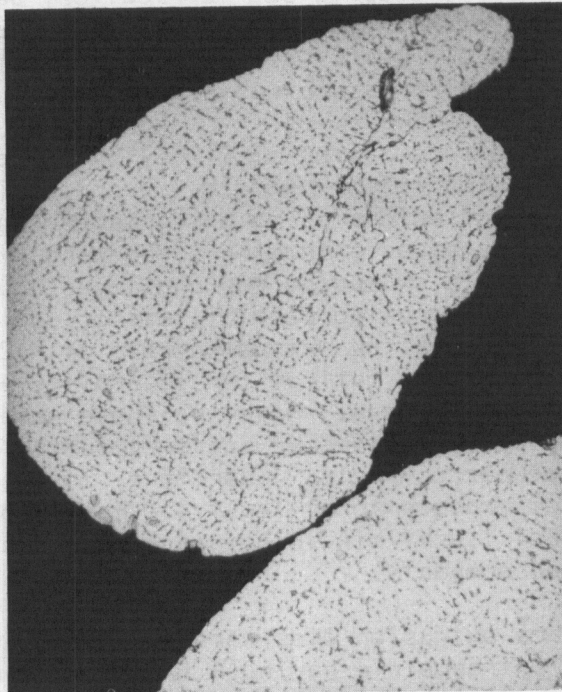


Fig. 3 Etched in 10% H<sub>3</sub>PO<sub>4</sub> 1000X

Microsection of a particle of metallized alloy powder, illustrating the extremely fine chill cast structure that was typical of these powders.

Alloy: Al + 4 $\frac{1}{4}$ % Mg + 1 $\frac{1}{4}$ % Mn.

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some graphite adheres to the surfaces of the completed compacts, careful examination provided no evidence of contamination of the interiors of the compacts, nor of the extrusions produced from them.

A study of the green densities of compacted billets as a function of applied cold compacting pressure led to the general conclusion that some compromise was still necessary, despite Dag lubrication, between a desirably high compacting pressure to effect good consolidation and a much lower pressure to avoid serious galling and die wear. As approximately the best compromise for cold compacting, 40,000 psi pressure was chosen.

Next, a series of billets compacted similarly by the above procedure were extruded at 12.8 extrusion ratio, using a conventional flat-face die, with different combinations of extrusion rate and temperature. A higher extrusion temperature and increased extrusion speed were required to obtain satisfactory surface quality in extruding the unalloyed aluminum powder compacts than were necessary in extruding cast billets of unalloyed aluminum. Previous experience had shown that an extrusion rate of 0.25 ft/min at an extrusion temperature of 795°F was optimum for laboratory apparatus extrusion of cast billets of unalloyed aluminum. However, these same extrusion conditions produced an unsatisfactory checked and torn surface on the extrusions from unalloyed aluminum powder compacts. Therefore, it was found necessary to raise the extrusion temperature to at least 850°F and to increase the extrusion speed to at least 1.0 ft/min to obtain a satisfactory extruded surface from the unalloyed compacts.

Conventional flat-face extrusion dies were employed with good success. In one trial of a flared-entrance die having a 1/8 in. lip radius, significantly higher extrusion pressure was required than for the conventional flat-face die. Furthermore, the surface of the extruded specimen was much rougher than that obtained with a conventional die under otherwise analogous extrusion conditions. In fact, the sharpness of the lip on flat-face dies was found to be important, since even slight wear and rounding of this edge produced readily detectable deterioration in the surface quality of the extruded specimens.

## 2. Mixtures of "pure" aluminum powders with alloying constituents.

The addition of 10 to 20% of alloying constituents (Table III) to the "pure" aluminum matrix powder resulted in decreased green densities after cold compacting. It was also found necessary to raise the extrusion temperature further, to 900°F (the maximum temperature for which the extrusion apparatus had been designed), in order to avoid checking and cracking of the extruded surfaces.

On the basis of the foregoing results, the following general procedure was selected as a tentative "standard" method for fabricating powder alloy specimens for studies of their properties:

- (1) "A" aluminum powder (see Table II) was sieved through -100 mesh to remove coarse powder clusters\*. Then, the desired

\* If these clusters were not removed, blistered extrusions resulted.

# Contrails

amount of powdered alloying constituent (Table III) was added, and the mixture was blended in a pigment mill. Before compacting, the blend was checked under the microscope for uniformity of constituent dispersion.

- (2) A 20 gram charge of the blended powders was cold compacted successively from each end at 40,000 psi pressure in a 0.620 in. diameter billet die previously lubricated with "Dag" colloidal graphite.
- (3) The green compact was charged into the extrusion container, whose temperature was controlled at 900°F, held five minutes for heating, and then extruded at 1.25 ft/min on a sharp-lipped flat-faced die of extrusion ratio 12.8:1. Dimensions of the extruded specimen were 0.175 in. diameter x 10 to 14 in. long.
- (4) Generally, the extruded specimens were straightened for subsequent mechanical tests by drawing through a conventional carbide wire die of 0.1718 in. diameter, giving a reduction in area of slightly less than 4%. This procedure also tended to eliminate minor surface imperfections which could have caused experimental difficulties in the dynamic modulus tests.

3. Pre-alloyed powders. Results obtained in fabricating pre-alloyed powders are still extremely limited and preliminary in nature. However, it has been observed that the billet densities after cold compacting at reasonable pressures tend to be discouragingly low, and the extrusions produced from such billets tend to be seriously blistered. It has, in fact, become evident that relatively radical improvements in the "standard" fabrication methods are essential.

In all probability, the best direction for achieving this improvement was suggested by the recent work of McKinnon<sup>(7)</sup>. His measurements showed a large, nearly discontinuous increase in the rate of sintering of aluminum and several aluminum alloys at temperatures crudely indicated\* to be in the neighborhood of 950° to 1000°F. Although not suggested by McKinnon, his observed drastic alteration in sintering behavior may be a consequence of structural transformation, at approximately this temperature, in the oxide films surrounding the powder particles, since there are other available data indicating such a transformation.

In any case, McKinnon's results were taken as evidence of the desirability of investigating hot pressing of the green billets at temperatures

\* For McKinnon's purpose, the specific temperature was unimportant. In fact, in a recently published version<sup>(8)</sup> of his original report, mention of the tests on aluminum has been completely omitted.

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at or near the 950°F-1000°F range prior to extrusion. For this purpose, the extrusion apparatus has recently been rebuilt to adapt it for hot pressing at temperatures up to approximately 1100°F. The redesign also incorporated the feature of hot forging (upsetting) the green billets by approximately 35% reduction in height to aid powder consolidation prior to extrusion. As a result of these procedural changes, hot pressed billet densities of essentially theoretical maximum appear to be obtained consistently. However, data are not yet available to permit conclusions regarding the effects of hot pressing on the properties of the extrusions produced from such billets.

## B. Properties of Powder Extrusions

The properties of powder extrusions prepared from the various grades of atomized "pure" aluminum powders (Table II) are summarized in Table IV. It is to be noted that the moduli and densities of these unalloyed powder extrusions are essentially equivalent and independent of the various powder grades employed in their preparation. However, an effect of powder grade is clearly indicated both in the as-compacted green densities and in the strengths, hardnesses, and ductilities of the resulting extrusions. In general, it appears that the green densities of the compacts are improved by increasing particle sizes and by increasing homogeneity in the particle size distributions. In the resulting extrusions, the finest powder grades (B, G, and L) produced the highest ultimate strengths and outstandingly high yield strengths combined with good elongations. The coarser powder grades produced lower ultimate and yield strengths, but tended, in general, to produce the most ductile extrusions.

Powder alloys have been prepared, using most of the proposed additions listed in Table I. The properties of these alloyed extrusions are summarized in Table V. Shown graphically in Figure 4 are the Young's moduli of the various alloys plotted as a function of the weight-percentage of alloying addition, while the corresponding specific modulus curves are presented in Figure 5.

It should be noted that all the alloying additions thus far tested have been effective in raising the modulus to at least some extent. These findings are in moderately good agreement with the theoretically predicted increase in all cases where these predictions could be made with sufficient precision to have significant meaning. The effect of the SiC and B<sub>4</sub>C additions were particularly outstanding in raising both the modulus and specific modulus. For example, the modulus improvement produced by a 20%, by weight, addition of SiC amounts to a gain of approximately 28% over the normal modulus for unalloyed aluminum powder extrusions. This can be compared with the approximately 17% maximum improvement in modulus achieved by Dudzinski<sup>(1)</sup> using other powder alloying additions.

None of the alloying additions except Cu had a particularly significant effect upon the tensile properties. This was not surprising, of course, since none of these additions, except Cu, is believed to exhibit important solid solubility in the aluminum matrix at any of the temperatures attained

TABLE IV

PROPERTIES OF EXTRUSIONS OF COMMERCIAL ATOMIZED ALUMINUM POWDERS  
FABRICATED BY THE "STANDARD" PROCEDURE

Powder Type	Green Density % of Theoretical Maximum	Surface Condition	Properties at Room Temperature -- as extruded condition					
			Density g/cc	Young's Modulus psi x 10 <sup>-6</sup>	UTS psi	YTS psi	% Elong in 2"	Hardness R <sub>15-T</sub>
A	90.4	Good	2.69	10.5	20,900	--	25.5	40
B	85.0	Sl. Blisters	--	--	30,300	21,700	28.5	60
C	93.3	Good	--	--	17,000	10,000	35.0	22
D	92.8	Good	--	--	17,700	11,000	33.5	21
E	93.0	Good	--	--	16,800	10,200	39.0	24
F	93.5	Good	--	--	18,700	11,900	34.0	29
G	88.5	Blisters	--	--	23,800	--	26.0	43
H	91.5	Good	--	--	18,900	12,500	36.0	29
J	94.8	Good	2.70	10.6	16,300	--	26.5	19
K	94.4	Good	2.70	10.6	16,600	12,400	30.5	23
L	93.6	Good	2.70	10.5	19,700	17,200	26.5	36

TABLE V  
PROPERTIES OF POWDER ALLOY EXTRUSIONS FABRICATED BY THE "STANDARD" PROCEDURE

Alloy Composition (weight %)	Surface Quality Obtained on Extruded Specimens	Specific Gravity			Young's Modulus $\times 10^{-6}$ (psi)		Specific Modulus $\times 10^{-6}$ (psi)		Tensile Properties	
		Theoretical cal.(b)	As Compacted	As Extruded	Theoretical(c)	As Extruded	As Extruded	As Extruded	UTS(psi)	YTS(psi)
A(a)	Good	2.70	2.47	2.694	-----	10.5	3.90	20,900	-----	25.5
A + 10 B <sub>4</sub> C	Slightly Rough	2.68	2.37	2.681	16.5-11.5	12.2	4.55	25,600	23,100	7.0
A + 20 B <sub>4</sub> C	Slightly Rough	2.66	2.38	-----	22.3-12.8	-----	-----	-----	-----	-----
A + 10 $\alpha$ Al <sub>2</sub> O <sub>3</sub>	Unsatisfactory	2.78	2.31	-----	13.5-11.2	-----	-----	-----	-----	-----
A + 10 TiC	Slightly Rough	2.83	2.54	2.823	12.6-11.0	11.7	4.15	25,700	22,800	19.0
A + 15 TiC	Slightly Rough	2.90	2.57	-----	13.8-11.3	-----	-----	-----	-----	-----
A + 20 TiC	Unsatisfactory	2.97	2.63	-----	14.9-11.6	-----	-----	-----	-----	-----
A + 5 SiC(d)	Good	2.72	2.41	2.726	11.0-10.7	11.4	4.18	25,800	22,600	20.0
A + 10 SiC(d)	Good	2.74	2.43	2.744	11.4-11.0	12.3	4.48	25,400	-----	5.0
A + 15 SiC(d)	Good	2.77	2.40	2.760	11.9-11.2	13.0	4.71	-----	-----	-----
A + 20 SiC	Slightly Rough	2.79	2.40	2.762	12.3-11.5	13.4	4.85	27,200	24,400	8.5
A + 30 SiC	Unsatisfactory	2.84	2.34	-----	13.3-12.1	-----	-----	-----	-----	-----
A + 10 Si	Good	2.65	2.36	2.659	11.1-10.9	11.2	4.21	24,700	22,400	11.0
A + 15 Si	Slightly Rough	2.63	2.30	-----	11.4-11.2	-----	-----	-----	-----	-----
A + 20 Si	Unsatisfactory	2.62	2.25	-----	11.7-11.4	-----	-----	-----	-----	-----
A + 30 Si	Unsatisfactory	2.58	2.15	-----	12.3-11.8	-----	-----	-----	-----	-----
A + 10 ZrO <sub>2</sub>	Slightly Rough	2.84	2.51	2.816	11.8-10.9	10.8	3.84	24,800	21,900	15.0
A + 10 WC	Good	2.94	2.57	2.852	12.2-10.7	10.8	2.79	24,900	22,200	18.5
A + 20 WC	Slightly Rough	3.24	2.90	-----	14.3-10.9	-----	-----	-----	-----	-----
A + 30 WC	Unsatisfactory	3.59	3.16	-----	16.9-11.2	-----	-----	-----	-----	-----
A + 10 TiAl	Good	2.78	2.52	2.800	11.2-10.9	11.1	3.96	23,500	20,200	16.5
A + 20 TiAl	Slightly Rough	2.87	2.59	2.898	11.9-11.3	11.3	3.90	24,800	22,700	5.0
A + 30 TiAl	Slightly Rough	2.96	2.64	-----	12.7-11.8	-----	-----	-----	-----	-----
A + 10 Cr	Good	2.88	2.59	2.878	11.5-10.8	11.2	3.89	22,800	13,100	30.0
A + 20 Cr	Good	3.08	2.74	3.083	12.6-11.2	11.7	3.80	26,200	23,400	16.5
A + 30 Cr	Slightly Rough	3.32	2.92	-----	14.0-11.7	-----	-----	-----	-----	-----

TABLE V  
 PROPERTIES OF POWDER ALLOY EXTRUSIONS FABRICATED BY THE "STANDARD" PROCEDURE  
 (CONTINUED)

Alloy Composition (weight %)	Surface Quality Obtained on Extruded Specimens	Specific Gravity		Young's Modulus $\times 10^{-6}$ (psi)		Specific Modulus $\times 10^{-6}$ (psi)		Tensile Properties As Extruded Condition		Per Cent Elong. in. 2"
		Theoretical (cc) - As Compacted	As Extruded	Theoretical (cc) - As Extruded	As Extruded	UTS (psi)	YTS (psi)	UTS (psi)	YTS (psi)	
A + 10 Mo	Unsatisfactory	2.91	2.60	11.6-10.8	---	---	---	---	---	---
A + 15 Mo	Unsatisfactory	3.04	2.72	12.3-10.9	---	---	---	---	---	---
A + 20 Mo	Good	3.17	2.85	13.0-11.0	11.3	3.60	---	---	---	---
A + 10 MnAl6	Good	2.75	2.45	---	11.0	4.00	23,000	19,600	---	15.0
A + 20 MnAl6	Slightly Rough	2.80	2.44	---	11.0	3.94	---	---	---	---
A + 10 FeAl3	Good	2.78	2.48	---	11.1	3.98	25,000	22,300	---	16.5
A + 20 FeAl3	Good	2.87	2.52	---	11.2	3.93	25,600	23,000	---	9.0
A + 30 FeAl3	Unsatisfactory	2.96	2.56	---	---	---	---	---	---	---
A + 10 Cu	Good	2.91	2.60	10.7-10.6	10.8	3.77	25,600	20,300	---	13.0
A + 20 Cu	Good	3.14	2.80	10.9-10.8	11.1	3.52	---	---	---	---
A + 30 Cu	Good	3.41	3.06	11.1-11.0	11.3	3.51	35,000	---	---	8.0
A + 50 Cu	Unsatisfactory	4.17	3.62	11.8-11.4	---	---	---	---	---	---

Notes: (a) "A" grade atomized aluminum powder.  
 (b) Calculated from the "rule of mixtures".  
 (c) Calculated for two extreme types of hypothetical microstructural distributions of the addition constituent as follows: | - Addition phase assumed distributed as longitudinal fibers, and || - Addition phase assumed distributed as transversely oriented platelets.  
 (d) Extruded at 850°F instead of "standard" 900°F.

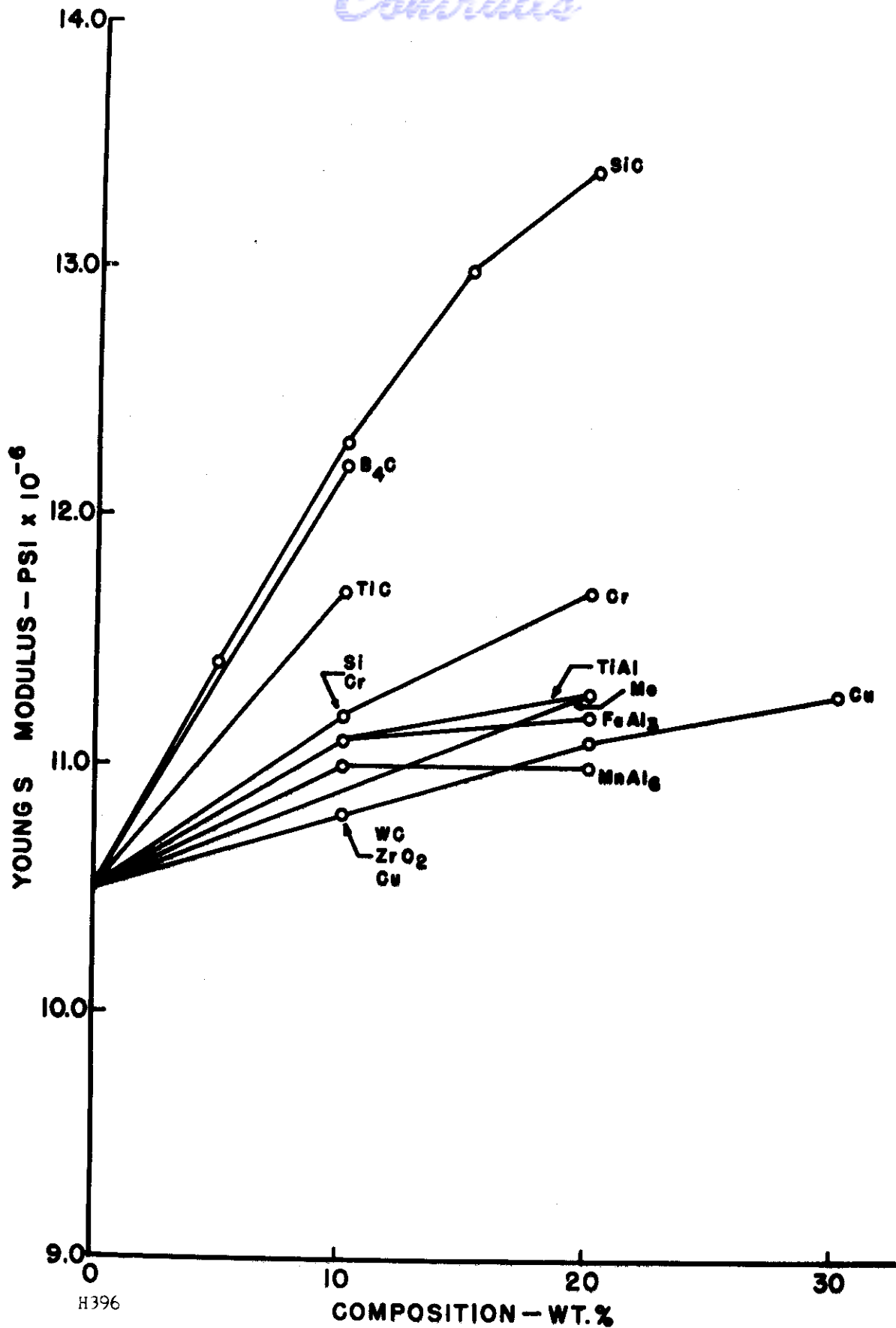
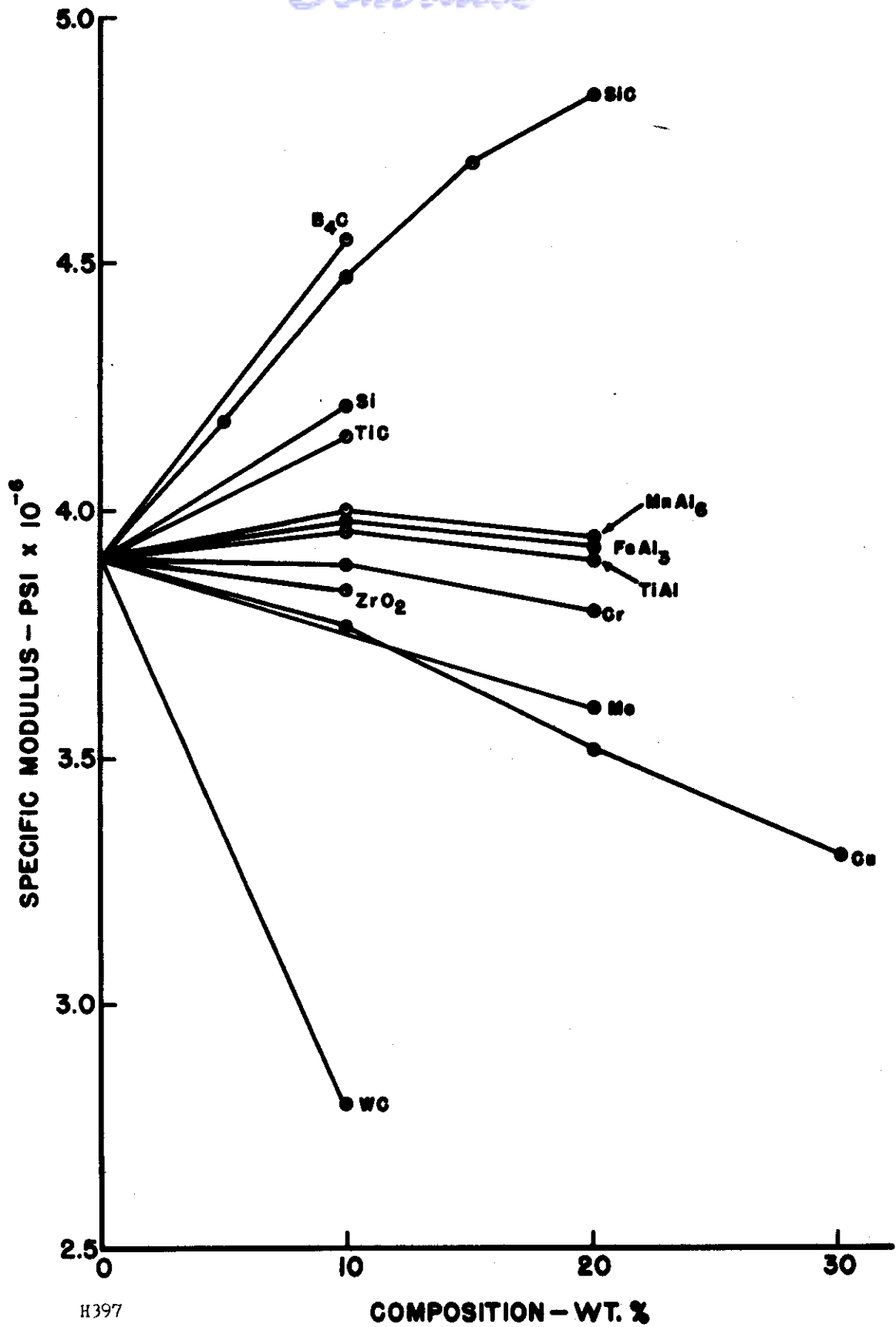


Fig. 4 Young's Modulus of Aluminum-base Powder Alloys as a function of Various Alloying Additions.





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Fig. 5 Specific Modulus of Aluminum-base Powder Alloys as a function of Various Alloying Additions.

# Contrails

during fabrication. Consequently, they could be expected to harden the matrix only by means of the mechanism of dispersion hardening. However, the microstructural dispersion of the particles of the added compounds achieved in these alloys was probably not sufficiently fine for truly effective dispersion hardening\*.

The use of pre-alloyed powders as a matrix for refractory dispersions of the SiC and B<sub>4</sub>C is expected to result in improved tensile properties while still retaining the improved modulus. Unfortunately, the period of time required to procure the pre-alloyed powders prevented the testing of this hypothesis during the term of the contract.

Short-time tensile properties at 600°F were determined for the single case of a powder alloy of Al + 20% by volume SiC, for general exploratory purposes. Results are shown in Table VI.

TABLE VI

TENSILE PROPERTIES AT 600°F OF POWDER EXTRUSIONS OF  
Al + 20% BY VOLUME SiC, AS-EXTRUDED CONDITION

Specimen No.	Test Temperature (°F)	Ultimate Strength (psi)	Proportional Limit* (psi)	Elong. in 1 in. (8D) (%)	Reduction of Area (%)
503**	620	9000	7700	11	26
505***	600	9500	8100	12	28

\* Approximate value from load vs. time curve

\*\* Prepared using No. 400 grit Buehler SiC

\*\*\* Prepared using No. 500B Norton Crystolon (SiC)

As far as can be judged from this limited test, the properties of the Al-SiC powder alloy at 600°F appear to be approximately comparable to those of the Al + 0.5% Al<sub>2</sub>O<sub>3</sub> powder alloy designated A.P.M.P.-M255<sup>(9)</sup>. Since the

\* The great difference in hardnesses between the matrix and the refractory additions in these powder alloys causes such excessive relief effects during metallographic preparation (even when diamond dust laps are employed) that photomicrographs which would be satisfactorily illustrative of the microstructures could not be obtained for presentation in this report.

# Conclusions

Al-SiC alloy probably contains an amount of  $Al_2O_3$  that is approximately equivalent to that present in M255, the conclusion seems inescapable that the 20%, by volume, of SiC addition is not particularly effective in significantly raising the elevated temperature strength. It is possible that the reason for this mediocre behavior lies in the fact that the degree of microstructural dispersion of the SiC constituent is relatively coarse, as compared to the fineness of the dispersion of the  $Al_2O_3$  in the Al- $Al_2O_3$  type powder alloys. This, too, suggests that further attempts should be made to compound the powder alloys of this project from the finest possible sizes of matrix and refractory powders. In the past, attempts to utilize extremely fine particle sizes have led to unsound fabricated specimens; however, it is believed that the recently developed hot pressing procedures may overcome these previous fabrication difficulties.

## IV. CONCLUSIONS

1. Aluminum-base powder alloys have been successfully prepared containing additions of various intermetallic and refractory compounds such as  $B_4C$ , SiC, TiC, TiAl, etc., which are not alloyable with aluminum by conventional methods.

2. Outstanding improvements in the Young's modulus of aluminum were produced by SiC and  $B_4C$  additions, amounting in the case of a 20%, by weight, addition of SiC to an increase in modulus of nearly 28%. TiC also effected relatively large improvements in modulus per unit of alloying addition, and merits further study.

3. The tensile strengths of these powder alloys were not outstanding in comparison to those of conventional alloys, presumably because the refractory and intermetallic additions were dispersed in a matrix of unalloyed aluminum, and because the dispersion of refractory particles was not sufficiently fine in scale.

4. Pre-alloyed aluminum alloy powders have been prepared by several methods to use in providing a stronger matrix in the powder alloys.

5. Fabrication of powder alloys using pre-alloyed matrix powders appears feasible by means of a hot pressing technique.

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