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STUDY OF INTERMETALLIC COMPOUNDS TASK A: DISPERSION-HARDENED TI--ETC(U)

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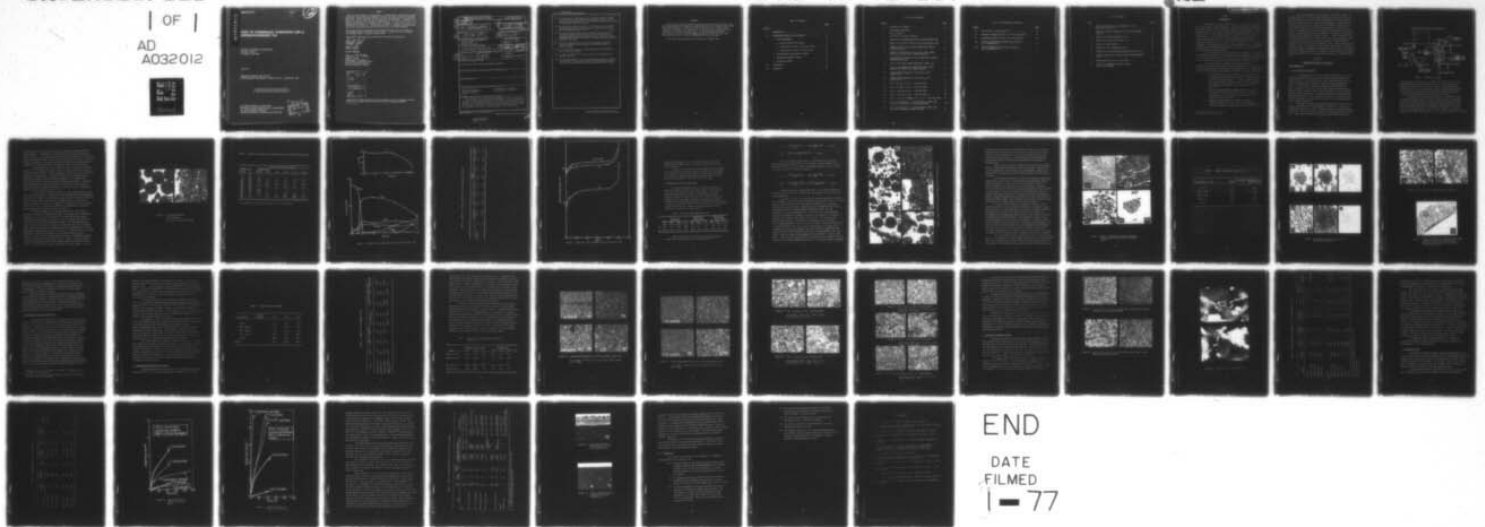
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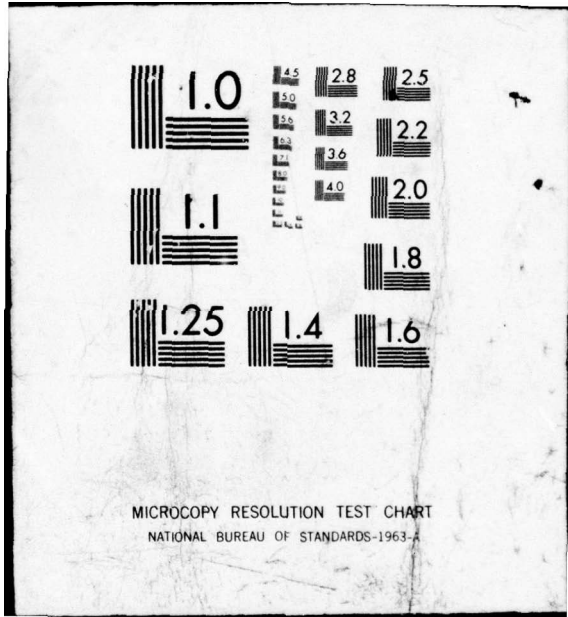
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**STUDY OF INTERMETALLIC COMPOUNDS TASK A:  
DISPERSION-HARDENED TIAI**

*BATTELLE COLUMBUS LABORATORIES  
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JULY 1976

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INTERIM REPORT FOR PERIOD 17 FEBRUARY 1975 - 16 FEBRUARY 1976

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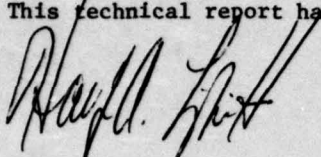
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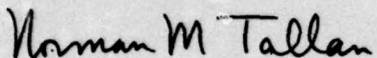
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The feasibility of increasing the high-temperature tensile and creep strength, the high-temperature oxidation resistance, and the low temperature ductility of the titanium aluminide, TiAl, by means of a dispersion of inert particles of yttria, has been studied. Alloys containing 1.5 and 3 volume percent Y <sub>2</sub> O <sub>3</sub> were produced by attritor milling of TiAl and Y <sub>2</sub> O <sub>3</sub> powders and hot extruding to bar stock. The major findings of the study are:  y			

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- (1) Attritor milling of TiAl powders for total milling times in excess of 100 hrs can be accomplished without problems. Milling for more than 80 hrs was required to achieve a uniform dispersion of yttria with the REP powder used in this program.
- (2) The oxygen and nitrogen contents of TiAl increased with increasing attritor milling time, and reached steady levels of 1300 ppm and 250 ppm respectively after 20-30 hrs.
- (3) The microstructures of the  $Y_2O_3$ -containing TiAl alloys comprised a predominantly basket-weave structure of  $\gamma$  and lamellar  $\alpha_2$ , with *gamma* *alpha 2* uniformly dispersed particles, apparently of  $\alpha_2$ , which were largely associated with the yttria dispersion. Few free yttria particles were observed in the alloy.
- (4) The yttria-containing alloys exhibited considerably increased tensile strength at room temperature and at 1652°F compared to yttria-free TiAl.
- (5) The tensile creep strengths of  $Y_2O_3$ -containing TiAl and  $Y_2O_3$ -free TiAl at 900°C were similar.
- (6) No effect was observed of the dispersoid on the room-temperature ductility of TiAl.
- (7) The oxidation behavior of the yttria-containing alloys was variable, some specimens oxidizing more slowly than the equivalent dispersion-free TiAl specimens, and most specimens exhibiting adherent scale. ↑

## PREFACE

This Interim Technical Report was prepared by the Metal Science Section, Metallurgy Department of Battelle Columbus Laboratories, Columbus, Ohio 43201, under AF Contract No. F33615-75-C-1168, Project No. 7021, "Structure and Properties of Solids", and administered by the Metals and Ceramics Division of the Air Force Materials Laboratory, Air Force Wright Aeronautical Laboratories, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, with Dr. Harry A. Lipsitt (AFML/LLM) as Project Engineer.

This report was submitted by the authors on 30 April 1976.

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## SECTION I

INTRODUCTION

The titanium aluminide, TiAl, possesses a combination of properties which make it an attractive candidate for structural components in advanced gas turbine engines. This intermetallic compound exhibits a high strength-to-density ratio, a high specific modulus, oxidation resistance up to about 1600°F (871°C), but very limited ductility at temperatures below about 1263°F (700°C). This lack of ductility constitutes a major drawback to the successful application of TiAl. Consequently an improvement in room temperature ductility from the current 1 percent elongation to 2 - 4 percent elongation is an important goal of this alloy development study. In addition, an increase in the oxidation resistance of TiAl up to 1800 to 1900°F (982 to 1038°C) would be highly advantageous.

The major objectives of this program were to implement the concepts of dispersion hardening and ductilizing of titanium aluminides by incorporation of a fine uniform dispersion of  $Y_2O_3$  particles, and to determine the extent to which dispersed particles improve the high temperature oxidation behavior.

It is well known that dispersed inert oxide particles can strengthen metals and alloys, and often the strengthening can be attributed to the Orowan mechanism of hardening by nondeforming particles. Research over the past decade has demonstrated additional benefits of dispersed particles:

1. Thermomechanical processing of dispersion-containing Ni-Cr, Co-Cr and Fe-Cr base alloys can result in a highly elongated recrystallized grain structure. At elevated temperatures, where grain boundary sliding is a major mode of deformation, this provides strengthening via the grain aspect ratio effect<sup>(1,2)\*</sup>.
2. Particle ductilizing has been demonstrated in Fe-ThO<sub>2</sub><sup>(3)</sup> and Cr-ThO<sub>2</sub><sup>(4)</sup> alloys, probably as a result of particles dispersing slip such that stress concentrations at crack nucleation sites are lowered.
3. Dispersed oxides improve the oxidation resistance of chromia-forming Ni-Cr, Co-Cr, and Fe-Cr base alloys by slowing the kinetics and enhancing oxide scale adhesion<sup>(5,6)</sup>.

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\*References are listed on page 40.

The proposed approach was essentially a feasibility study on one base material, TiAl (64Ti-36Al, weight percent) to assess the validity of the strengthening, ductilizing and enhanced oxidation resistance concepts. The first step was to obtain a final uniform dispersion of approximately 1.5 and 3 volume percent  $Y_2O_3$  particles by mechanically alloying  $Y_2O_3$  with TiAl powder produced by the rotating electrode process, followed by hot extrusion. Dispersion-free TiAl was processed in the same way, the purpose being to obtain extruded material to determine if mechanical alloying led to significant contamination (particularly oxygen). It has been shown<sup>(8)</sup> that it is virtually impossible to separate (in a Ti- $Y_2O_3$  alloy) oxygen which is in solid solution from that in the  $Y_2O_3$  dispersion. Characterization included optical and electron metallographic studies, chemical analyses, and tension testing at room temperature and elevated temperature, and selected screening studies to determine high temperature creep strength and isothermal and cyclic oxidation resistance. The results of these studies were compared with those obtained at the Aerospace Research Laboratory on dispersion-free TiAl.

## SECTION II

### EXPERIMENTAL RESULTS AND DISCUSSION

#### ALLOY PREPARATION

##### a. Attritor Milling Procedures

The experimental attritor mill assembly was based on a Szegvari 1-SB machine modified to operate under a purified helium atmosphere, as shown in Figure 1. The helium used was Airco commercial grade, which was dried by passage through a column of anhydrous  $CaSO_4$  and deoxygenated ( $P_{O_2} = 2.3 \times 10^{-45}$  atm) by passage through titanium turnings held at 615°C. The helium lines were of copper tubing except for connections to glassware, where a minimum amount of Tygon connection was used to minimize ingress of air by diffusion. As a further precaution the whole apparatus was maintained under a positive pressure of helium of 2 cm of silicone oil. The water jacket of the attritor mill was kept at 5°C by recirculating the water through a cooling unit.

All powder handling and weighing operations were carried out in a helium-filled glove-box. Before loading, the attritor mill system was flushed with helium for 24 hours. A glass container was filled with powder and sealed in the glove-box, then attached to the input leg of the attritor mill. After twice evacuating and backfilling with purified helium the

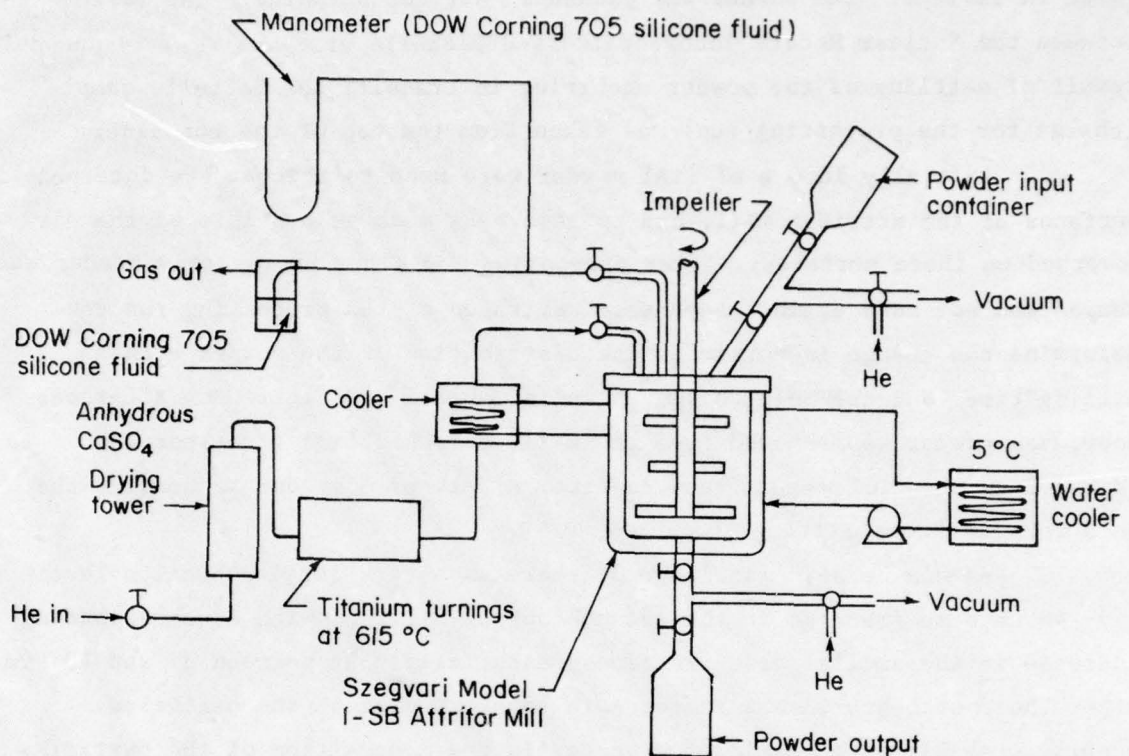


FIGURE 1. ATTRITOR MILL ASSEMBLY

connection between the valves on the container and attritor mill, both valves were opened and the powder was admitted to the mill. After filling was completed the input valve on the mill was closed. The unloading procedure was the reverse of the loading procedure, an empty container being connected to the output leg of the attritor mill and twice evacuated and backfilled with purified helium before powder was admitted from the mill.

Initial attritor milling studies were made to determine the change in oxygen and nitrogen content of TiAl with milling time, and to investigate the extent of mechanical alloying of  $Y_2O_3$  with TiAl as a function of milling time, so that a rational basis for choosing the processing conditions of the dispersion hardened alloys could be established. Thirty pounds of -35 mesh Ti-35.5 Al alloy powder (Batch No. 395), hereafter referred to as TiAl, was obtained from Nuclear Metals Incorporated. This alloy was prepared by the rotating.

electrode process in helium, and exhibited a typically spherical particle shape (Figure 2). The particle size analysis of the as-received powder is given in Table 1. The powder was packed in air for shipping. The difference between the Nuclear Metals Incorporated and Battelle size analyses is probably a result of settling of the powder occurring in transit; the Battelle sample (charge for the precoating run) was taken from the top of the container.

Initially 1000 g of TiAl powder were used to precoat the internal surfaces of the attritor mill, and to remove as much as possible of the air absorbed on these surfaces. After precoating for eight hours, this powder was dumped and not used again. Advantage was taken of the precoating run to determine the change in particle size distribution of the powder with milling time, and the results are shown in Table 1 and Figure 3. After one hour, the powder was removed from the mill, screened in a glove box and returned to the mill, again removed after a further one hour to provide the data for two hours milling time, and so on.

As can be seen in Figure 3, there is a very large reduction in the 149-240  $\mu\text{m}$  size fraction in the second hour of milling, with a corresponding increase in the smaller size fractions, especially that between 37 and 105  $\mu\text{m}$ . After the fourth hour, some increase in apparent size of the particles occurs, probably as a result of a change in the composition of the particles from broken pieces of the original spheres to conglomerates of shattered spheres cold-welded together, as shown in Figure 2.

After precoating, a run was made with 1200g of TiAl powder to determine the uptake of oxygen and nitrogen with milling time. Samples were taken after 1, 5, 10, 20, 40, and 80 hours, these milling times being accumulated during daytime running only, since the machine was shut down overnight to avoid unattended running problems. The resulting analyses, shown in Table 2 and Figure 4, indicate a relatively sharp rise in analyzed oxygen and nitrogen levels with milling times up to approximately 20 hours, after which a leveling off occurs as a plateau or saturation level is reached. After 80 h, the mill was allowed to run continuously with the aim of sampling at 160 h, but after approximately 20 hours of continuous running a build-up of powder occurred such that the mill began to vibrate violently as the ball charge and powder became trapped between the impeller and the walls. At this point the run was stopped and the charge dumped. The very

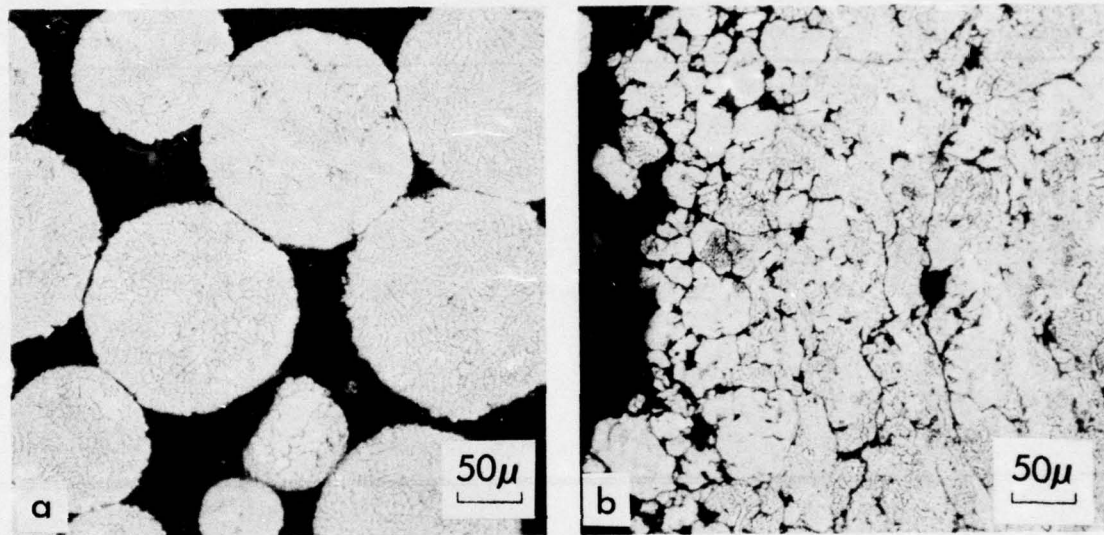


FIGURE 2. TiAl POWDER PARTICLES

(a) As received

(b) After 3 h attritor milling

TABLE 1. CHANGE IN PARTICLE SIZE DISTRIBUTION OF TiAl WITH ATTRITOR MILLING

SCREEN SIZE		PERCENTAGE RETAINED ON SCREEN					
		AS RECEIVED		1 hr.	2 hr.	4 hr.	8 hr.
US MESH	$\mu\text{m}$	VENDOR	BCL*				
35	500	0	-	-	-	-	-
45	354	0.4	-	-	-	-	-
60	250	2.8	0.1	0.1	0.1	0.1	0.4
80	177	27.9	-	-	-	-	-
100	149	-	89.6	84.1	2.6	1.7	14.0
120	125	41.5	-	-	-	-	-
140	105	-	1.8	9.5	21.4	17.9	20.7
170	88	10.5	-	-	-	-	-
230	63	10.8	-	-	-	-	-
325	44	5.0	-	-	-	-	-
400	37	1.1	8.2	6.1	60.9	65.6	53.7
< 400	< 37	-	0.3	0.2	15.0	14.8	11.3

\* This sample is the same as that after 1, 2, 4, and 8 hrs.

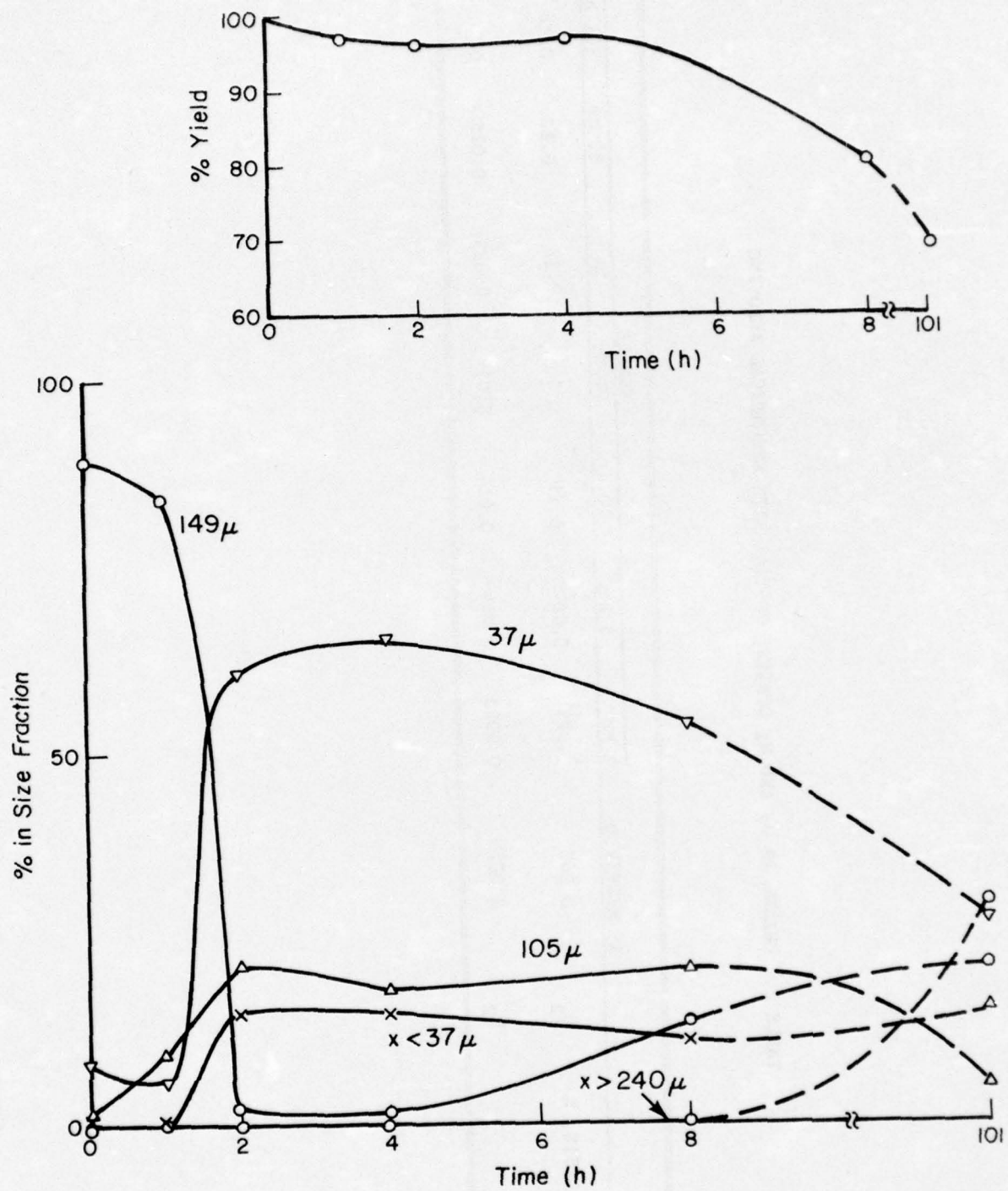


FIGURE 3. CHANGE IN TiAl POWDER PARTICLE SIZE WITH MILLING TIME

TABLE 2. CHANGE IN O<sub>2</sub> AND N<sub>2</sub> CONTENT OF TiAl WITH MECHANICAL ALLOYING

ANALYSIS, %	AS RECEIVED		SAMPLE						
	1 hr	5 hr.	10 hr.	20 hr.	40 hr.	80 hr.	101 hr.		
O <sub>2</sub>	0.067	0.075	0.087	0.11	0.10	0.12	0.13	0.36	
N <sub>2</sub>	0.0075	0.0095	0.014	0.014	0.019	0.023	0.025	0.14	

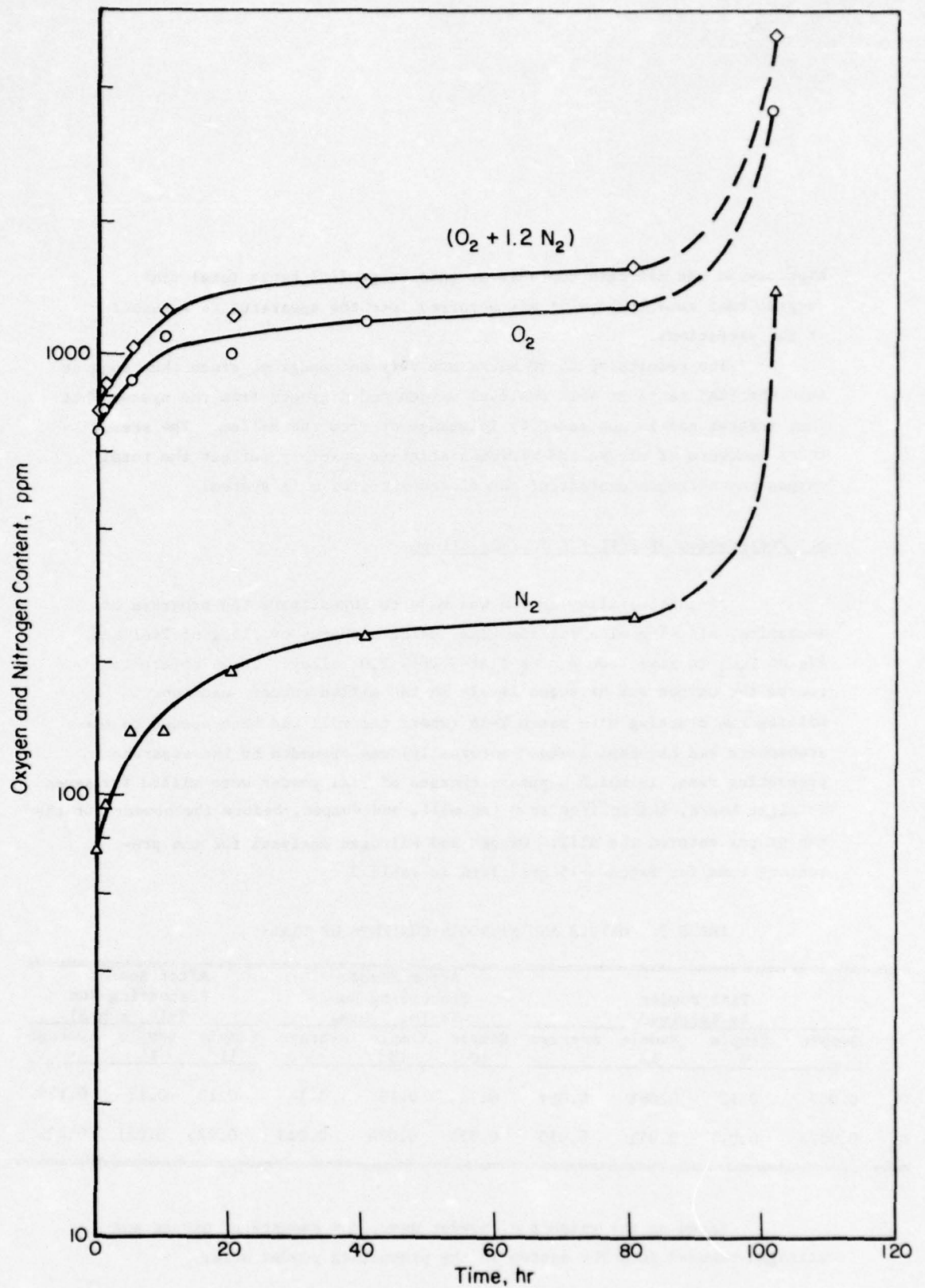


FIGURE 4. CHANGE IN O<sub>2</sub> AND N<sub>2</sub> CONTENT OF TiAl WITH MILLING TIME

high oxygen and nitrogen contents at this stage (101 hours total time) suggest that some leakage of air occurred into the apparatus as a result of the vibrations.

The results up to 80 hours are very encouraging, since they suggest that the TiAl takes up some residual oxygen and nitrogen from the system, but that further gas is not added by inleakage or from the helium. The steady state contents of oxygen and nitrogen attained possibly reflect the total oxygen and nitrogen content of the closed attritor mill system.

b. Preparation of TiAl + 3v/o Y<sub>2</sub>O<sub>3</sub> Alloys

An initial alloying run was made to investigate the progress of mechanical alloying with milling time, using a charge of 1151g of TiAl and 49g of Y<sub>2</sub>O<sub>3</sub> to give 1200 g of a TiAl-3 vol% Y<sub>2</sub>O<sub>3</sub> alloy. In an effort to reduce the oxygen and nitrogen levels in the milled powder, each new milling run starting with Batch T-16 (where the mill had been opened to the atmosphere and had been cleaned internally) was preceded by two separate precoating runs, in which separate charges of TiAl powder were milled for seven to eight hours, shaken free from the mill, and dumped, before the powder for the run proper entered the mill. Oxygen and nitrogen analyses for the pre-coating runs for Batch T-16 are given in Table 3.

TABLE 3. OXYGEN AND NITROGEN CONTENTS OF TiAl

	TiAl Powder As Received				After First Precoating Run (T-16, 7 hrs)			After Second Precoating Run (T-16, 8 hrs)		
	Sample 1	Sample 9	Sample 12	Average	Sample 10	Sample 13	Average	Sample 11	Sample 14	Average
O <sub>2</sub>	0.067	0.12	0.081	0.089	0.15	0.13	0.14	0.13	0.12	0.125
N <sub>2</sub>	0.0075	0.011	0.011	0.010	0.022	0.024	0.023	0.025	0.021	0.023

Based on the weights of powder used, the amounts of oxygen and nitrogen removed from the system in the precoating powder were:

$$O_2: \frac{878 (0.13-0.089)}{100} + \frac{1120 (0.125-0.089)}{100} = 0.851 \text{ g}$$

$$N_2: \frac{(878 + 1120) (0.023-0.010)}{100} = 0.260 \text{ g}$$

If the precoating powder used in a previous run to determine oxygen and nitrogen uptake with time are assumed to have attained 0.14%  $O_2$  and 0.023%  $N_2$ , then the total oxygen and nitrogen taken up from the system in 80 hrs of attritor milling time would be:

$$O_2: \frac{799 (0.14-0.089)}{100} + \frac{1200 (0.13-0.089)}{100} = 0.899 \text{ g}$$

$$N_2: \frac{099 (0.023-0.010)}{100} + \frac{1200 (0.025-0.010)}{100} = 0.284 \text{ g}$$

Using these data it appears that most of the residual oxygen and nitrogen can be removed from the attritor mill system by the use of a pre-coating technique.

In order to obtain the best possible initial dispersion of yttria, the TiAl powder was attritor milled alone for three hours to reduce its overall particle size (see Figure 2b) before being blended with the yttria and returned to the attritor mill. The mill was then run during daytime only, with samples removed for metallographic examination after the same intervals as in the contamination run. Cross sections of mounted powder particles are presented in Figure 5, and show the progress of mechanical alloying with residence time in the attritor mill. The size fractions represented in Figure 5 are probably not typical because of settling effects during mounting. After one hour, the powder consists predominantly of spherical particles as found in the original powder, with some small fragments and a few large, composite particles which have apparently been compacted onto and then removed from the walls or the surfaces of the ball charge. After two hours, much fragmentation of particles is evident, and after 5 hours, mechanical alloying can be observed of such fragments to the surfaces of the larger remaining particles of the original powder. Spherical

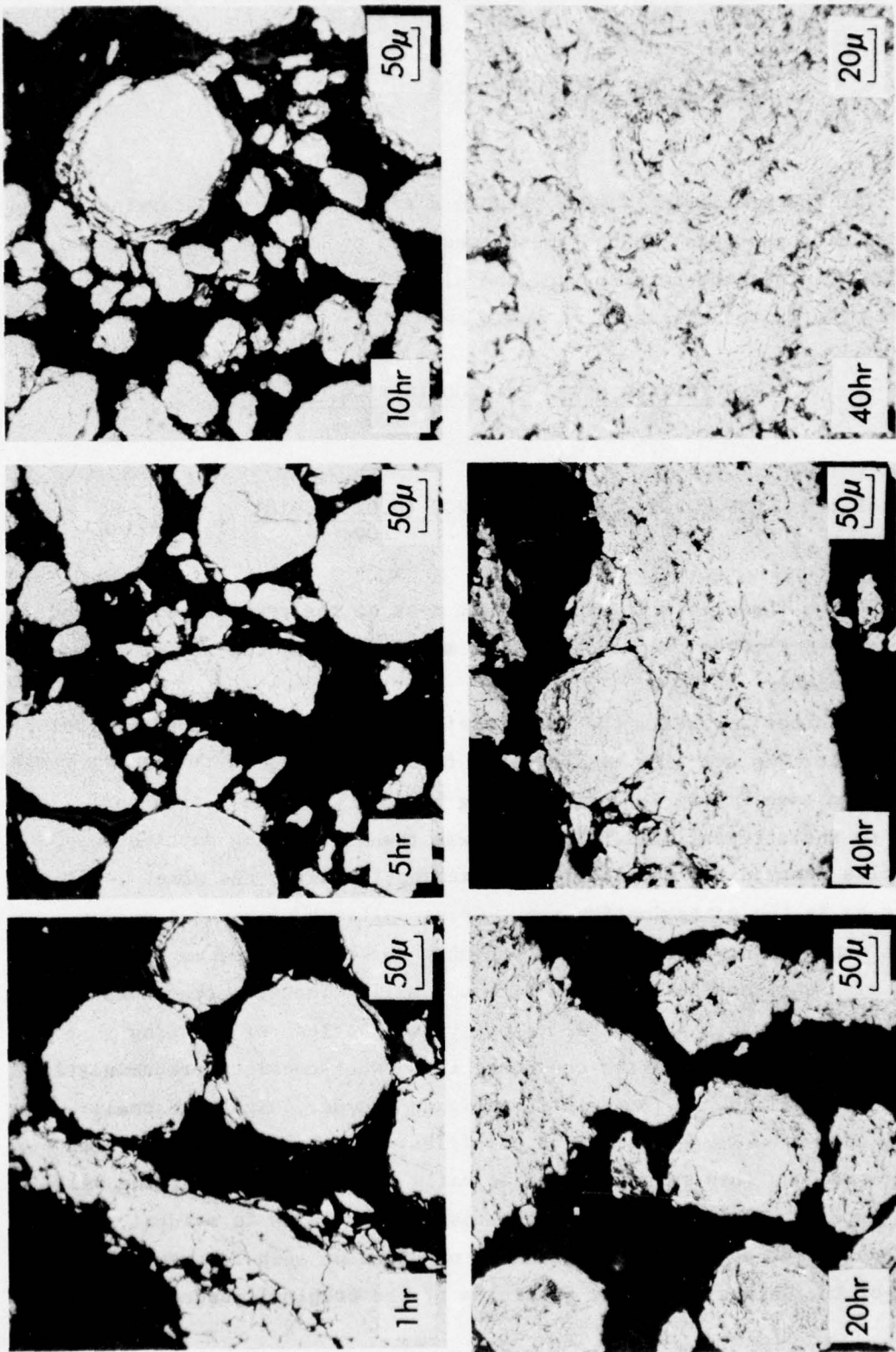


FIGURE 5. CHANGES IN TiAl-3% Y<sub>2</sub>O<sub>3</sub> ALLOY POWDER WITH TIME OF ATTRITOR MILLING

particles of TiAl, some without a mechanically alloyed rim, can still be distinguished after 20 hours, but after 40 hours, the bulk of the powder consists of large composite particles with an occasional unalloyed TiAl particle discernable (Figure 5). Also after 40 hours the mechanically alloyed powder itself still contains areas some 20  $\mu\text{m}$  in diameter of unalloyed TiAl.

After 51 hours, the drive coupling to the attritor mill impeller sheared, although without breaking the seal to the powder. However, when it proved impossible to replace the drive coupling without opening the attritor mill and exposing the powder charge to ambient air, this run was terminated.

In a subsequent alloying run (Batch T-16), samples of powder were removed after 56 hrs, 68 hrs, 79 hrs and 85 hrs and mounted for metallographic examination. As can be seen from the sections in Figure 6, pieces of unalloyed TiAl particles are still discernable in the powder after 56 and 68 hrs, but after 79 hrs mechanical alloying is nearly complete. The final powder, after 85 hrs milling, is considerably finer than the original TiAl powder, and in fact the larger size fractions, indicated in Table 4, comprised conglomerates of particles in the small size fractions which were welded together. The uniformity of the yttria dispersion in the final powder can be judged from the X-ray images in Figure 7.

A further batch of TiAl-3 vol.%  $\text{Y}_2\text{O}_3$  powder (Batch T-17) was milled for 100 hrs to provide sufficient material for subsequent testing, and the structure of this powder after 75 and 100 hrs is shown in Figure 8. The particle size of Batch T-17 powder after 100 hrs of milling was exceedingly fine (approximately 100% less than 37  $\mu\text{m}$ ), and very few pieces of unalloyed TiAl could be observed. The dispersion of yttria in this powder is apparently very fine and uniform (Figure 7). However, towards the end of the T-17 run, an external oil sealing ring on the impeller drive shaft of the attritor mill worked loose, and examination of the internal oil seals and oil trap after the end of the run raised the possibility that some contamination of the powder by oil may have occurred, even though the powder was free-flowing and had not adhered to the walls of containers.

As a precautionary measure, another batch (T-18) of the same alloy was run. After 56 hrs of attritor milling in Run T-18, an 'O'-ring retaining disc on the underside of the lid of the mill was lost, allowing some off-axis movement of the impeller shaft, although a positive helium pressure was

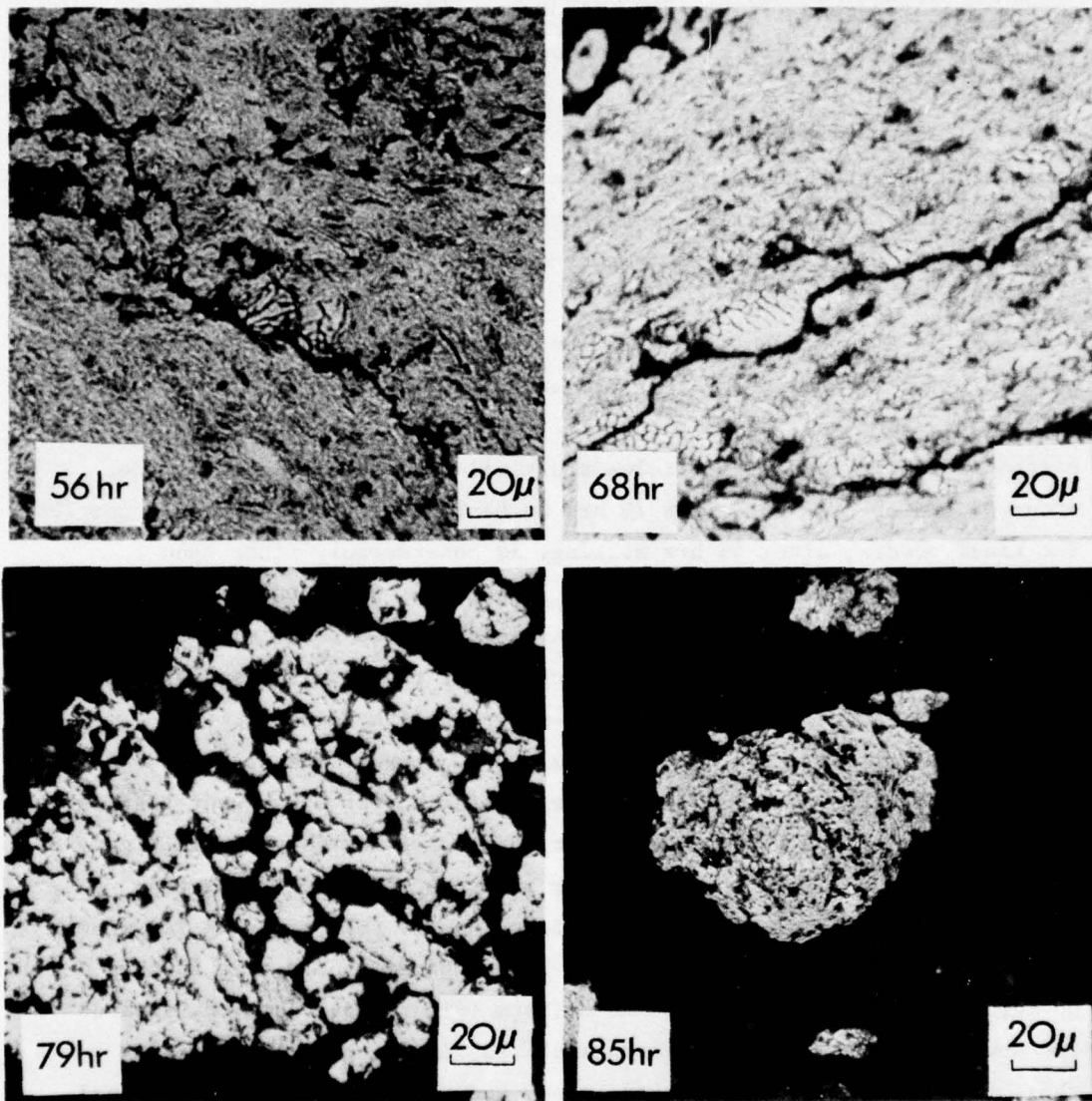


FIGURE 6. CHANGES IN STRUCTURE OF TiAl-3% Y<sub>2</sub>O<sub>3</sub> ALLOY POWDER (BATCH T-16) WITH TIME OF ATTRITOR MILLING

TABLE 4. POWDER SIZE ANALYSIS FOR BATCH T-16

Size Fraction, x, ( $\mu\text{m}$ )	Percentage of Fraction	
	Start	Finish (83 hrs)
x > 240	1.0	21.5
240 >x> 149	42.2	18.2
149 >x> 105	30.2	8.1
105 >x> 37	23.2	39.9
x < 37	3.4	12.3

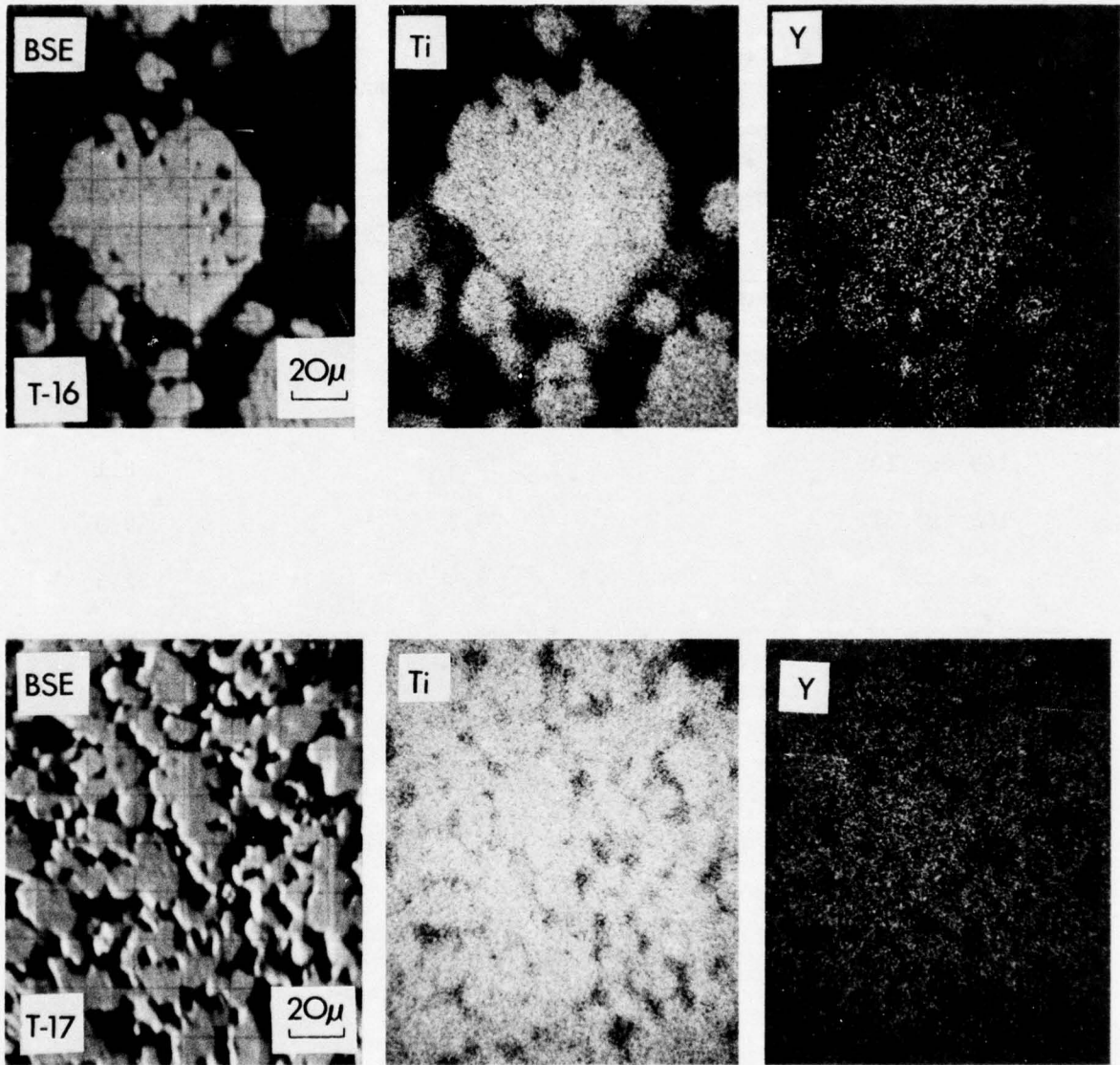


FIGURE 7. DISTRIBUTION OF YTTRIUM IN ALLOY POWDERS BY ELECTRON-PROBE MICROANALYSIS

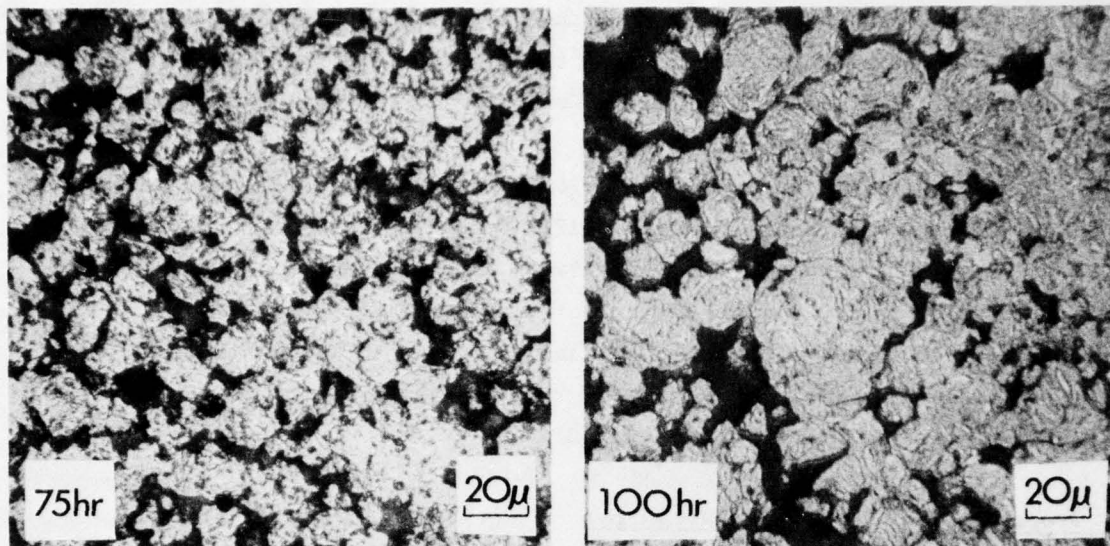


FIGURE 8. TiAl-3% Y<sub>2</sub>O<sub>3</sub> ALLOY POWDER STRUCTURES (BATCH T-17)

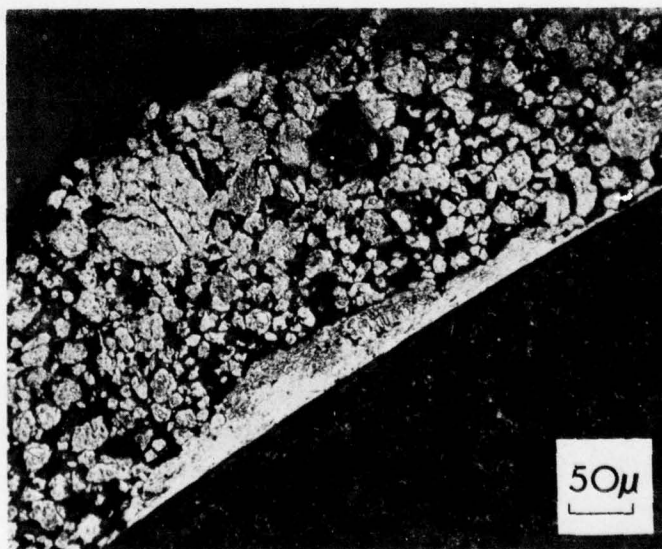


FIGURE 9. TiAl-3% Y<sub>2</sub>O<sub>3</sub> DEPOSIT ON CARBON STEEL BALL AFTER FAILURE OF ATTRITOR MILL IMPELLER SHAFT. Preceding failure the mill ran continuously for more than 10 hours.

maintained. Possibly as a result of this increased free play in the impeller motion, powder agglomerated on the walls of the attritor, and after 74 hrs of running the vibration of the mill was such that the run was terminated. The yield of powder was only 10.7%, the rest being solidly compacted on the attritor walls.

The three batches of TiAl-3%  $Y_2O_3$  powder were canned separately in Ti-6Al-4V extrusion cans of standard AFML design, outgassed and sealed by EB welding, and extruded.\* The conditions of extrusion were 2575°F at 15.3:1 reduction ratio, and the lengths of usable rod obtained were approximately 20 inches of Batches T-16 and T-17, and 10 inches of Batch T-18. A final batch (No. 21) of TiAl-3%  $Y_2O_3$  was prepared at the end of the runs for the TiAl-1.5%  $Y_2O_3$  discussed next.

#### c. Preparation of TiAl-1.5% $Y_2O_3$ Alloys

A major problem encountered in preparing the TiAl-3%  $Y_2O_3$  alloys by attritor milling was the agglomeration of large amounts of powder on the walls of the attritor mill, and the consequent mechanical failure of the equipment. It appeared that the extent of particle agglomeration increased with the length of continuous milling time: the population of the larger effective particle size fractions decreased with short milling times and increased at extended milling times, free powder yield decreased with increasing milling time, and powder build-up on the walls of the attritor occurred in the first two runs after 11 to 20 hrs of continuous running. The hard deposits on the attritor walls, impeller and ball charge which resulted in the jamming and breaking of the impeller shaft in the run to produce powder for metallographic analysis, all had the form of tightly-welded fine particles as typified by the deposit on a carbon steel ball from this run, shown in Figure 9. The particles contact each other at small points to form a rigid, open structure, and few massive, dense particles are evident.\*\* It appeared that the formation of such deposits had resulted from heating up of the milling charge during prolonged processing to a point where diffusion bonding of individual particles occurred

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\* Thanks are due to Mr. A. M. Adair of the AFML for arranging for the extrusions to be carried out.

\*\* The compact is very hard and grit blasting is required to remove the material from the attritor walls.

without their being smeared out and mechanically alloyed. Because of the poor heat transfer characteristics inherent in the design of the milling system, solutions to this problem are rather restrictive, the possibilities were: (a) operate intermittently for relatively short times, with sufficient time allowed for the charge to cool between periods of milling, (b) cool the working fluid in the mill by recycling through a refrigeration unit, or (c) resort to wet milling.

The alloying runs which were voluntarily terminated (T-16 and T-17) were carried out using a milling-cooling cycle having short milling periods (three to nine hrs), punctuated by cooling periods, a procedure which is, however, time consuming and labor intensive. The technique adopted for the attritor milling of the TiAl + 1.5 v/o  $Y_2O_3$  alloys was to run the mill for cycles of four hours on and two hours off to minimize accumulation of the charge on the internal surfaces of the mill.

Two batches of a TiAl + 1.5%  $Y_2O_3$  alloy (batch numbers T-19 and T-20) were prepared using this technique. No difficulties were experienced in attritor milling these two batches, and the amount of alloy powder remaining attached to the internal surfaces of the mill remained reasonably constant at 550-700 g. In addition, a further batch of a TiAl + 3%  $Y_2O_3$  alloy, Batch Number T-21, was run for 132 hrs. to provide additional material. Details of the yield from each run and of the size distribution of the final alloy powders are given in Table 5.

The alloy powders were canned in Ti-6Al-4V alloy cans of standard AFML design, and extruded as indicated in Table 6. Alloy T-21 was immersed in a tank filled with vermiculite to reduce the rate of cooling, in an attempt to decrease the level of internal stress noted in alloys extruded earlier, assuming the source of the stress was differential thermal expansion between the matrix and dispersoid. All the alloys proved difficult to extrude, and the section of the extruded rods containing the TiAl often contained transverse cracks.

## 2. METALLOGRAPHIC EVALUATION OF ALLOYS

Representative etched cross sections of the TiAl-3%  $Y_2O_3$  alloys (Batches T-16, T-17, T-21/7 and T-21/8) and of the TiAl-1.5%  $Y_2O_3$  alloys

TABLE 5. DETAILS OF ALLOY POWDERS

Size Fraction	% in Size Fraction	T-19	T-20	T-21
> 420 $\mu\text{m}$		4.4	4.5	8.4
> 149 < 420 $\mu\text{m}$		3.7	11.0	15.0
> 105 < 149 $\mu\text{m}$		3.7	6.6	7.6
< 37 < 105 $\mu\text{m}$		69.7	42.7	30.1
> 37 $\mu\text{m}$		18.6	35.2	39.0
YIELD %		52.5	58.8	69.1

TABLE 6. SUMMARY OF ALLOY PREPARATION DATA

Alloy	TiAl + 3 vol.% Y <sub>2</sub> O <sub>3</sub>			TiAl + 1.5 vol.% Y <sub>2</sub> O <sub>3</sub>		
	T-16	T-17	T-18	T-19	T-20	(T-19+T-20)
Milling Time, hr.	74.5	104	85.5	127	130	128.5
Extrusion T, °F	2575	2575	2575	2575	2575	2575
Reduction Ratio	15.2:1	15.4:1	15.2:1	20.42:1	21:1	20.5:1
Size of Starting Powder, μm	<105>37	<37	--	<105>37	<105>37	<37

(Batches T-19 and T-20) are shown in Figures 10 to 16. In general, the alloys appear reasonably homogeneous with little real difference resulting from the size of the alloy powder before extrusion. Voids observed in some areas of alloys (Figure 12) extruded at the lower nominal reduction ratio (16:1) attest to the nonuniform mode of extrusion often occurring in these materials. The main feature of the alloy microstructure is the occurrence of a precipitate, some 1 to 3  $\mu\text{m}$  in diameter and slightly elongated in the extrusion direction, which is probably  $\alpha_2$ . Positive identification has not been made, but the inference from electron-probe microanalysis is that the precipitate is not as rich in aluminum as the matrix.

A summary of data from ten-second counts made with the electron beam situated over typical precipitate particles and over the adjacent, apparently particle-free matrix, is given in Table 7. Considering that the X-ray source in the alloy is approximately 2  $\mu\text{m}$  in diameter at the beam acceleration voltage used, and that the particle size is of a similar order, then the difference in aluminum content between the particles and matrix is significant, but not quantitatively equal to that between  $\text{Ti}_3\text{Al}$  and  $\text{TiAl}$ . A further important observation is that the yttrium signal from the particles is much greater than from the matrix, suggesting that the  $\alpha_2$  precipitated on yttria particles. Many, but not all, of the precipitate particles investigated showed this trend in yttrium content.

TABLE 7. SUMMARY OF ELECTRON-PROBE MICROANALYSIS  
(ALLOY T-17)

AREA	AVERAGE 10 SEC. COUNTS			UNCORRECTED %		
	Ti	Al	Y	Ti	Al	Y
Particle No. 1	12764	23412	2070	46.7	21.1	21.7
Adjacent Matrix	15761	26690	95	57.6	25.0	1.0
Particle No. 2	12635	20804	1393	46.2	18.7	14.6
Adjacent Matrix	15162	23853	123	55.4	21.5	1.3

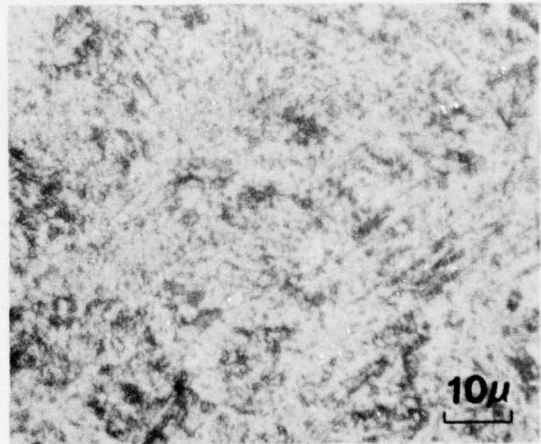
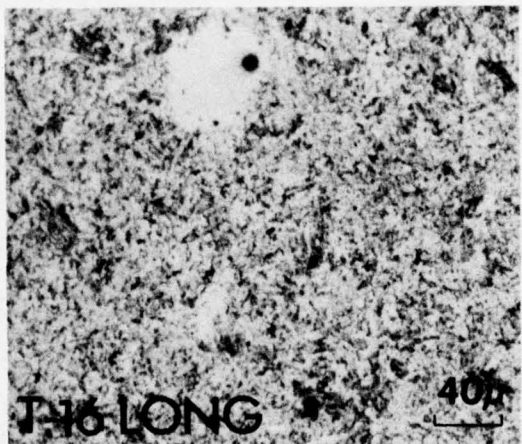
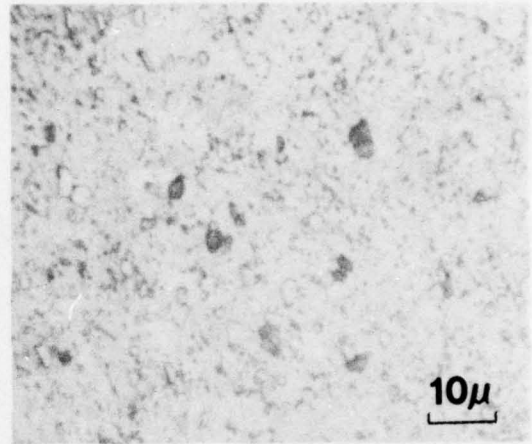
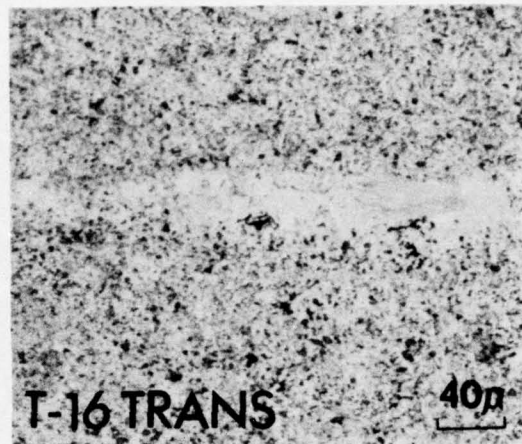


FIGURE 10. ETCHED MICROSTRUCTURES OF  $TiAl+3\% Y_2O_3$  ALLOY (BATCH T-16)

As extruded: 2575°F, 15.2:1, Air cooled, initial powder size  $< 105\mu m$ .

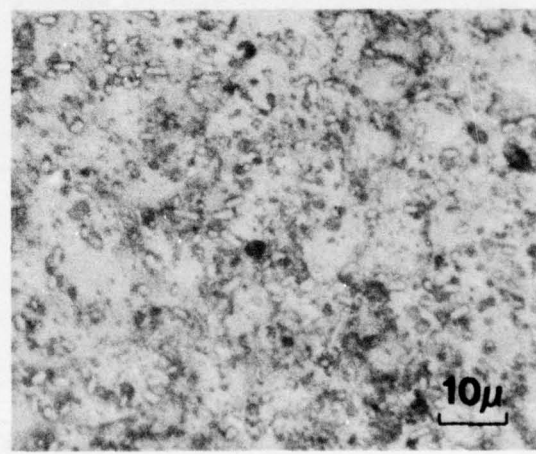
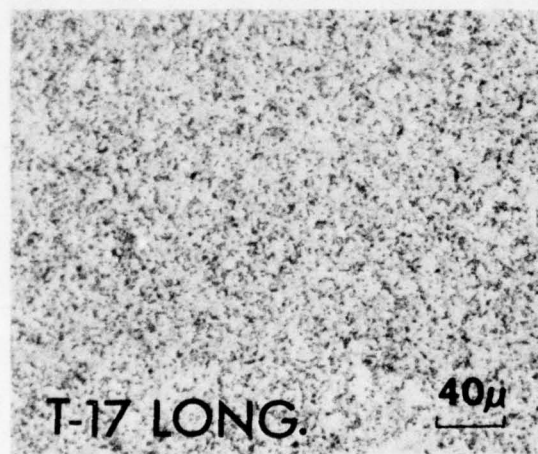
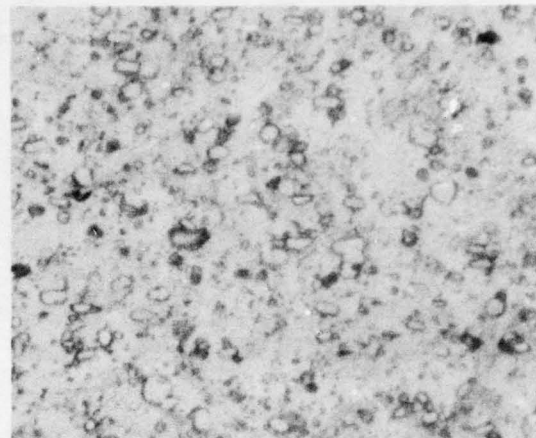
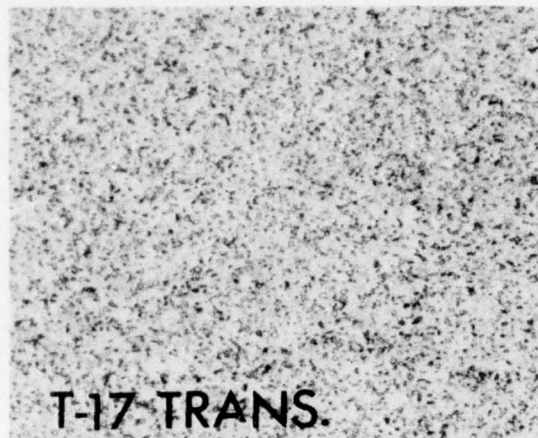


FIGURE 11. ETCHED MICROSTRUCTURES OF  $\text{TiAl}+3\% \text{Y}_2\text{O}_3$  ALLOY (BATCH T-17)

As extruded:  $2575^\circ\text{F}$ , 15.4:1, Air cooled; initial powder size  $< 37\mu\text{m}$ .

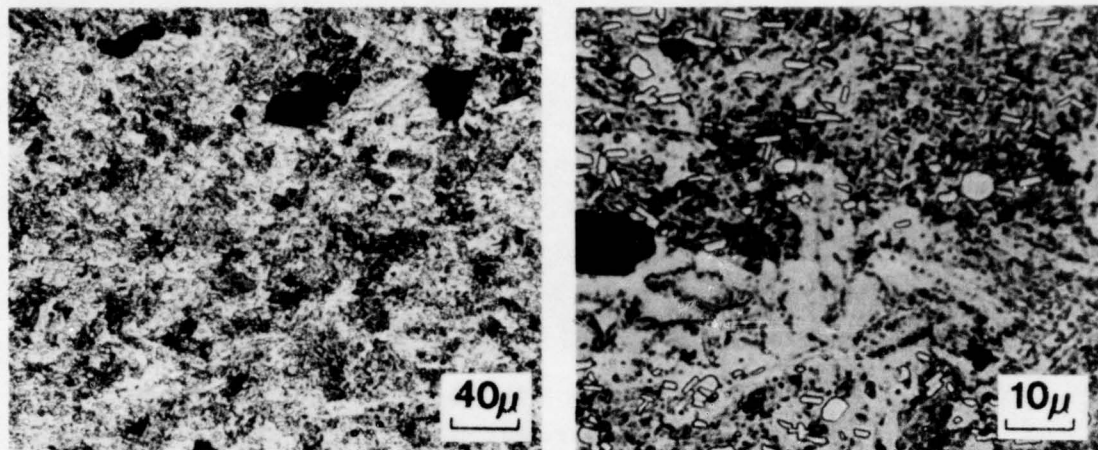


FIGURE 12. TiAl + 3% Y<sub>2</sub>O<sub>3</sub> (T-21/7). X250 AND X1000

As extruded: 2575°F, 16:1, slowly cooled,  
initial powder size < 37 μm.

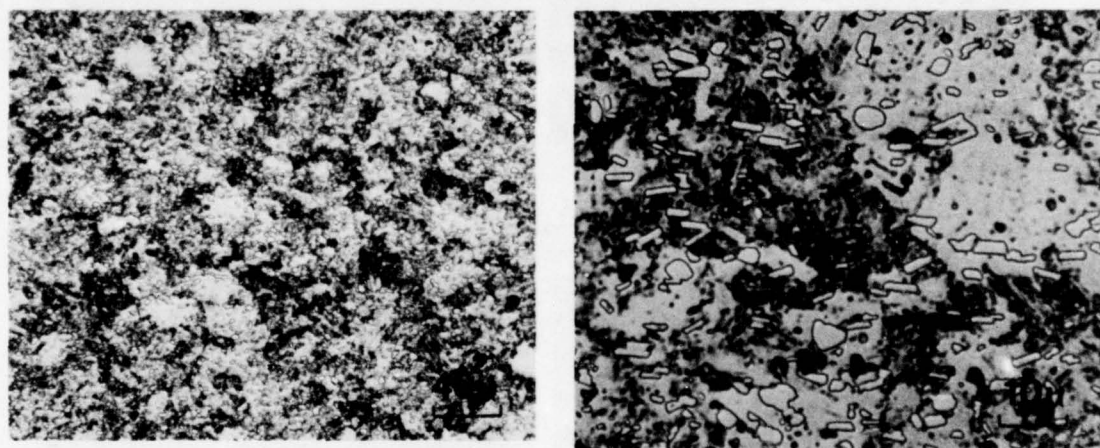


FIGURE 13. TiAl + 3% Y<sub>2</sub>O<sub>3</sub> (T-21/8). X250 AND X1000

As extruded: 2575°F, 16:1, slowly cooled,  
initial powder size < 105 μm > 37 μm.

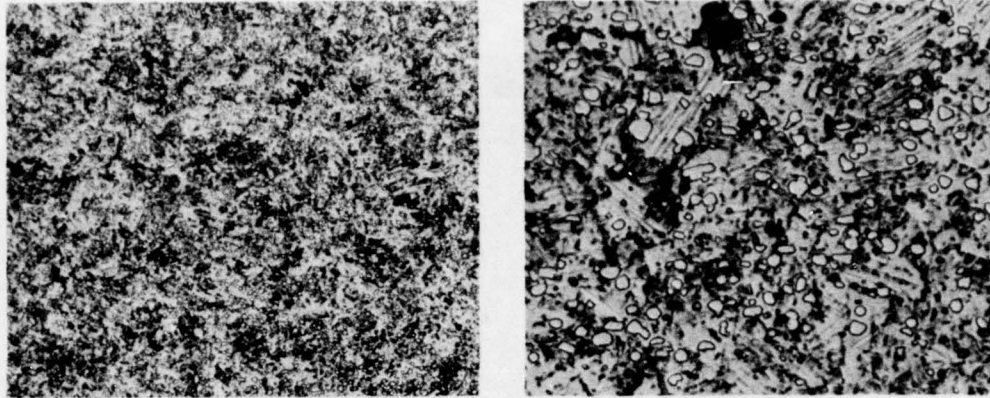


FIGURE 14. TiAl + 1.5% Y<sub>2</sub>O<sub>3</sub> (T-19). X250 AND X1000  
 As extruded: 2575°F, 20:1, air cooled, initial  
 powder size < 105 > 37μm.

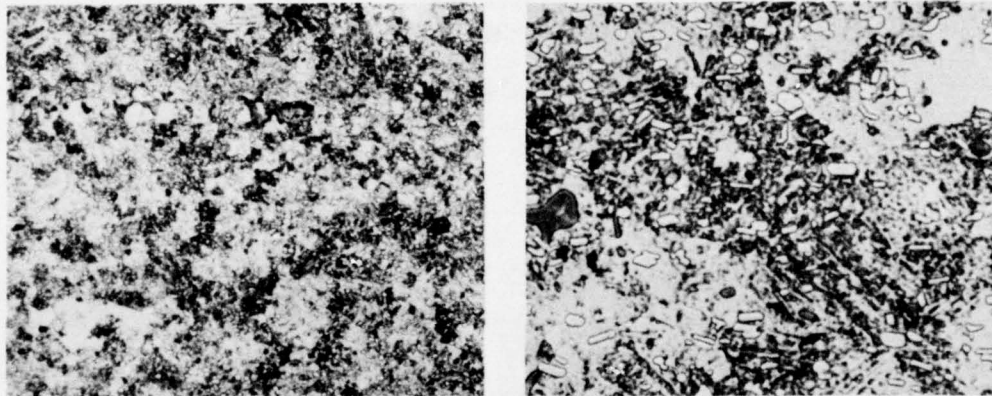


FIGURE 15. TiAl + 1.5% Y<sub>2</sub>O<sub>3</sub> (T-20). X250 AND X1000  
 As extruded: 2575°F, 20:1, air cooled, initial  
 powder size < 105 > 37μm.

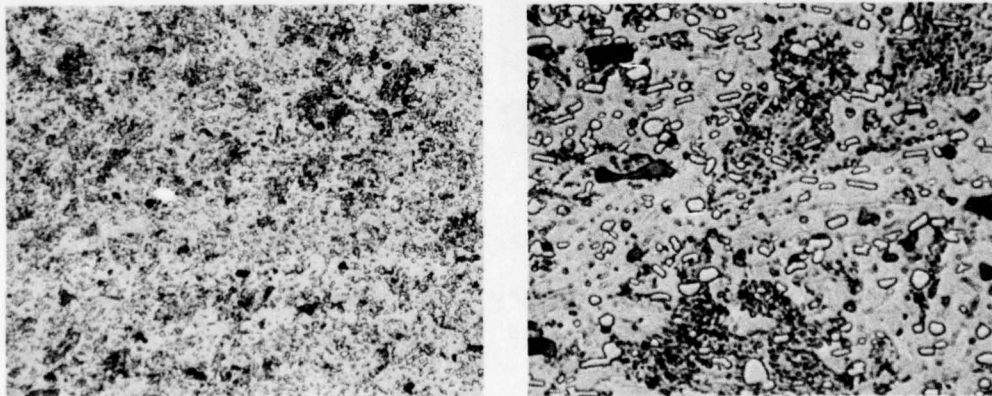


FIGURE 16. TiAl + 1.5% Y<sub>2</sub>O<sub>3</sub> (POWDERS T-19 + T-20). X250 AND X1000  
 Mixed and extruded: 2575°F, 20:1, air cooled  
 initial powder size < 37μm.

The alloy grain size is not evident after chemical etching, but preliminary thermal etching experiments indicate small, equiaxed grains. In some areas of most alloys, but especially in alloys T-16 and T-19, areas showing a basketweave structure, probably lamellar  $\alpha_2$ , were observed. Small regions of unalloyed TiAl, together with translucent oxide particles ( $\text{Al}_2\text{O}_3$ ?) were also apparent in most alloys.

Attempts were made to change the form of the  $\alpha_2$  precipitate, or at least to free the yttria particles from association with  $\alpha_2$ , by heat treatment. Specimens of alloy T-17 were held at 1200°C for two hours (in the  $\gamma$  "nose") and then ice-brine quenched or furnace cooled. The microstructures were little affected, however, as shown in Figure 17 and 18, which suggests that the kinetics of  $\alpha_2$  precipitation are very fast, or that the kinetics of  $\alpha_2$  dissolution are very slow.

Transmission microscopy of the TiAl-3%  $\text{Y}_2\text{O}_3$ , Batch T-17, showed none of the small  $\text{Y}_2\text{O}_3$  dispersoid (200-400 Å) expected. Instead there were only large, presumably  $\alpha_2$ , particles (Figure 19). There was little evidence of the lamellar  $\alpha_2$  observed in TiAl without  $\text{Y}_2\text{O}_3$  particles.<sup>(9)</sup> The absence of a fine, uniform dispersion of  $\text{Y}_2\text{O}_3$  undoubtedly inhibited the development of improved creep strength over that of TiAl as discussed in the following section.

### 3. MECHANICAL PROPERTY STUDIES

Button head tensile specimens having a gaged length of 0.4 inch were machined from extruded rods T-16, T-17 and T-21 containing 3%  $\text{Y}_2\text{O}_3$  and from rods T-19 and T-20 containing 1.5%  $\text{Y}_2\text{O}_3$ . The tensile strength was determined at room temperature and 1800F for rods T-16 and T-17 and at room temperature and 1652F for rods T-19, T-20, and T-21, at a strain rate of  $0.008 \text{ min}^{-1}$ . Tensile creep tests were performed at 1800F on rods T-16 and T-17 and at 1652F on rods T-19, T-20, and T-21.

The tensile results are shown in Table 8 along with results of Lipsitt, et al.<sup>(10)</sup> on TiAl. The room temperature strength of the dispersion-containing alloys is higher than for TiAl.<sup>(10)</sup> However, there is no ductilizing effect from the dispersion as shown previously to exist in Cr-ThO<sub>2</sub><sup>(4)</sup>. Part of the reason for this is that a fine uniform

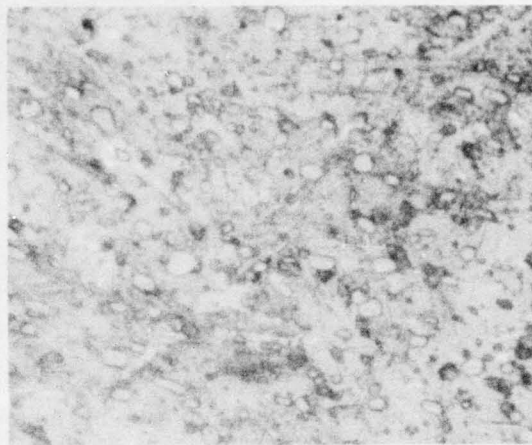
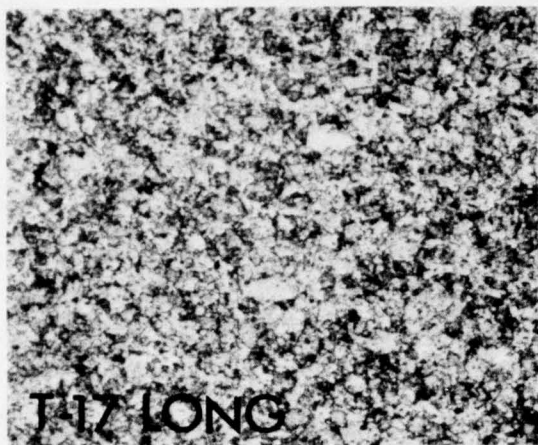


FIGURE 17. TiAl + 3% Y<sub>2</sub>O<sub>3</sub> (T-17). X250 AND X1000, AFTER 2 HRS AT 1250°C FOLLOWED BY AN ICED BRINE QUENCH.

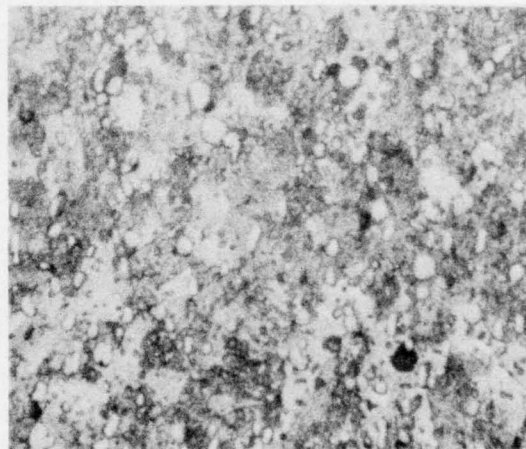
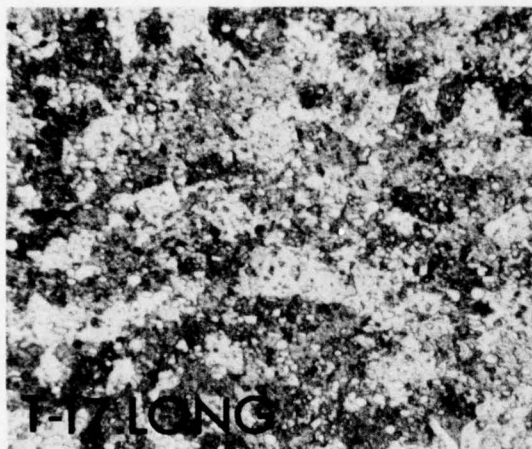


FIGURE 18. TiAl + 3% Y<sub>2</sub>O<sub>3</sub> (T-17). X250 AND X1000, AFTER 2 HRS AT 1200°C FOLLOWED BY FURNACE COOLING.

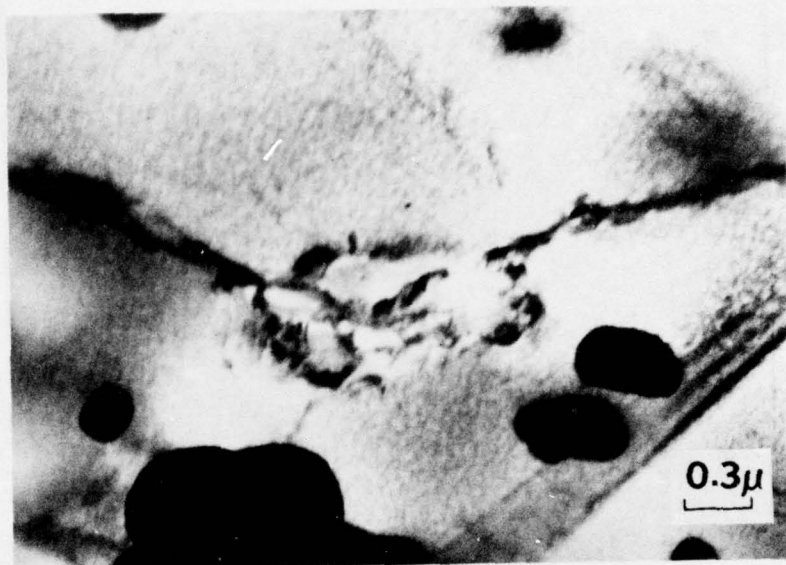


FIGURE 19. TEM OF TiAl + 3% Y<sub>2</sub>O<sub>3</sub> ALLOY T-17.

TABLE 8. TENSILE RESULTS FOR TIAL+Y<sub>2</sub>O<sub>3</sub> ALLOYS WITH COMPARISON TO TIAL

Alloy (a)	Material Rod	Test Temperature °F	0.2% Yield Strength ksi	Ultimate Tensile Strength ksi	Elongation %	Reduction in Area %	Reference
TIAL (b)		75	--	69	0	0	10
TIAL+1.5% Y <sub>2</sub> O <sub>3</sub>	T-19	75	--	89	0	0	This Study
TIAL+1.5% Y <sub>2</sub> O <sub>3</sub>	T-20	75	--	87	0	0	"
TIAL+3% Y <sub>2</sub> O <sub>3</sub>	T-21	75	--	91	0	0	"
TIAL+3% Y <sub>2</sub> O <sub>3</sub>	T-17	75	--	98	0	0	"
30 TIAL (b)		1652	21.5	32	32	39	10
TIAL+1.5% Y <sub>2</sub> O <sub>3</sub>	T-19	1652	44	51	25	24	This Study
TIAL+1.5% Y <sub>2</sub> O <sub>3</sub>	T-20	1652	50	52	17	18	"
TIAL+3% Y <sub>2</sub> O <sub>3</sub>	T-21	1652	49	53	13	13	"
TIAL+3% Y <sub>2</sub> O <sub>3</sub>	T-17	1800	23	28	2.8	--	"
TIAL (c)		1800	23	39	17	25	11
TIAL (b)		1800	17.6	22	--	37	10

(a) All the alloys containing Y<sub>2</sub>O<sub>3</sub> are Ti-36 wt. % Al.

(b) Ti-40 wt. % Al.

(c) Ti-36 wt. % Al.

dispersion of  $Y_2O_3$  was not obtained, and for the operation of possible ductilizing mechanisms such as provision of numerous dislocation sources and dispersing of slip, such a distribution is necessary.

The creep behavior of the TiAl +  $Y_2O_3$  alloys shows the usual 3-stage creep curve, but with a low initial plastic and primary creep strain. The creep results are shown in Table 9. Comparison with the TiAl results of McAndrew and Kessler<sup>(11)</sup> can be made on the basis of the 100-hour rupture stress. From the limited data in Table 9, the 100-hour rupture stresses are estimated to be 8 ksi at 1800F for the TiAl-1.5%  $Y_2O_3$  and about 10 ksi at 1652F for TiAl-3%  $Y_2O_3$ . For cast Ti-36 wt % Al, the 100-hour rupture stresses are 7.5 ksi at 1800F and 14.5 ksi at 1652.<sup>(11)</sup>

The comparison between these strengths cannot be made directly since the grain size of the extruded rod is small, on the order of tens of micrometers, and the grain size of cast TiAl would almost certainly be larger. The high temperature creep strength of the finer-grain size material will always be lower due to grain boundary sliding in the absence of strong grain boundary pinning by precipitates. This aspect of the creep strength cannot be measured from these data, but it would be expected that a coarser grain size in the TiAl +  $Y_2O_3$  alloys would increase the creep strength. However, in view of the poor quality of the dispersion, the increase in strength in these alloys would probably be slight.

The relatively large total elongations of the dispersion strengthened material at 1652 and 1800F show the creep strength is not being limited by a low creep ductility.

#### 4. OXIDATION STUDIES

Specimens for oxidation studies, measuring approximately 1 x 1 x 0.15 cm, were sliced from the as-extruded rods in the longitudinal direction. Specimens were oxidized with either a "deformation-free" surface finish (polished through 0.03  $\mu$ m alumina) or an "abraded" surface finish (240 SiC grit). Coupons of yttria-free TiAl, 0.9 cm diam. x 0.12 cm thick, were supplied by AFML and oxidized to provide comparative data.

Typical oxidation kinetics obtained for the TiAl + 1.5%  $Y_2O_3$  and TiAl + 3%  $Y_2O_3$  alloys are shown in Figures 20 and 21, and individual

TABLE 9. CREEP RESULTS FOR TIAL + Y<sub>2</sub>O<sub>3</sub> ALLOYS

Alloy	Material Rod	Test Temperature °F	Stress ksi	Minimum Creep Rate h <sup>-1</sup>	Rupture Life h	Total Elongation %	Reference
TIAL-1.5% Y <sub>2</sub> O <sub>3</sub>	T-16	1800	10	2.46 x 10 <sup>-3</sup>	35.6	12	This Study
TIAL-1.5% Y <sub>2</sub> O <sub>3</sub>	T-16	1800	20	6.91 x 10 <sup>-2</sup>	0.63	9	"
TIAL-1.5% Y <sub>2</sub> O <sub>3</sub>	T-21	1652	15	1.90 x 10 <sup>-3</sup>	19.0	13	"
TIAL-3% Y <sub>2</sub> O <sub>3</sub>	T-19	1652	15	2.0 x 10 <sup>-3</sup>	32.0	20	"
TIAL-3% Y <sub>2</sub> O <sub>3</sub>	T-20	1652	25	1.08 x 10 <sup>-2</sup>	4.0	16	"
TIAL-3% Y <sub>2</sub> O <sub>3</sub>	T-20	1652	11	1.26 x 10 <sup>-4</sup>	61.0	18	"

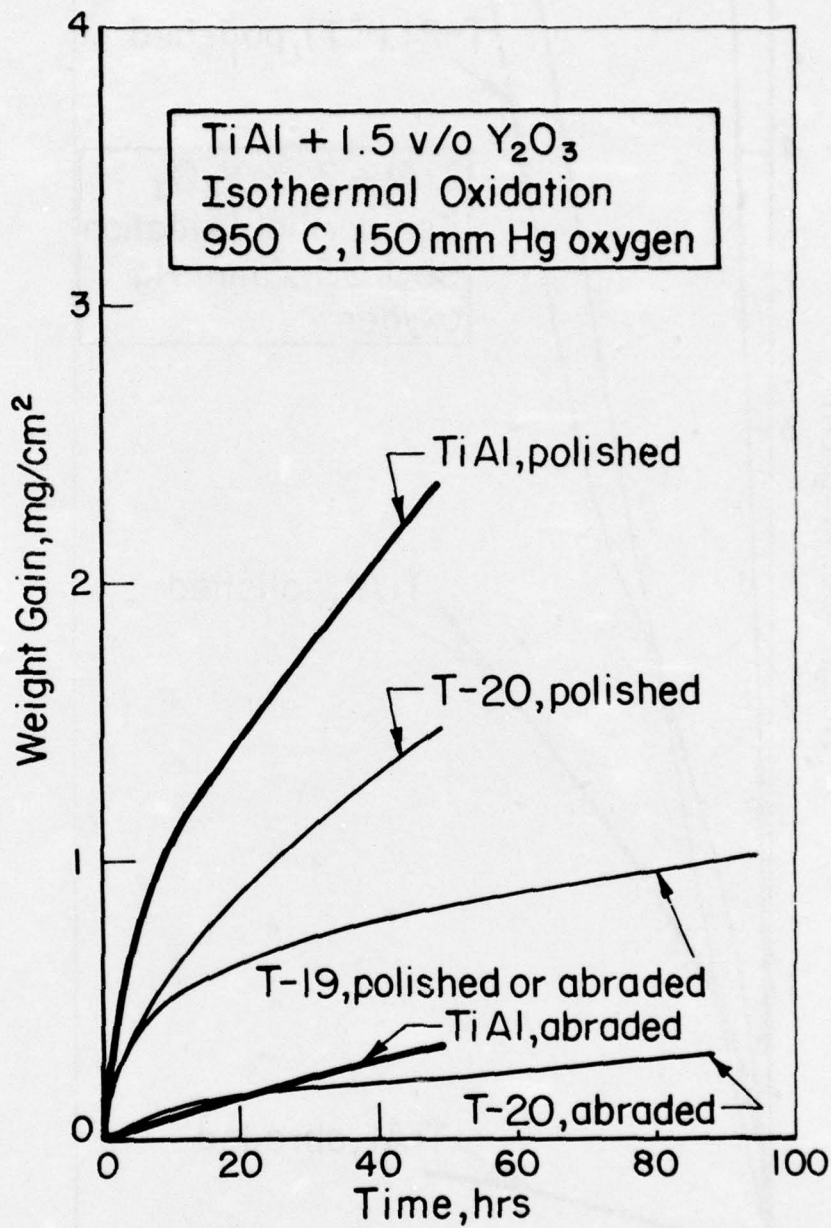


FIGURE 20. OXIDATION KINETICS OF TiAl + 1.5 v/o Y<sub>2</sub>O<sub>3</sub> ALLOYS

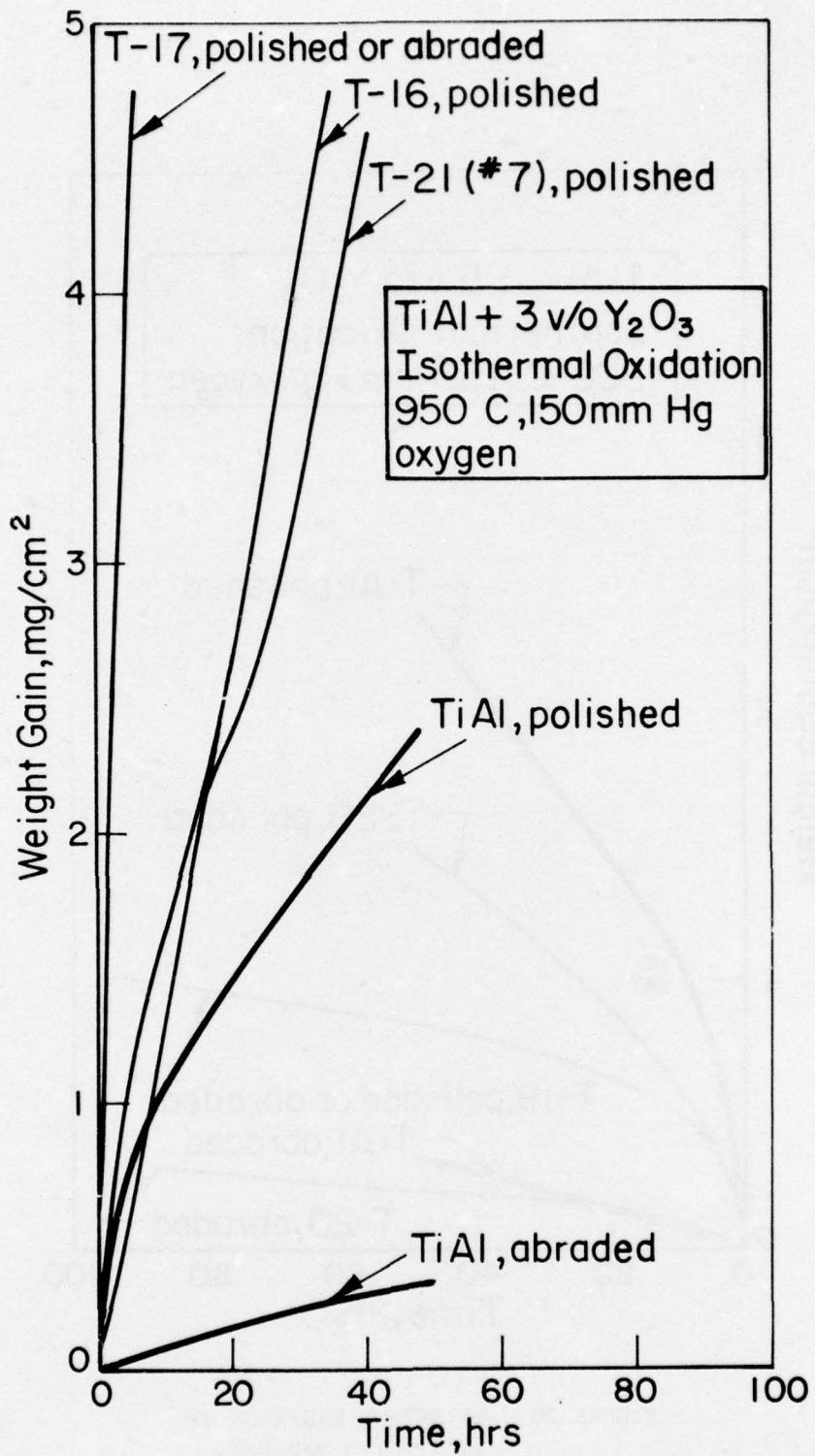


FIGURE 21. OXIDATION KINETICS OF TiAl + 3v/o Y<sub>2</sub>O<sub>3</sub> ALLOYS

specimen details are given in Table 10. For yttria-free TiAl, the effect of introducing cold work into the surface was to reduce the oxidation rate to an extent that suggested a fundamental change in scale type, probably from TiO<sub>2</sub>-rich to Al<sub>2</sub>O<sub>3</sub>-rich. The scales on all specimens of TiAl were of similar outward appearance (red-brown), and spalled extensively on cooling. Data obtained for TiAl by J. W. Hinze at AFML using the same nominal conditions were very similar<sup>(12)</sup>, with a parabolic rate constant of 10<sup>-5</sup> to 10<sup>-6</sup> g<sup>2</sup> cm<sup>-4</sup> h<sup>-1</sup> for deformation-free TiAl, and of  $\sim 10^{-9}$  g<sup>2</sup> cm<sup>-4</sup> h<sup>-1</sup> (weight gain of 0.21 mg/cm<sup>2</sup> after 45 hrs) for cold-worked TiAl. The cold-worked specimens were determined at AFML to form larger amounts of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> in the scale than deformation-free specimens.

The oxidation behavior of the yttria-containing alloys was very variable. The TiAl + 3% Y<sub>2</sub>O<sub>3</sub> alloys oxidized faster than yttria-free TiAl in all surface conditions, and formed thick but generally adherent scales, typified in Figure 22a. Specimens of these alloys oxidized with a cold-worked surface did not necessarily exhibit slower oxidation rates than specimens with deformation-free surfaces.

Most specimens of the TiAl + 1.5% Y<sub>2</sub>O<sub>3</sub> alloys oxidized predictably with respect of surface finish, and at slower rates than the equivalent dispersion-free TiAl specimens. The scales on these TiAl + 1.5% Y<sub>2</sub>O<sub>3</sub> alloys were all adherent, generally grey/white in color, and single-layered, as shown in Figure 22b.

There appeared to be no obvious major difference in the detailed microstructure of the alloys, as observed metallographically, which would explain the differences in oxidation behavior of the TiAl + 3% Y<sub>2</sub>O<sub>3</sub> alloys and the TiAl + 1.5% Y<sub>2</sub>O<sub>3</sub> alloys. The distribution of what appears to be particles of  $\alpha_2$  throughout the alloy microstructure could provide a reason for faster oxidation kinetics than for a more homogeneous TiAl structure. Initial oxidation of the 2-3  $\mu$ m diameter  $\alpha_2$  particles present in these alloys would probably produce islands of TiO<sub>2</sub> scale distributed throughout the alloy surface while the oxide covering the rest of the surface would probably comprise an initiate mixture of nuclei of TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. Where conditions are favorable for the development of an Al<sub>2</sub>O<sub>3</sub>-rich scale (high surface aluminium level, high aluminium diffusion rate at the alloy surface), the initial TiO<sub>2</sub> nuclei may be undercut and isolated from the alloy by a

TABLE 10. DETAILS OF SPECIMENS OXIDISED AT 950°C IN 150 mm Hg OXYGEN

Alloy	Spec. No.	Surface Finish*	Exposure Duration (h)	Weight Change (mg/cm <sup>2</sup> )		Rate Constant
				Thermobalance	Check Weighing	
TiAl	T5	P	48	2.3830	Scale Spalled	$k_p = 1.11 \times 10^{-7} \text{ g cm}^{-4} \text{ h}^{-1}$
	T4	A	63	18.8170	--	
	T6	A	45	0.2754	Scale Spalled	$k_p = 6.08 \times 10^{-6} \text{ g cm}^{-2} \text{ h}^{-1}$
TiAl + 1.5% Y <sub>2</sub> O <sub>3</sub> (T-19)	T9	A	24	0.6540	0.5443	∞ cubic
	T15	A	48	13.0271	12.8899	$k_p = 4.70 \times 10^{-6} \text{ g cm}^{-4} \text{ h}^{-1}$ (>20h)
	T10	P	95	1.0168	0.8179	∞ cubic
TiAl + 1.5% Y <sub>2</sub> O <sub>3</sub> (T-20)	T11	P	49	1.4878	1.6091	$k_p = 4.59 \times 10^{-8} \text{ g cm}^{-4} \text{ h}^{-1}$
	T14	A	88	0.3151	0.4042	∞ cubic
TiAl + 3% Y <sub>2</sub> O <sub>3</sub> (T-16)	T7	P	7	0.2435	0.2095	$k_p = 1.01 \times 10^{-8} \text{ g cm}^{-2} \text{ h}^{-1}$
	T8	P	64	8.2065	8.1006	$k_L = 1.36 \times 10^{-4} \text{ g cm}^{-2} \text{ h}^{-1}$
TiAl + 3% Y <sub>2</sub> O <sub>3</sub> (T-17)	T1	P	28.5	23.4138	Pt support wire embedded in scale	$k_L = 6.96 \times 10^{-4} \text{ g cm}^{-2} \text{ h}^{-1}$
	T2	A	43	21.8842	--	$k_L = 5.80 \times 10^{-4} \text{ g cm}^{-2} \text{ h}^{-1}$
	T3	P	10 min	0.0693	--	--
TiAl + 3% Y <sub>2</sub> O <sub>3</sub> (T-21)	T12	P	46.5	5.0741	4.9617	$k_p = 2.93 \times 10^{-7} \text{ g cm}^{-2} \text{ h}^{-1}$
	T13	A	63.5	21.1213	Scale spalled	$k_L = 6.07 \times 10^{-4} \text{ g cm}^{-2} \text{ h}^{-1}$
Ti-40Al**	--	A	46.5	18.0647	--	$k_L = 3.5 \times 10^{-4} \text{ g cm}^{-2} \text{ h}^{-1}$
Ti-40Al-3% Y <sub>2</sub> O <sub>3</sub>	--	A	48.5	11.1580	--	$k_p = 1.25 \times 10^{-6} \text{ g cm}^{-4} \text{ h}^{-1}$ (<15h) $k_L = 1.96 \times 10^{-4} \text{ g cm}^{-2} \text{ h}^{-1}$ (>15h)

\* A = cold worked surface finish, P = deformation-free surface finish.

\*\* At 1000°C in 100 mm Hg oxygen (Reference 13).

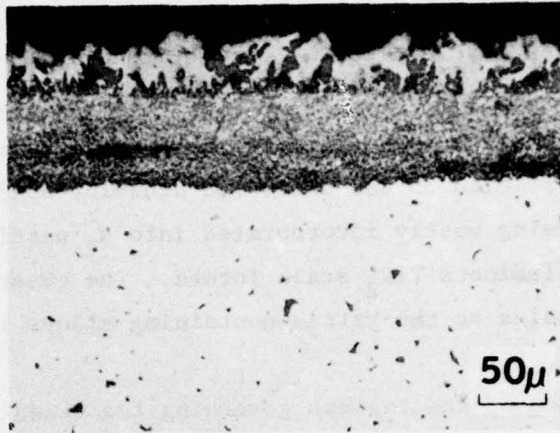


FIGURE 22a. TYPICAL NON-PROTECTIVE  
OXIDE SCALE (TiAl+1.5%  
Y<sub>2</sub>O<sub>3</sub>, SPECIMEN T-15)

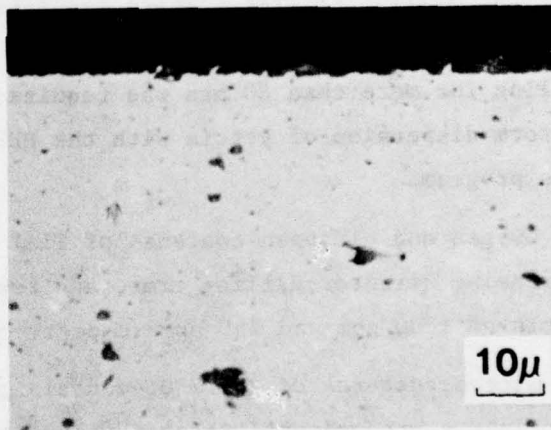


FIGURE 22b. TYPICAL PROTECTIVE OXIDE  
SCALE (TiAl+1.5%Y<sub>2</sub>O<sub>3</sub>,  
SPECIMEN T-14)

spreading alumina film. However, the larger islands of  $TiO_2$  formed over surface  $\alpha_2$  particles may lead to the production of  $TiO_2$ -rich nodules in the scale, and possibly to disruption of the areas of protective  $Al_2O_3$ -rich scale. It might also be anticipated that the effectiveness of the dispersed yttria particles on the oxidation behavior would be reduced as a result of their being mostly incorporated into  $\alpha_2$  particles, and so being carried into the voluminous  $TiO_2$  scale formed. The observed good macro adherence of the scales on the yttria-containing alloys is therefore somewhat surprising.

Elucidation of the factors governing the oxidation behavior of these alloys requires a more detailed examination of the nature of the alloy surface before and during the early stages of oxidation than has been possible during the program described.

### III. CONCLUSIONS

The results of this study of the feasibility of dispersion-strengthening of TiAl indicate that:

- (1) Attritor milling of TiAl powders for total milling times in excess of 100 hrs can be accomplished without problems. Milling for more than 80 hrs was required to achieve a uniform dispersion of yttria with the REP powder used in this program.
- (2) The oxygen and nitrogen contents of TiAl increased with increasing attritor milling time, and reached steady levels of 1300 ppm and 250 ppm respectively after 20-30 hrs.
- (3) The microstructures of the  $Y_2O_3$ -containing TiAl alloys comprised a predominantly basket-weave structure of  $\gamma$  and lamellar  $\alpha_2$ , with uniformly dispersed particles, apparently of  $\alpha_2$ , which were largely associated with the yttria dispersion. Few free yttria particles were observed in the alloy.

- (4) The yttria-containing alloys exhibited considerably increased tensile strength at room temperature and at 1652°F compared to yttria-free TiAl.
- (5) The tensile creep strengths of  $Y_2O_3$ -containing TiAl and  $Y_2O_3$ -free TiAl at 900°C were similar.
- (6) No effect was observed of the dispersoid on the room-temperature ductility of TiAl.
- (7) The oxidation behavior of the yttria-containing alloys was variable, some specimens oxidizing more slowly than the equivalent dispersion-free TiAl specimens, and most specimens exhibiting adherent scale.

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