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A STUDY OF THE DEPENDENCE OF MICROSEGREGATION ON CRITICAL SOLIDIFICATION PARAMETERS IN RAPIDLY- QUENCHED STRUCTURES

Prepared by

D.B. Snow

Final Report

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Mechanically-blended powder alloys of Ti-6Al-4V, Ti-6Al-4V blended with $\leq$ 3Er, Y or Nd, and Ti-6Al-2Sn-4Zr-6Mo-1Er (wt%) were laser-consolidated and rapidly solidified ($\sim 5 \times 10^4$ °C/s) by the LAYERGLAZE^{SM} process. This procedure was used to form the fusion zones of butt welds in Ti-6Al-4V plate, premachined with straight-sided 3 mm gaps, which were filled with sequential layers of laser-melted powder feedstock. After melting, the rare-earth elements reacted with dissolved oxygen in the titanium to form a dispersion of equilibrium-structure oxides. The

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Rare-Earth Oxides
Oxide Dispersion Strengthening
Erbium Oxide

alpha

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as-solidified microstructure of each alloy consisted of large, columnar grains of acicular α' HCP martensite. The largest rare earth oxide size and spatial dispersions were observed in alloys LAYERGLAZED from Ti-6Al-4V-3Nd powder; the finest, in those made from Ti-6Al-4V-1.5Er. The largest oxide particles were located at interdendritic and, in the case of Ti-6Al-4V-1.5Er, at intergranular sites. The erbium oxide size distribution was bimodal, with mean sizes of 280 and 40 nm. The oxide structures were identified by analysis of selected area and convergent beam diffraction patterns, obtained from particles determined to contain the appropriate rare earth element by EDS. Neither the ambient temperature tensile strength nor the microhardness of either aged (800°C, 75 min) or as-LAYERGLAZED alloys were significantly improved by the oxide dispersions. Presumably, this occurred because the anticipated dispersion strengthening was offset by the removal of oxygen from solid solution. However, the tensile ductility of Ti-6Al-4V was increased by the addition of yttrium. The 40 nm Er_2O_3 particles observed in the alloy LAYERGLAZED from Ti-6Al-4V-1.5Er might be reasonably expected to improve creep strength.

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East Hartford, Connecticut 06108

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A Study of the Dependence of Microsegregation
on Critical Solidification Parameters in
Rapidly-Quenched Structures

FINAL REPORT

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A Study of the Dependence of Microsegregation of Critical
Solidification Parameters in Rapidly Quenched Structures

INTRODUCTION

During the four-year period of this contract, several different aspects of the microsegregation and microstructures produced by laser materials processing were investigated. In compliance with the instructions of the controlling office, the material contained in the three annual technical reports previously issued under this contract (Refs. 1-3) are not presented again here. In brief, the first of these modeled and experimentally determined the cooling rates associated with continuous, CO_2 laser welding, and examined the dependence of cracking on composition in laser-welded Ni-Al-Mo alloys (Ref. 1). The second annual technical report described the variation in continuous, CO_2 laser welding efficiency and fusion zone segregation on beam power and cladding thickness in stainless-clad AH32 carbon steel plate (Ref. 2). The third examined the structure and properties of narrow-gap butt welds formed by the powder-feed LAYERGLAZE [SM] process in Ti-6Al-4V plate (Ref. 3). The present report describes the results of the final year of research under this contract. In this study, the procedures described in Ref. 3 were utilized to alloy Ti-6-4 and Ti-6-2-4-6 powder feedstock with rare earth elements to create oxide dispersions in the LAYERGLAZE - processed fusion zones.

Titanium and titanium base alloys have been considered for several decades as potential candidates for dispersion strengthening by stable oxides, particularly rare earth oxides (Refs. 4-6). The relative insolubility of most rare earth elements in titanium (Ref. 7), together with their strong affinity for oxygen, have caused embrittling segregation (Ref. 8) and/or relatively coarse (> 260 nm) dispersions (Refs. 5,9) in conventionally-solidified Ti-rare earth alloys. However, rapidly solidified Ti-Er alloys have been observed to contain more refined, homogeneous second phase dispersions (Refs. 10-12), which have a significant strengthening effect (Ref. 9). The compounds which form the second phase dispersions in rapidly solidified Ti-Al-Y and Ti-Er alloys have been positively identified as Y_2O_3 and Er_2O_3, respectively (Refs. 13-15). The Er_2O_3 dispersion was reported to have good thermal stability at 700°C (Ref. 15). The present investigation was intended to demonstrate that similar rare earth oxide dispersions can be achieved in titanium base alloys by using mechanically - blended powder as feedstock for narrow-gap laser butt welds (LAYERGLAZE/narrow - gap welding) (Ref. 16).

EXPERIMENTAL PROCEDURE

A.   Laser Processing

The specific laser operating characteristics and apparatus used in this investigation were similar to those utilized in a previous study of the LAYERGLAZE/narrow-gap welding of Ti-6Al-4V (Ref. 16). The narrow-gap butt welds were made in 1.27 or 5.08 cm, mill-annealed, ELI Ti-6Al-4V plate. Specimen edges were premachined so that straight sided, 3 mm gaps were formed when two pieces were fitted together (Fig. 1). The sides of the weld gap were chemically cleaned just before welding in a solution of 30% nitric and 3% hydrofluoric acid, then rinsed with water and dried with forced air. Specimen warpage during welding was minimized by clamping each in a retention fixture (Fig. 2).

All 1.27 cm thick specimens welded with Ti-6Al-4V feedstock were processed with a continuous, cross-flow carbon dioxide laser, operated in the unstable resonator mode (annular beam energy cross-section). A 47 cm focal length copper mirror directed the laser beam downward toward the bottom of the weld gap, which was positioned 2.86 cm below its focal plane (Fig. 3). These conditions gave a 3.2 mm (defocused) beam spot diameter at the fusion zone surface. The laser beam power at the work station was 3.9 kW, so that the power per unit area at the fusion zone surface was 48.5 kW/cm^2, maintained by lowering the specimen height after every four deposited layers. The powder feedstock was transported from its reservoir to a point just ahead of the beam impingement point by vibration-assisted gravity flow (Ref. 16) while the specimen was traversed at 2.12 cm/s. Specimens were enclosed during welding in a chamber filled with helium flowing at 5.7 m^3/h, which had a narrow opening at its top to allow the beam to enter (Ref. 16).

Two groups of specimens were processed with a UTRC-developed, modular CO_2 laser (Ref. 18): 5.08 cm plate (center gap closure, Fig. 1), welded with prealloyed Ti-6Al-4V powder feedstock; and 1.27 cm plate (bottom gap closure, Fig. 1), welded with Ti-6Al-2Sn-4Zr-6Mo-1Er. These specimens were LAYERGLAZED with the work-station shown in Fig. 4, using a focusing mirror with a 61 cm focal length and a (defocused) beam spot diameter of 3 mm. A modular, cross-beam, continuous CO_2 laser with stable oscillator optics (Gaussian mode) (Ref. 18) was operated so as to achieve a beam power of 2.25 kW (32 kW/cm^2) at the specimen surface. The specimen traverse and powder feed procedures were those described previously.

B.   Powder Feedstock

Prealloyed Ti-6Al-4V powder without additional alloying elements was used for welds in both the 1.27 and 5.08 cm thick specimens to establish a basis of comparison with the results of previous research (Refs. 3,16). However, all of the alloys which contained rare earth elements were prepared as mechanically blended powder, and had the following nominal compositions: Ti-6Al-4V; Ti-6Al-4V + 1.5Er, or 3Nd; and Ti-6Al-2Sn-4Zr-6Mo-1Er (wt%). The characteristics of the component powders used to blend these feedstock compositions are described in Table I.

Table I

Powder Feedstock Specifications

Powder	Purity (wt%) (metallics)	Mesh Size	Source
Ti-6Al-4V(prealloyed)	99.98	-35, +325	Nuclear Metals
Ti-6Al-4V(mech. blend)	99.95	-170, +325	Amerimet Corp.
Er, Y, Nd	99.9	-40	Research Chemicals
Ti	99.8	-170, +325	Alloy Metals
Al	99.5	-100, +325	CERAC
Sn	99.9	-170	Fisher Scientific
Zr	99.9	-80	Alfa Products
Mo	99.8	-100	A. D. MacKay

C.   Structure and Mechanical Properties

The as-LAYERGLAZED microstructure of each alloy was examined by light microscopy, TEM/STEM (Philips EM400T), electron microprobe analysis (Cameca - "CAMEBAX"), and scanning Auger electron spectroscopy (Perkin-Elmer PHI 600). As-processed fusion zone compositions were obtained by wet chemical analysis, inert gas fusion (Leco), and wavelength-dispersive X-ray spectroscopy (electron microprobe).

Tensile specimens were machined from the fusion zone of each of the alloys based on Ti-6-4 (Fig. 5). The load axis of each was oriented parallel to the welding direction, so that the gage section consisted entirely of LAYERGLAZE-processed material (Fig. 6). The diamond pyramid microhardness of each alloy was measured with a 200 g load on transverse cross-sections of the fusion zones.

As-LAYERGLAZED specimens of each alloy were heat treated at 800°C for 75 min in continuously-purified, flowing argon ($<10^{-7}$ ppm O_2) and furnace cooled.

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RESULTS

A. Chemical Analysis

The results of the chemical analyses of the fusion zone of each alloy are shown in Table II. The microprobe results are assumed to be the most reliable, since standards of known composition were used, and the specimen surfaces were unetched. The results show that some loss of the alloying elements occurred during laser melting. This loss was somewhat more pronounced for those elements with relatively low melting points and for the rare earths.

Table II

Chemical Analyses, As-LAYERGLAZED Alloys (wt%)

Powder Composition	Al	V	Er	Y	Nd	O	C	Sn	Zr	Mo
Ti-6Al-4V, prealloyed powder, Ref. 13	5.8	4.1	-	-	-	.14	ND*	-	-	-
Ti-6Al-4V, mech. blend	7.2	3.9	-	-	-	.13	.02	-	-	-
Ti-6Al-4V-1.5Er	6.4	4.0	>.2	-	-	.12	.02	-	-	-
Ti-6Al-4V-1.5Er**	4.8	4.1	0.7	-	-	ND	ND	-	-	-
Ti-6Al-4V-1.5Y	6.6	ND	-	<3	-	.10	.02	-	-	-
Ti-6Al-4V-3Nd	6.2	3.6	-	-	>.9	.26	ND	-	-	-
Ti-6Al-2Sn-4Zr-6Mo-1Er**	4.9	-	.9	-	-	ND	ND	.8	3.2	4.6

*Not Determined
**Electron Microprobe Data

B. Tensile and Microhardness Data

The tensile properties and/or microhardness of the alloys were determined in the as-LAYERGLAZED condition, and in some cases after an aging heat treatment (800°C, 75 min) as well (Table III). Irregularly-distributed fusion zone porosity in the gage section of several tensile specimens invalidated their test results, and made the data in Table III less complete than anticipated.

Table III

Tensile* and Microhardness Data

Feedstock** Composition (wt%)	Processing History	0.2% YS MPa(ksi)	MPa(ksi)	% ε	Microhardness, DPH, 200g load (mean, ±2s
Ti-6Al-4V	As-LAYER-	931 (135)	1027 (149)	11	388 ± 26
Ti-6Al-4V-1.5Er	GLAZED	848 (123)***	876 (127)	9	376 ± 38
Ti-6Al-4V-1.5Er		945 (137)	1027 (149)	15	373 ± 20
Ti-6Al-4V-3Nd		938 (136)	965 (140)	3	382 ± 32
Ti-6246-1Er		-	-		381 ± 10
Ti-6Al-4V	Annealed,	917 (133)	979 (142)	6	346 ± 10
Ti-6Al-4V-1.5Er	800°C,	-	-	-	361 ± 22
Ti-6Al-4V-1.5Y	75 min	833 (128)	965 (140)	14	371 ± 18
Ti-6Al-4V-3Nd		910 (132)	945 (137)	6	374 ± 10

*Load axis parallel to welding direction: 100% fusion zone gage section
**Nominal composition, mechanically blended powder
***Macroscopic fracture surface porosity

C.   Macrostructure

The macrostructure and fusion zone surfaces of LAYERGLAZE/narrow-gap welds in both 1.27 and 5.08 cm thick specimens are shown in Figs. 7-11. The only distinctive difference between these specimens and those previously LAYERGLAZED from prealloyed Ti-6Al-4V powder (Refs. 3,16) was the relatively dull appearance of the fusion zone free surface of specimens alloyed with rare earth elements (Figs. 8,9). Microstructural analysis (subsequently presented) suggests that this was due to the presence of rare earth oxide particles.

D.   Microstructure, Conventional Metallography

Examination of the as-processed fusion zone microstructures of all specimens by conventional metallography (Figs. 12-23) revealed the columnar grain structure previously described in similar Ti-6Al-4V specimens (Refs. 3,16), and typical of LAYERGLAZE processing in general (Ref. 18). These grains were formed by sequential epitaxial nucleation at the interface of each layer as it solidified (Figs. 10, 12 - 16,18,20). The 5.08 cm specimen was welded by LAYERGLAZING 2.5 cm deep gaps from each side, and displayed some of the large porosity and incompletely-filled fusion zone areas observed similarly-processed 1.27 cm thick specimens (Figs. 12,13). Although the fusion zone grain structures formed by each alloy were quite similar, a greater amount of macroscopic porosity was observed in those which contained a rare earth element. This effect was particularly evident in the fusion zones LAYERGLAZED from Ti-6Al-4V-3Nd and Ti-6246-1Er powder (Figs. 20-22). In the latter case, the different sizes and shapes of the constituent powders caused intermittent blockage of the feedstock flow, so that previously-deposited layers in the fusion zone were often remelted.

Examination of the as-LAYERGLAZED specimens at higher magnification revealed a fully martensitic microstructure (Figs. 17,19,21,23) with no evidence of grain boundary precipitation, as described in detail in Refs. 3 and 16. Also visible were small (0.5-1.6 µm) particles, subsequently identified as rare earth oxides. These were distributed entirely in interdendritic rows in the specimen LAYERGLAZED from Ti-6Al-4V-1.5Y (Fig. 19), and with both random and interdendritic distribtion in the case of the alloys to which Nd or Y had been added (Figs. 17,21).

E.   Aged Specimens

Specimens cut from the as-LAYERGLAZED Ti-6Al-4V base specimens were aged at 800°C for 75 min in an attempt to determine whether additional are earth oxide precipitation would occur. No evidence for additional dispersion _ngthening was apparent in the tensile test data (Table III), although the alloys wi   rare earth additions were somewhat harder after aging than the Ti-6Al-4V alor   iable III. Metallographic observation of these specimens revealed only martensite   e coarsening in the case of Ti-6Al-4V, and some precipitation at grain boun .r   in the Ti-6Al-4V specimens which had been alloyed with rare earth elements ys. 24,25. Previously published results indicated that some Er supersaturation could be anticipated (Ref. 15), but difficulty in TEM specimen preparation from LAYERGLAZED and aged Ti-6Al-4V + Er samples has not as yet allowed a search for the very fine Er_2O_3 precipitation that may have resulted.

F.   Analytical Electron Microscopy

The second phase dispersions in the as-LAYERGLAZED Ti-6Al-4V base alloys were examined by both conventional and scanning transmission electron microscopy. The annular dark field STEM mode was used with a camera constant chosen to suppress Bragg contrast from the martensitic matrix (e.g., Figs. 26,27), and thus reveal the particle distribution more clearly than in a CTEM bright field image. Specimens as-LAYERGLAZED from Ti-6Al-4V + 1.5Er powder contained a bimodal second phase dispersion (Figs. 26,27), with irregularly-shaped particles of 28 nm mean size present at grain boundaries and at random locations within the grains, and some suggestion of interdendritic precipiation (left side of Fig. 26). At higher magnifications, numerous fine, 40 nm precipitates were observed throughout the microstructure (Figs. 27,28). Stereoscopic imaging revealed that some of the rows of fine particles shown in Figs. 27,28 were in fact planar arrays (Fig. 29). This observation suggests either precipitation at martensite plate boundaries or between secondary dendrite arms. The 40 nm particles did not all precipitate with the same orientatin, even within the same martensite plate, and so only a few were in contrast at the same time in conventional dark field images. However, backscattered electron STEM images of areas previously photographed in the annular dark field STEM mode (Fig. 29) showed that all particles contained an element of high atomic number (Er).

Energy-dispersive X-ray spectra were used to confirm that the particles shown in Figs. 26-29 contained Er. An example of such data is presented in Fig. 30, taken in the STEM mode at 120 kV with a 5 nm nominal beam diameter and a 480s live counting time. The smaller particles in this specimen of as-LAYERGLAZED Ti-6Al-4V + Er are shown to produce an X-ray spectrum similar to that from one of the large ones. When the orientation of particles near the foil edge was favorable for diffraction analysis, selected area and convergent beam diffraction patterns were obtained from a particular particle first, then EDS was used to confirm that it contained the appropriate rare earth element.

The crystal structure of each of the three rare earth oxide particles observed in the alloys based on Ti-6Al-4V was determined by quantitative analysis of selected area diffraction patterns, rather than by point and space group identification from convergent beam diffraction patterns as in previous investigations Refs. 13,14. The oxides remained much thicker than the surrounding martensite after electropolishing, so that only those near or at the foil edge were thin enough for detailed study. Interplanar spacings were calculated from selected area diffraction patterns or major zone axes. The diffraction spots from the adjacent martensite, previously determined to have lattice parameters of $a_o = 2.93$Å and $c_o = 4.675$Å in these specimens, were used to calculate the camera constant of each individual pattern. In the case of Er_2O_3 and Y_2O_3, the [100], [110], and [$\bar{1}$11] zone axis patterns were obtained; for Nd_2O_3 [01$\bar{1}$1] and [12$\bar{1}$3] were used. The observed lattice spacings agreed with those calculated from the lattice parameters of the equilibrium oxide structures to within ± 0.02A (Tables IV-VI). Diffraction patterns from the three major zone axes of >100A nm Er_2O_3 particles in specimens as-LAYERGLAZED from Ti-6Al-4V-1.5Er powder are shown in Figs. 31-33. The strong intensities of the fourth order reflections in the convergent beam diffraction patterns are consistent with diffraction from a crystal structure which has space group $I(2_1/a)3$, as noted by Ref. 14.

In contrast to the bimodal oxide size distribution observed in the Ti-6-4 + Er alloy, the Y_2O_3 particles in as-LAYERGLAZED Ti-6Al-4V + Y were observed to be 200-300 nm in size, and to have precipitated only between the primary dendrites (Figs. 34-36). No significant quantity of particles of <60 nm were detected. EDS and diffraction data similar to those obtained from erbia are presented in Figs. 37-38.

The largest rare earth oxide particles (< 600 nm) were present in the specimens as-LAYERGLAZED from Ti-6Al-4V-3Nd, and these had a pronounced interdendritic and intergranular distribution (Figs. 39-41). A few areas of Nd_2O_3 of <100 nm size occurred (Fig. 39), but in general the martensite between the interdendritic rows did not contain precipitates. As with the other specimens, EDS was used to confirm the presence of Nd in a particular particle (Fig. 42); and whenever possible that particle was also analyzed by selected area and convergent beam diffraction (Fig. 43).

Table IV

Selected Area Diffraction Data: Er_2O_3 in as-LAYERGLAZED Ti-6Al-4V

hkl	Calculated Interplanar Spacings (Å)*	Observed Interplanar Spacings (Å) for Zone Axes:		
		[001]	[011]	[$\bar{1}$11]
01$\bar{1}$	7.46	-	7.47	7.48
200	5.27	5.28	5.30	-
211	4.31	-	4.32	4.30
220	3.73	3.73		

*Equilibrium Structure Er_2O_3: a_o = 10.547A; C-M_2O_3 Structure;

Strukturbericht D5$_3$; Space Group $I(2_1/a)3$ (Ia3)

Table V

Selected Area Diffraction Data: Y_2O_3 in as-LAYERGLAZED Ti-6Al-4V

hkl	Calculated Interplanar Spacings (Å)*	Observed Interplanar Spacings (Å) for Zone Axes:		
		[001]	[011]	[1̄11]
011̄	7.48	-	7.47	7.48
200	5.29	5.30	5.30	-
211̄	4.32	-	4.32	4.34
220	3.74	3.75	-	-
310	3.35	3.36	-	-
222̄	3.05	-	3.05	-

*Equilibrium Structure Y_2O_3 : a_o = 10.58Å; C-M₂O₃ Structure; Strukturbericht $D5_3$; SpaceGroup I(2_1 /a)3, (Ia3)

Table VI

Selected Area Diffraction Data: Nd_2O_3 in as-LAYERGLAZED Ti-6Al-4V

hkl	Calculated Interplanar Spacings (Å)*	[011̄1]	[0111]	[1̄21̄3]
101̄0	3.33	-		3.31
11̄01	2.91	2.91		-
1011	2.91	2.92		-
011̄1̄	2.91	-		2.90
01̄12	2.23	2.22		-
2̄11̄0	1.92	1.92		-
112̄1̄	1.83	-		1.83

*Equilibrium Structure Nd_2O_3 : a_o = 3.84Å, c_o = 6.00Å; A-M₂O₃ Structure; Strukturbericht $D5_2$; Space Group P3ml

Er₂O₃ particle distributions similar to those described in Ti-6Al-4V + Er were observed in the alloy LAYERGLAZED from Ti-6Al-2Sn-4Zr-6Mo-1Er powder (Figs. 44,45). 200-400 nm erbia appeared at grain boundaries, and more randomly throughout the α' HCP martensitic microstructure (Fig. 44). 40-60 nm particles were also present, and had a spatial dispersion and characteristic X-ray spectrum very similar to those observed in Ti-6Al-4V + Er (Fig. 45). No evidence for the interdendritic segregation of the other alloying elements was found, as exemplified by the Mo Lα X-ray map shown in Fig. 44.

G.   Auger Electron Spectroscopy

Auger electron spectra were obtained with a 100 nm stationary beam from the sputter-cleaned surfaces of a specimen as-LAYERGLAZED from Ti-6Al-4V-1.5Er. The AES data from a 200 nm interdendritic Er_2O_3 particle and the adjacent matrix are shown in Fig. 46. The α' Ti-6-4 martensite contained some dissolved oxygen and carbon, as expected in this alloy (Fig. 46a), but no carbon signal was detected from the Er_2O_3 particle itself (Fig. 46b). Unfortunately, it was not possible to determine with certainty whether or not the Ti peaks in Fig. 46b actually came from the surrounding matrix. Also, no information could be obtained concerning the incorporation of Al in the Er_2O_3, due to the overlap of the Er and Al signals in the range of 1393-1396 eV. A similar Auger spectrum was obtained from a 250 nm Er_2O_3 particle in a carbon extraction replica taken from a specimen as-LAYERGLAZED fromTi-6Al-2Sn-4Zr-6Mo-1Er powder (Fig. 47). The results are essentially the same as for erbia in Ti-6Al-4V (Fig. 46b), save for the presence of a strong carbon peak, an artifact produced by the replicating material.

DISCUSSION

This study represents a preliminary examination of the potential of rapid solidification technology, and specifically laser processing, for the creation of ODS titanium alloys. The rare earth oxide dispersions observed here were comparable to those reported in other titanium base alloys which had been rapidly solidified by laser processing (Refs. 12-15). The previous identification of these second phases as equilibrium-structure rare earth oxides (Refs. 13-15) was confirmed by quantitative analysis of selected area diffraction patterns from Er_2O_3 and Y_2O_3. The tensile and microhardness data reflected a balance between the removal of oxygen from solid solution by rare earth oxide formation and the strengthening from the dispersion thus formed (Ref. 19). Quantitative evaluation of the alloy strength increase due to the dispersion would have been more accurately achieved at elevated temperature, where the effects of interstitial content and martensitic microstructure are not present.

Of the three rare earth elements added to Ti-6Al-4V in this investigation, only Er formed a fine dispersion (40 nm) of oxide particles within the dendrites, rather than between them. The randomly-oriented rows in which some of these were aligned (Figs. 27-29) suggested that they may have precipitated from solid solution at martensite plate boundaries. A similar observation has been reported in laser-melted Ti-Er (Ref. 15). These results, together with the observation of a bimodal dispersion of Er-rich particles in the alloy LAYERGLAZED from Ti-6246-1Er, indicate that oxide dispersion strengthening via erbium alloying and rapid solidification can be achieved in many commercial titanium alloys.

CONCLUSIONS

1. Good chemical homogenity (μm scale) was observed in specimens LAYER-GLAZED from Ti-6Al-4V-1.5Er and Ti-6Al-2Sn-4Zr-6Mo-1Er. Some loss of alloying elements was observed, particularly for Sn and Er.

2. The primary dendrite spacing of 2-3 μm which was observed in all as-processed alloys indicated an approximate cooling rate during solidification of ~5 x 10^4 °C/s.

3. The addition of Er, Y, or Nd to the Ti-6Al-4V powder feedstock increased the amount of macroscopic fusion zone porosity observed after LAYERGLAZE processing.

4. Rare earth elements react with the oxygen dissolved in titanium to form dispersed, equilibrium-structure oxides during LAYERGLAZE processing.

5. Oxide particle size and spatial distribution were coarsest for Nd_2O_3; finest for Er_2O_3 (bimodal distribtion with 280 nm and 40 nm mean sizes in Ti-6Al-4V).

6. Er_2O_3 particles were observed in specimens as-LAYERGLAZED from Ti-6246-1Er, with a similar distribution and lower quantity than in specimens LAYER-GLAZED from Ti-6-4-1.5Er.

7. The rare earth oxide dispersions did not improve the ambient temperature tensile properties of as-LAYERGLAZED or aged Ti-6Al-4V. However, the 40 nm Er_2O_3 could be expected to improve creep strength. Tensile ductility was increased in both as-processed and aged alloys consolidated from Ti-6Al-4V-1.5Y.

ACKNOWLEDGEMENT

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FIG. 1

NARROW GAP WELD SPECIMEN CONFIGURATIONS
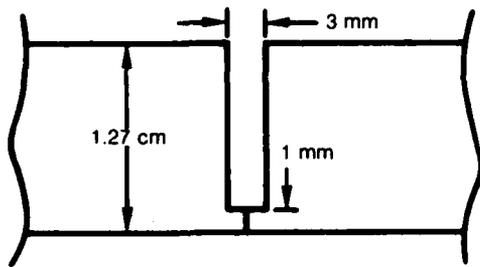
BOTTOM CLOSURE

CENTRAL CLOSURE

84-2-40-1

FIG. 2
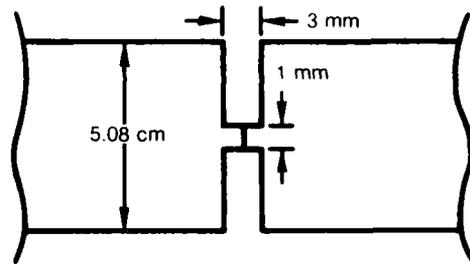
NARROW GAP WELD SPECIMEN POSITIONING FIXTURE

81-448-D

82-8-94-14

FIG. 3
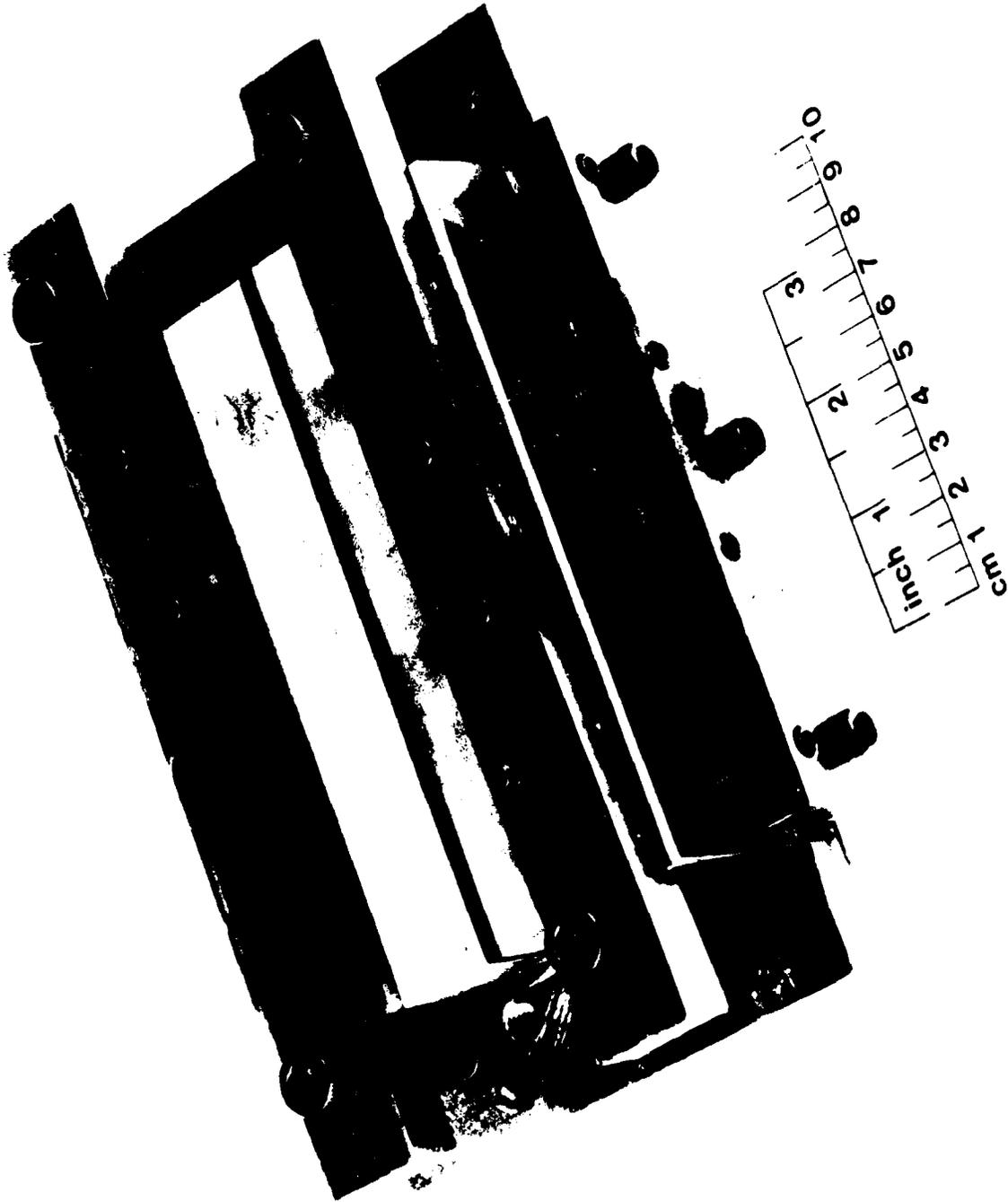
LAYERGLAZE WORK STATION FOR 1.27-cm Ti-6Al-4V PLATE
PROTECTIVE ATMOSPHERE CHAMBER DOOR OPEN

FOCUSING MIRROR
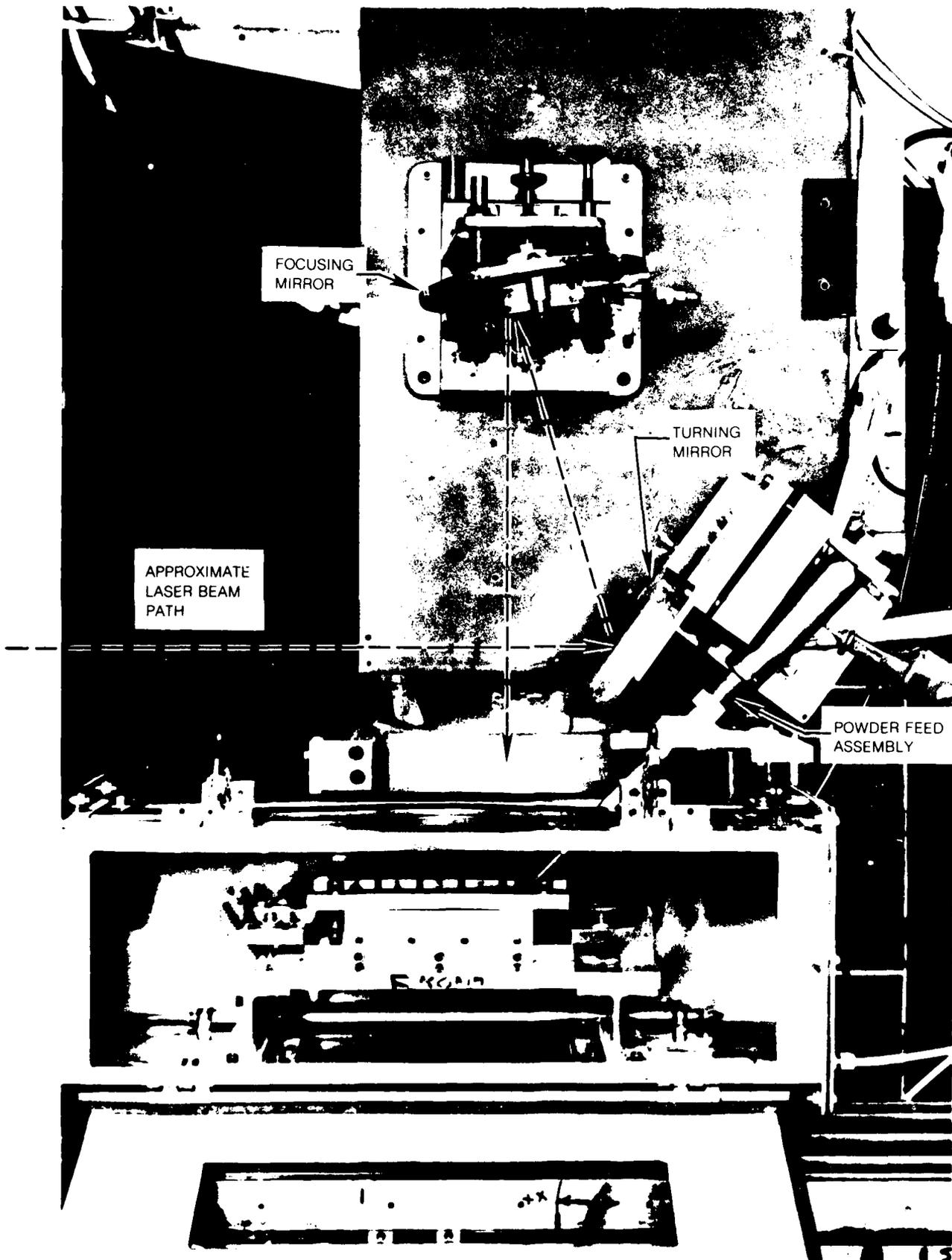
TURNING MIRROR

APPROXIMATE LASER BEAM PATH

POWDER FEED ASSEMBLY

81-448-8

82-8-94-12

LAYERGLAZE WORK STATION FOR 2.54 AND 5.08-cm T-6Al-4V PLATE
MACHINING TABLE LOWERED TO SHOW PROTECTIVE ATMOSPHERE CHAMBER AND SPECIMEN POSITIONING FIXTURE

FIG. 5
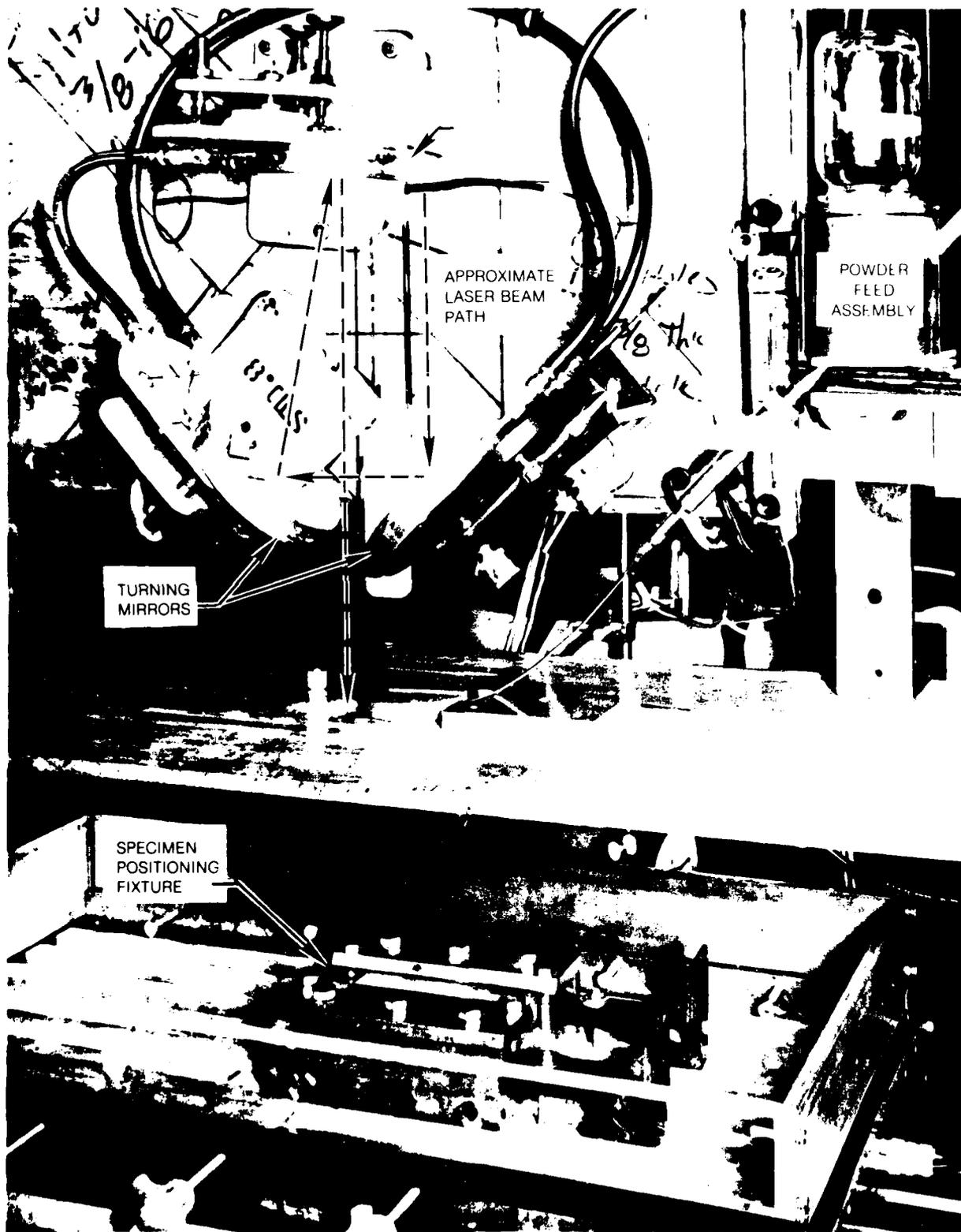
FUSION ZONE TENSILE SPECIMEN

5 mm
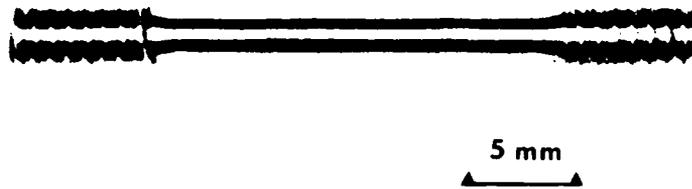
FIG. 6

**LOCATION OF TENSILE SPECIMENS IN FUSION ZONE
... WELDS IN FIG. 7**

FIG. 7
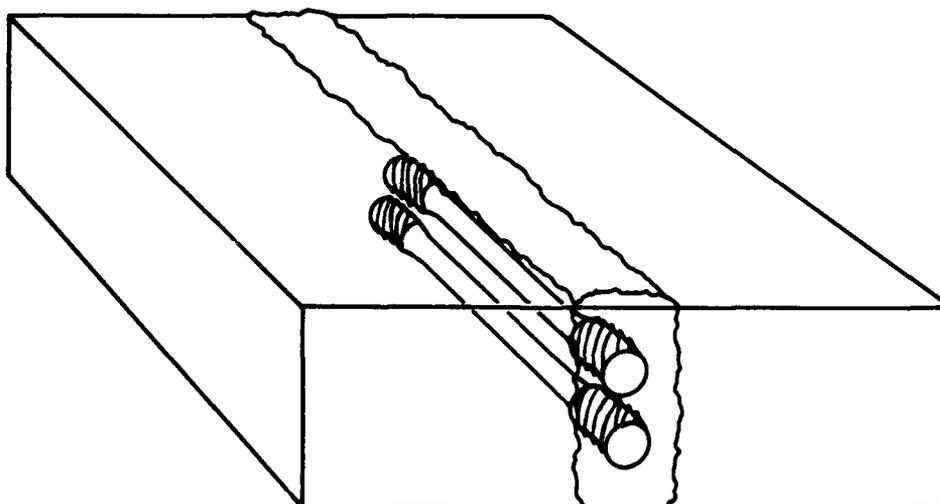
R84-915797-4

NARROW-GAP/LAYERGLAZE WELD SPECIMENS

1.27-cm Ti-6Al-4V PLATE WITH BOTTOM GAP CLOSURE

5 cm

84-2-40-3

FIG. 8
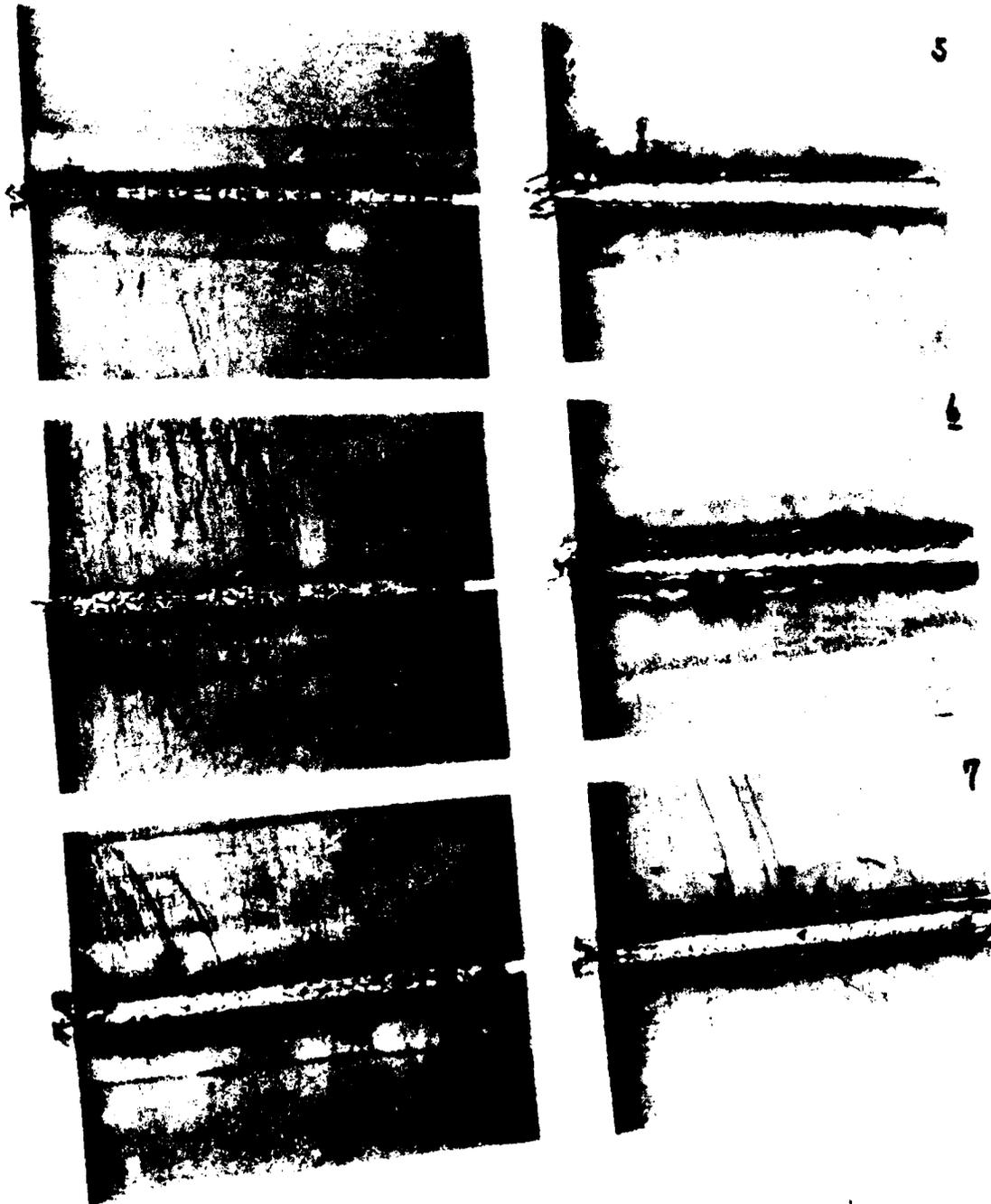
UPPER FREE SURFACE, NARROW-GAP/LAYERGLAZE WELDS IN FIG. 7

Ti-6Al-4V (WT %) POWDER FEEDSTOCK: (1 AND 2) PREALLOYED Ti-6Al-4V. (7) MECHANICALLY BLENDED Ti-6Al-4V

(7)

(2)

(1)

UPPER FREE SURFACE, NARROW-GAP/LAYERGLAZE WELDS IN FIG. 7

MECHANICALLY BLENDED POWDER FEEDSTOCK (WT %): SPECIMEN (3) Ti-6Al-4V-1.5Er, SPECIMEN (5) Ti-6Al-4V-1.5Y, SPECIMEN (6) Ti-6Al-4V-3Nd

LAYERGLAZE/NARROW-GAP WELD SPECIMEN

5.08-cm Ti-6Al-4V PLATE WITH CENTRAL GAP CLOSURE. PREALLOYED Ti-6Al-4V POWDER FEEDSTOCK

2 cm

MACRO PHOTOGRAPH, SIDE 1

SIDE ONE   SIDE TWO

1 cm

WELD CROSS SECTION

84—2—40—7

FIG. 11

TOP SURFACES OF FUSION ZONE, 5.08-cm NARROW-GAP/LAYERGLAZE WELD SPECIMEN

SIDE ONE

1 cm

SIDE TWO

1 cm
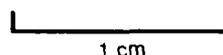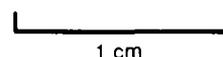
5.08-cm LAYERGLAZE/NARROW-GAP WELD SPECIMEN; GRAIN STRUCTURE OF SIDE ONE

3 mm

5.08-cm LAYERGLAZE/NARROW-GAP WELD SPECIMEN; GRAIN STRUCTURE OF SIDE TWO

3 mm

84-2-40-9

1.27-cm LAYERGLAZE/NARROW GAP WELD SPECIMEN NO. 7, FUSION ZONE GRAIN STRUCTURE

(MECHANICALLY-BLENDED Ti-6Al-4V POWDER FEEDSTOCK. NO RARE-EARTH ELEMENT ADDED)

LASER BEAM
DIRECTION

℄ OF
FUSION ZONE

1mm

84-2-40-20

1.27-cm LAYERGLAZE/NARROW GAP WELD SPECIMEN NO. 3, FUSION ZONE GRAIN STRUCTURE

(MECHANICALLY-BLENDED Ti-6Al-4V-1.5 Er POWDER FEEDSTOCK)

LASER BEAM
DIRECTION

₵ OF
FUSION ZONE

1mm

84-2-40-19

...

R84-915797-4

FIG. 16

1.27-cm LAYERGLAZE/NARROW GAP WELD SPECIMEN NO. 3.
VARIATION IN MICROSTRUCTURE ACROSS WELD

(MECHANICALLY-BLENDED Ti-6Al-4V-1.5Er POWDER FEEDSTOCK)

84-2-40-18
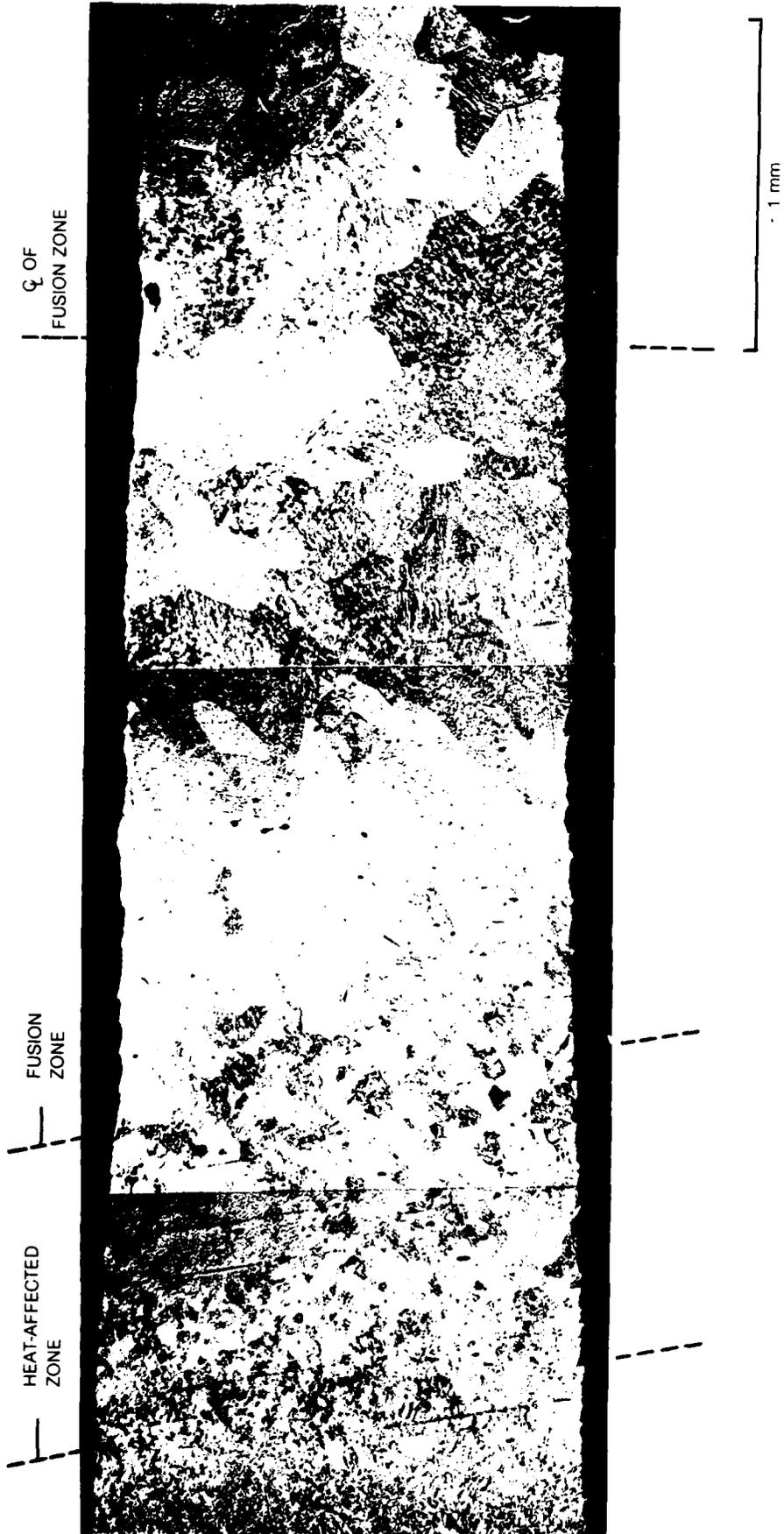
AS-LAYERGLAZED FUSION ZONE MICROSTRUCTURE, SPECIMEN NO. 3
(LIGHT MICROGRAPH, ERBIUM OXIDE PARTICLES IN α' HCP MARTENSITE)

10µm

84-2-40-17

FIG. 18

1.27-cm LAYERGLAZE/NARROW GAP WELD SPECIMEN NO. 5, FUSION ZONE GRAIN STRUCTURE

(MECHANICALLY-BLENDED Ti-6Al-4V-1.5Y POWDER FEEDSTOCK)

LASER BEAM DIRECTION

₵ OF FUSION ZONE

1mm

84-2-40-16

FIG. 19

R84-915797-4

AS-LAYERGLAZED FUSION ZONE MICROSTRUCTURE, SPECIMEN NO. 5
(LIGHT MICROGRAPH, YTTRIUM OXIDE PARTICLES IN α' HCP MARTENSITE)

10μm

1.27-cm LAYERGLAZE/NARROW GAP WELD SPECIMEN NO. 6, FUSION ZONE GRAIN STRUCTURE
(MECHANICALLY-BLENDED Ti-6Al-4V-3Nd POWDER FEEDSTOCK)

LASER BEAM
DIRECTION

₵ OF
FUSION ZONE

1mm

84-2-40-14

FIG. 21

AS-LAYERGLAZED FUSION ZONE MICROSTRUCTURE, SPECIMEN NO. 6

(LIGHT MICROGRAPH, NEODYMIUM OXIDE IN α' HCP MARTENSITE)

10μm

FUSION ZONE GRAIN STRUCTURE
SPECIMEN AS-LAYERGLAZED FROM MECHANICALLY-BLENDED Ti-6Al-2Sn-4Zr-6Mo-1Er POWDER FEEDSTOCK

MICROHARDNESS
INDENTATION

POROSITY

200μm

84-2-40-12

FIG. 23
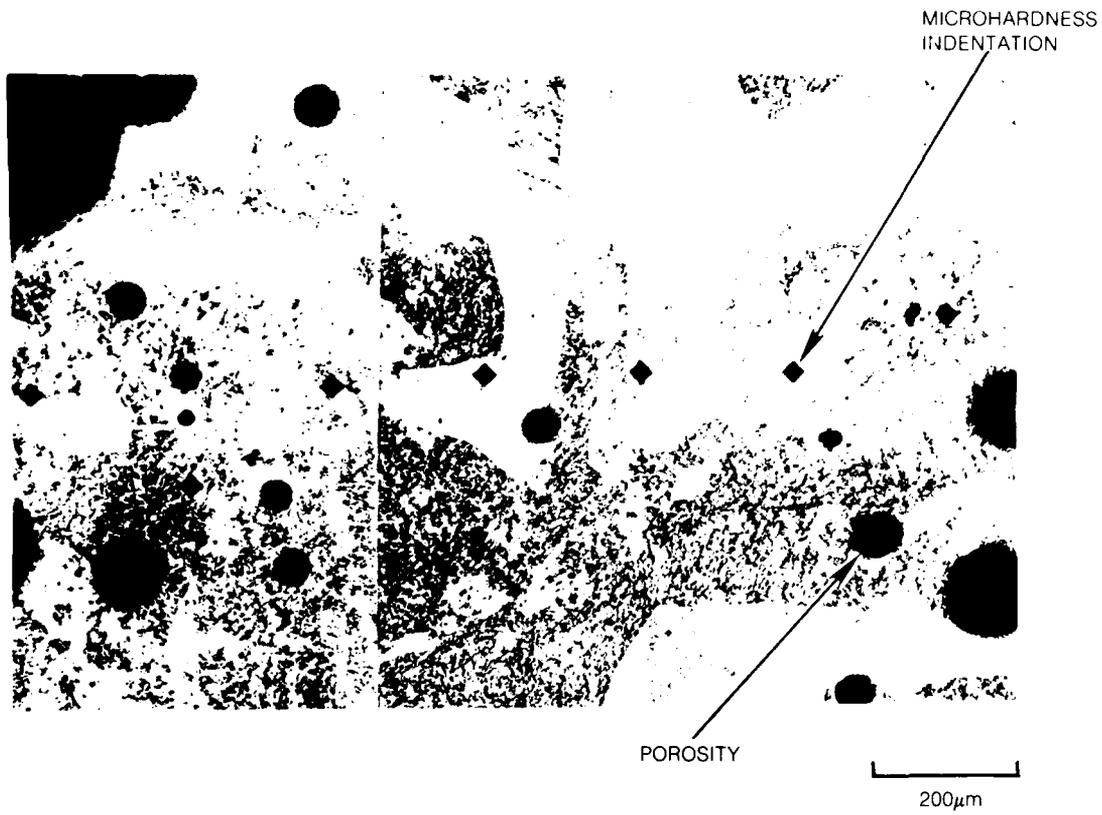
FUSION ZONE MICROSTRUCTURE, SPECIMEN
AS-LAYERGLAZED FROM Ti-6242-1 Er POWDER

(α' HCP MARTENSITE; MOST ERBIUM OXIDE PARTICLES TOO SMALL TO BE VISIBLE)

20µm

84-2-40-11

AGED FUSION ZONE MICROSTRUCTURES
(SPECIMENS LAYERGLAZED AND ANNEALED AT 800°C (1472°F) FOR 75 min)
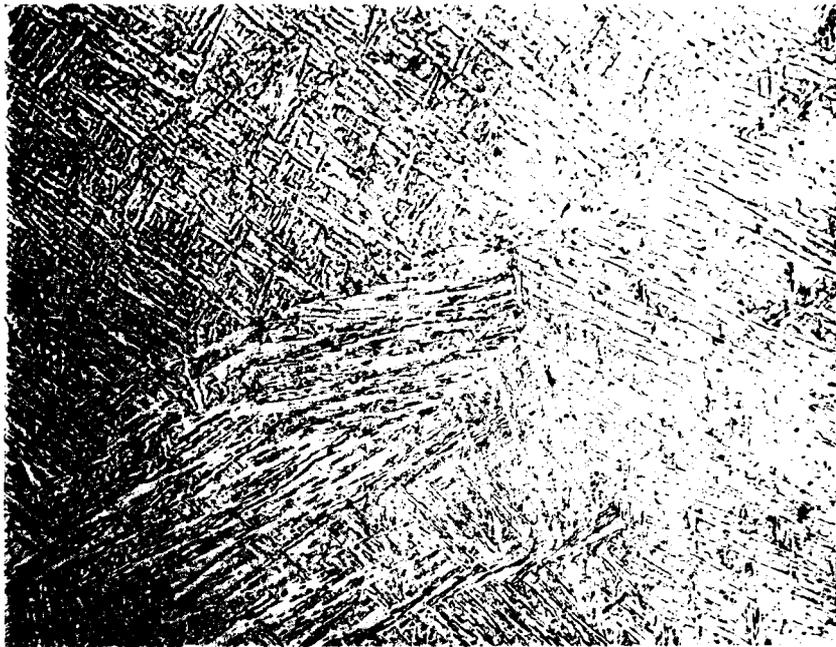
MECHANICALLY-BLENDED Ti-6Al-4V
POWDER FEEDSTOCK

20μm

MECHANICALLY BLENDED Ti-6Al-4V-3Nd
POWDER FEEDSTOCK

20μm

84—2—40—31

FIG. 25
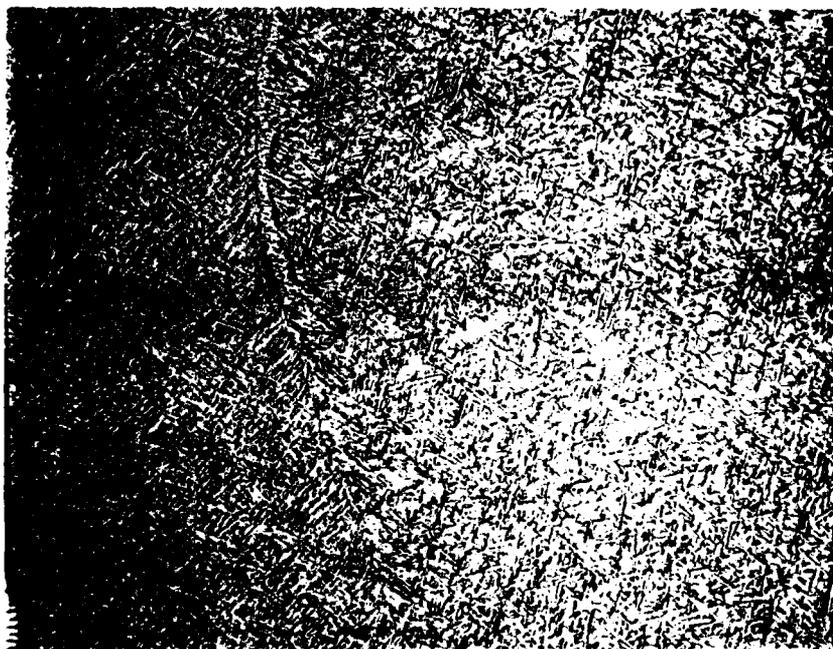
AGED FUSION ZONE MICROSTRUCTURES
(SPECIMENS LAYERGLAZED AND ANNEALED AT 800°C (1472°F) FOR 75 min)

MECHANICALLY-BLENDED Ti-6Al-4V-1.5Y FEEDSTOCK
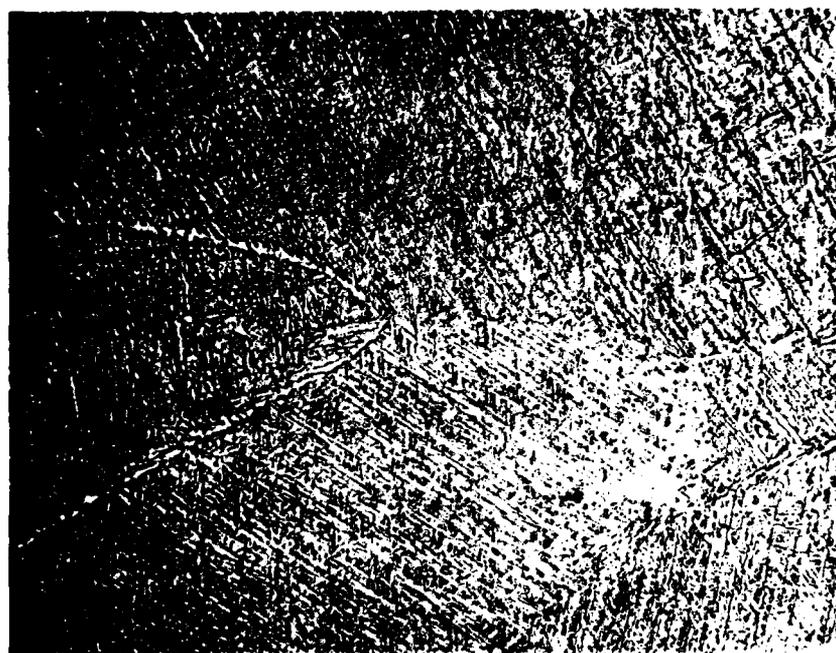
MECHANICALLY-BLENDED Ti-6Al-4V-1.5Er FEEDSTOCK

20μm

DISTRIBUTION OF Er_2O_3 IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Er POWDER

ANNULAR DARK FIELD STEM IMAGE

GRAIN
BOUNDARY

B

C

A
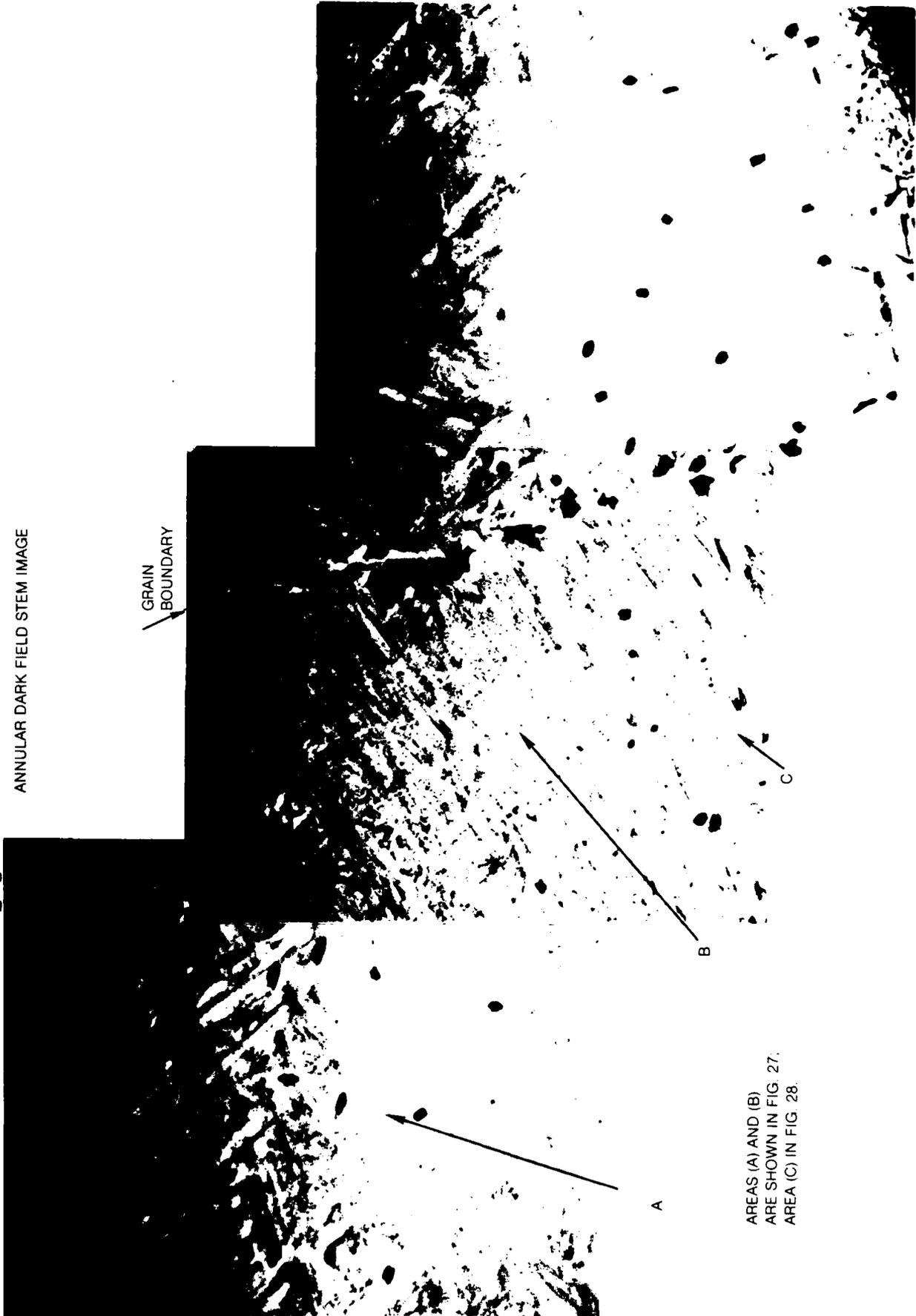
AREAS (A) AND (B)
ARE SHOWN IN FIG. 27.
AREA (C) IN FIG. 28.

84—2—40—29

FIG. 27

BIMODAL SIZE DISTRIBUTION OF Er_2O_3 IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Er

MEAN SIZES OF 280 AND 40 nm
ANNULAR DARK FIELD STEM IMAGES
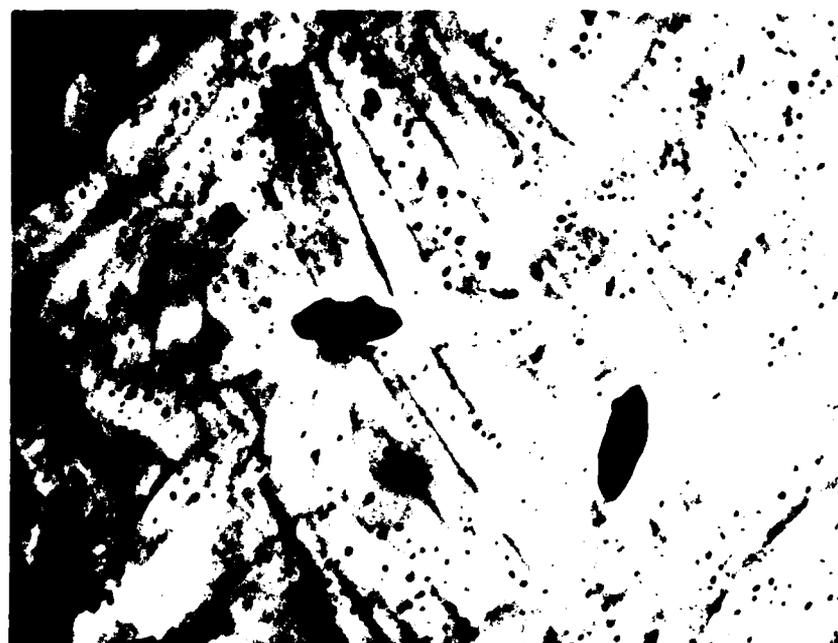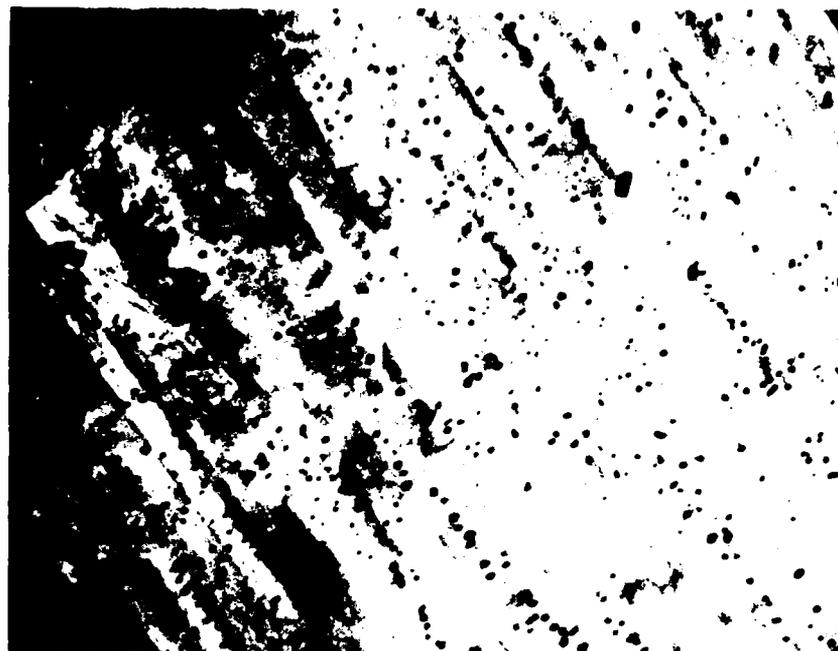
AREA (B) IN FIG. 26

400 nm

AREA (A) IN FIG. 26

FIG. 28

BIMODAL SIZE DISTRIBUTION OF Er_2O_3 IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Er POWDER

AREA (C) IN FIG. 26
BRIGHT FIELD CTEM IMAGE

$1 \mu m$

84—2—40—27

FIG. 29
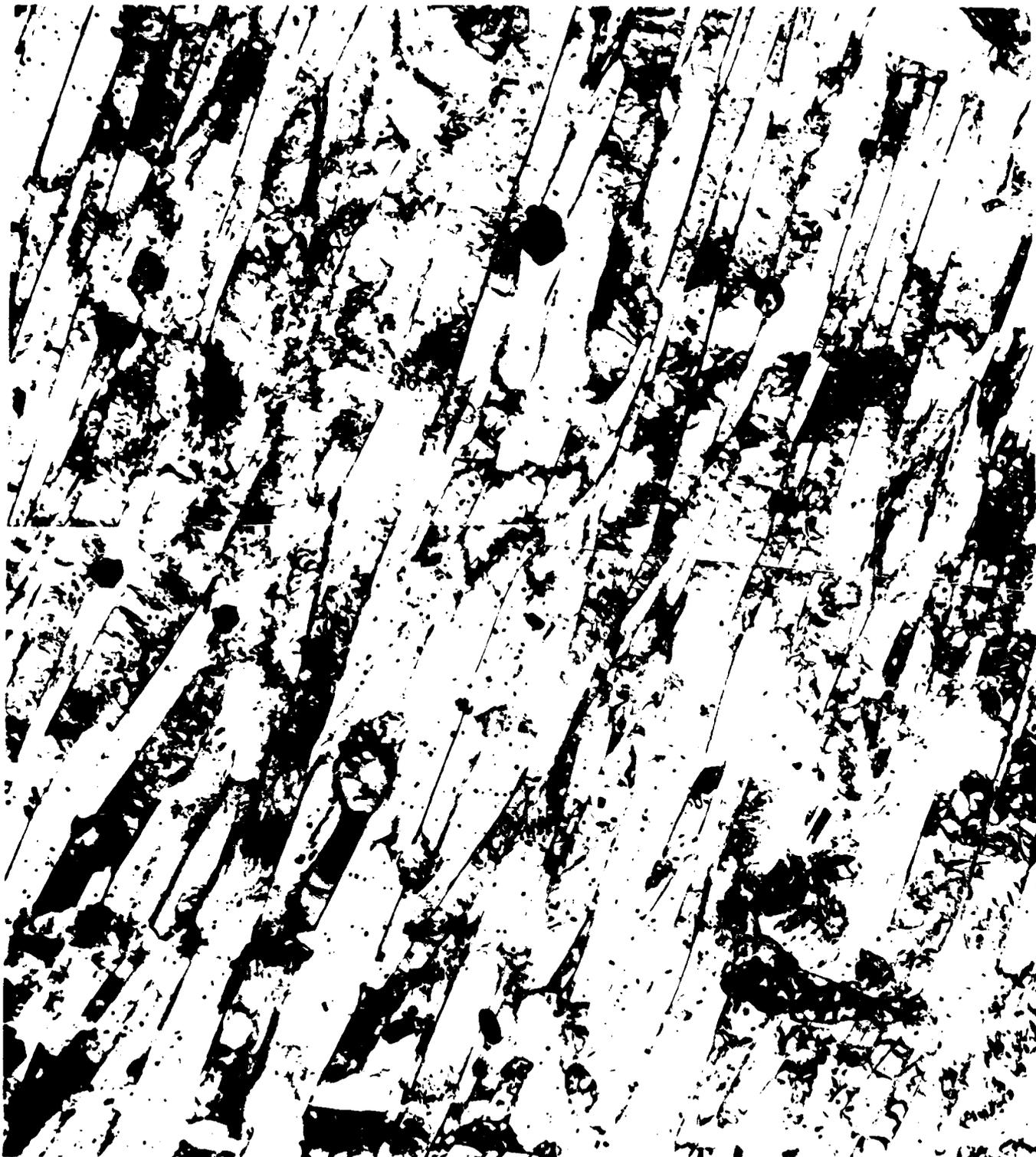
Er_2O_3 DISTRIBUTION IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Er POWDER

(B) BACKSCATTERED ELECTRON IMAGE, AREA OF (A)

400 nm
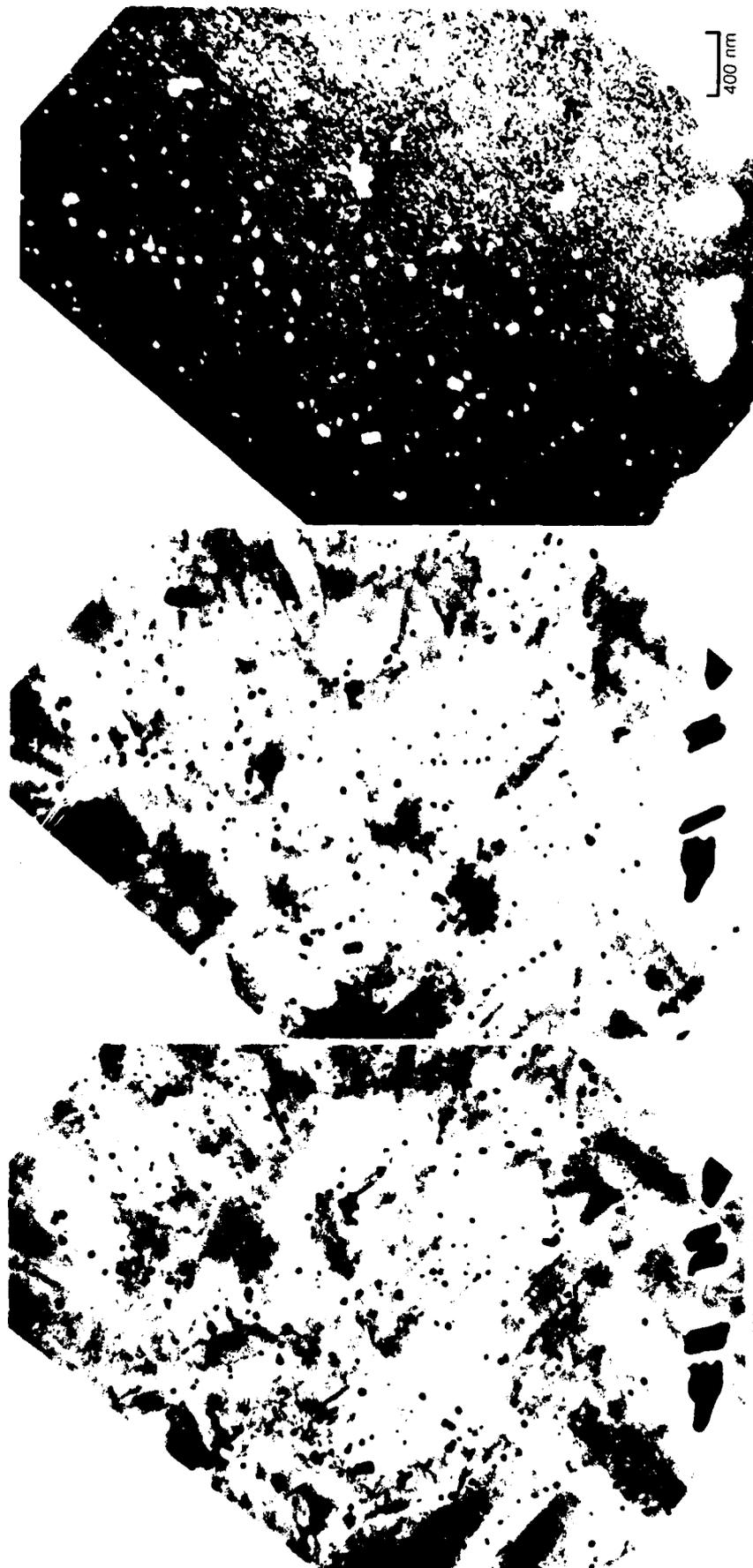
(A) ANNULAR DARK FIELD STEM STEREO IMAGE

84-2-40-26

FIG. 30

STEM IMAGE AND ENERGY DISPERSIVE X-RAY SPECTRUM, Er_2O_3 IN SPECIMEN
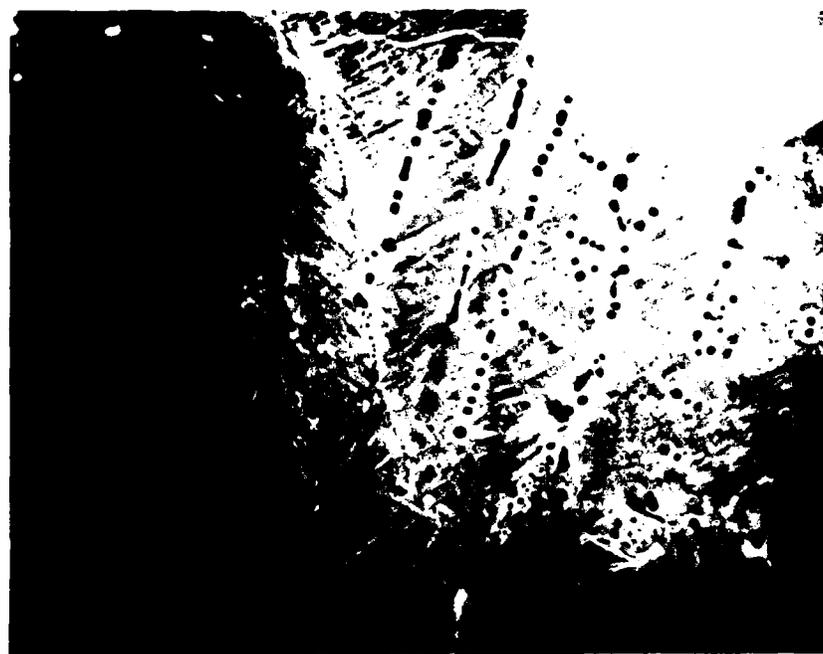AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Er POWDER

120 kV; 5 nm BEAM SIZE; Al Kα AND Er Mα PEAKS OVERLAP

FIG 31
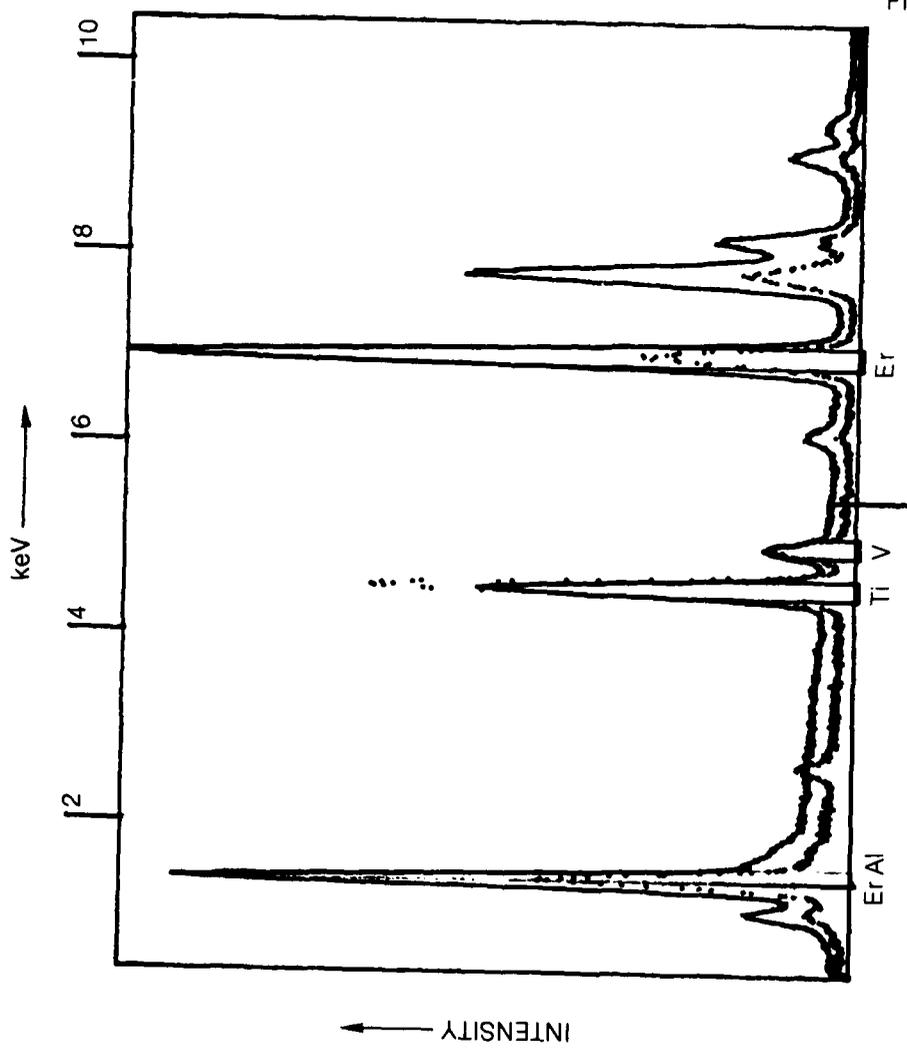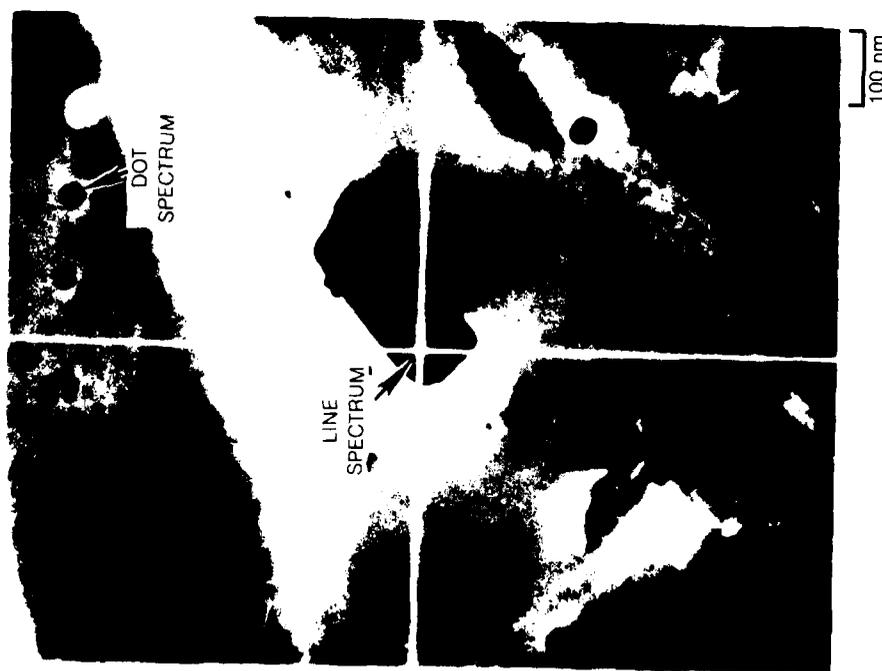
INTERDENDRITIC Er$_2$O$_3$ IN SPECIMEN AS-LAYERGLAZED^{SM} FROM Ti-6Al-4V-1.5Er POWDER

(A) DARK FIELD CTEM, g = 400 (B) SELECTED AREA DIFFRACTION PATTERN FROM (A). [001] ZONE AXIS. POINTER MARKS 400
(C) AND (D) [001] ZONE AXIS CONVERGENT BEAM DIFFRACTION PATTERNS FROM (A). 40 nm BEAM SIZE

(A)

(B)

(C)
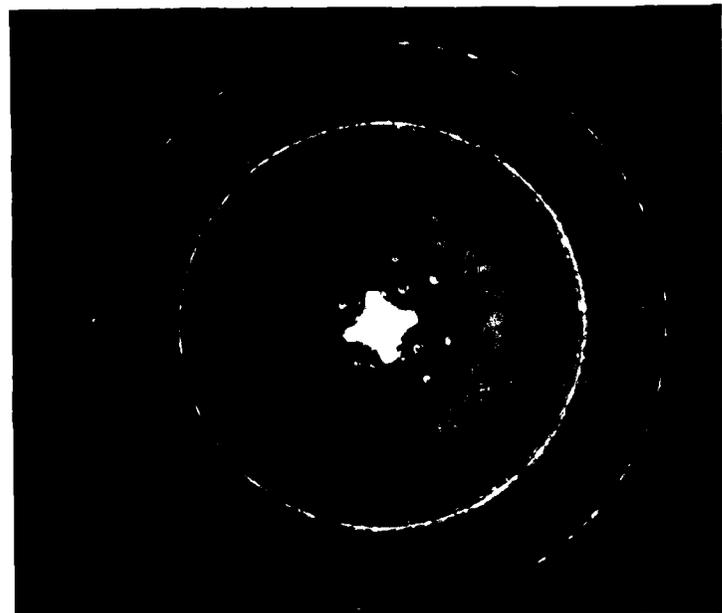
(D)

84-2-40-34

FIG. 32

INTERDENDRITIC Er$_2$O$_3$ IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Er POWDER

(A) DARK FIELD CTEM. g = 22$\bar{2}$. (B) SELECTED AREA DIFFRACTION PATTERN FROM (A). [011] ZONE AXIS. POINTER MARKS 22$\bar{2}$
(C) AND (D) [011] ZONE AXIS CONVERGENT BEAM DIFFRACTION PATTERNS FROM (A). 40 nm BEAM SIZE
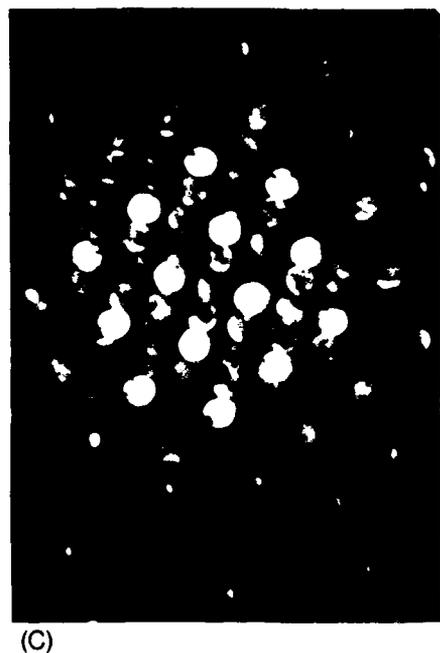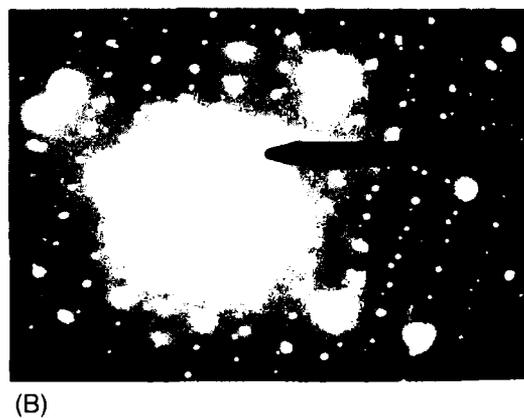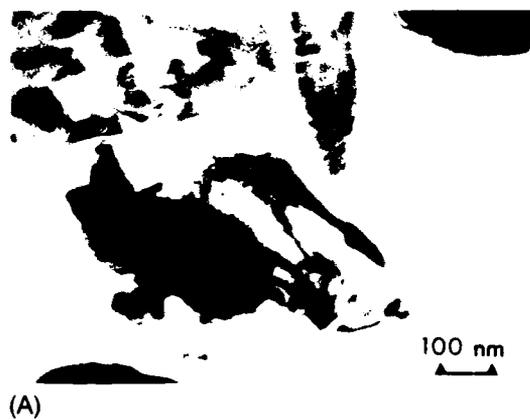
(A)   100 nm

(B)

(C)

(D)

84-2-40-33

INTERDENDRITIC Er_2O_3 IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Er POWDER

(A) DARK FIELD CTEM, g = 440. (B) SELECTED AREA DIFFRACTION PATTERN FROM (A), [$\bar{1}$11] ZONE AXIS. POINTER MARKS 440
(C) AND (D) [$\bar{1}$11] ZONE AXIS CONVERGENT BEAM DIFFRACTION PATTERNS FROM (A), 40 nm SPOT SIZE

(A)

(B)

(C)

(D)

84-2-40-35
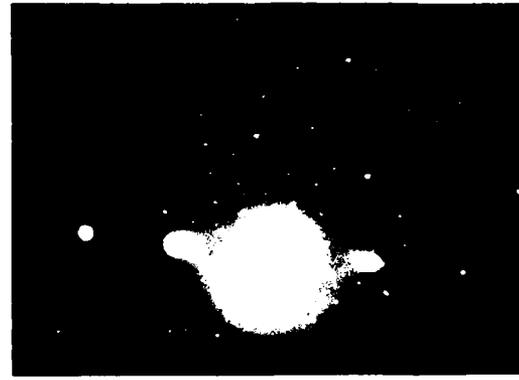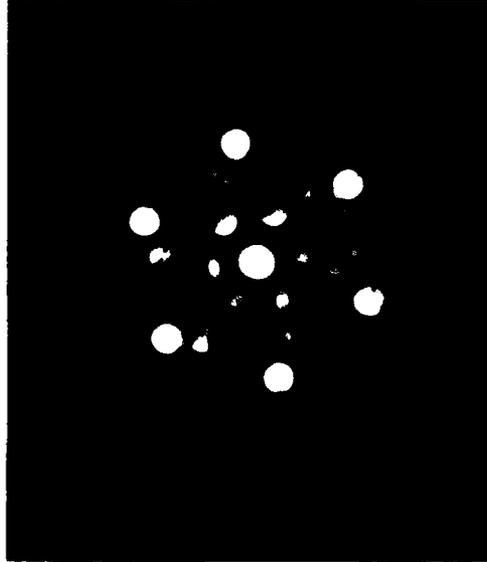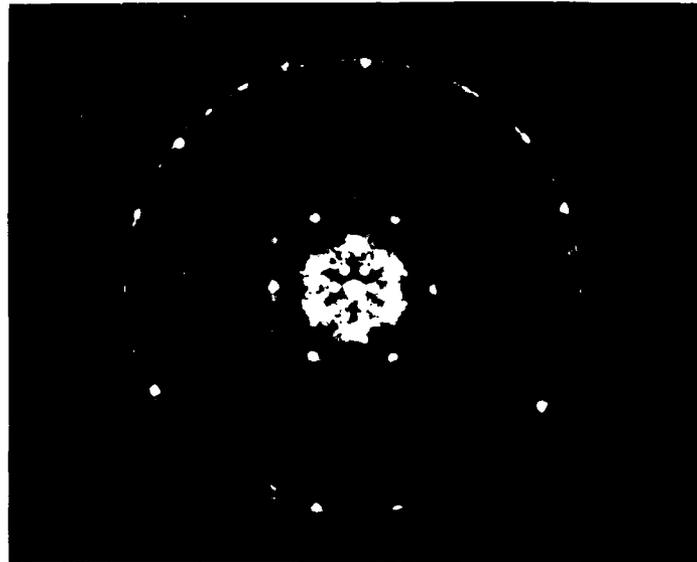
DISTRIBUTION OF Y_2O_3 IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Y

PREDOMINATELY INTERDENDRITIC Y_2O_3 PRECIPITATION

ANNULAR DARK FIELD STEM IMAGE
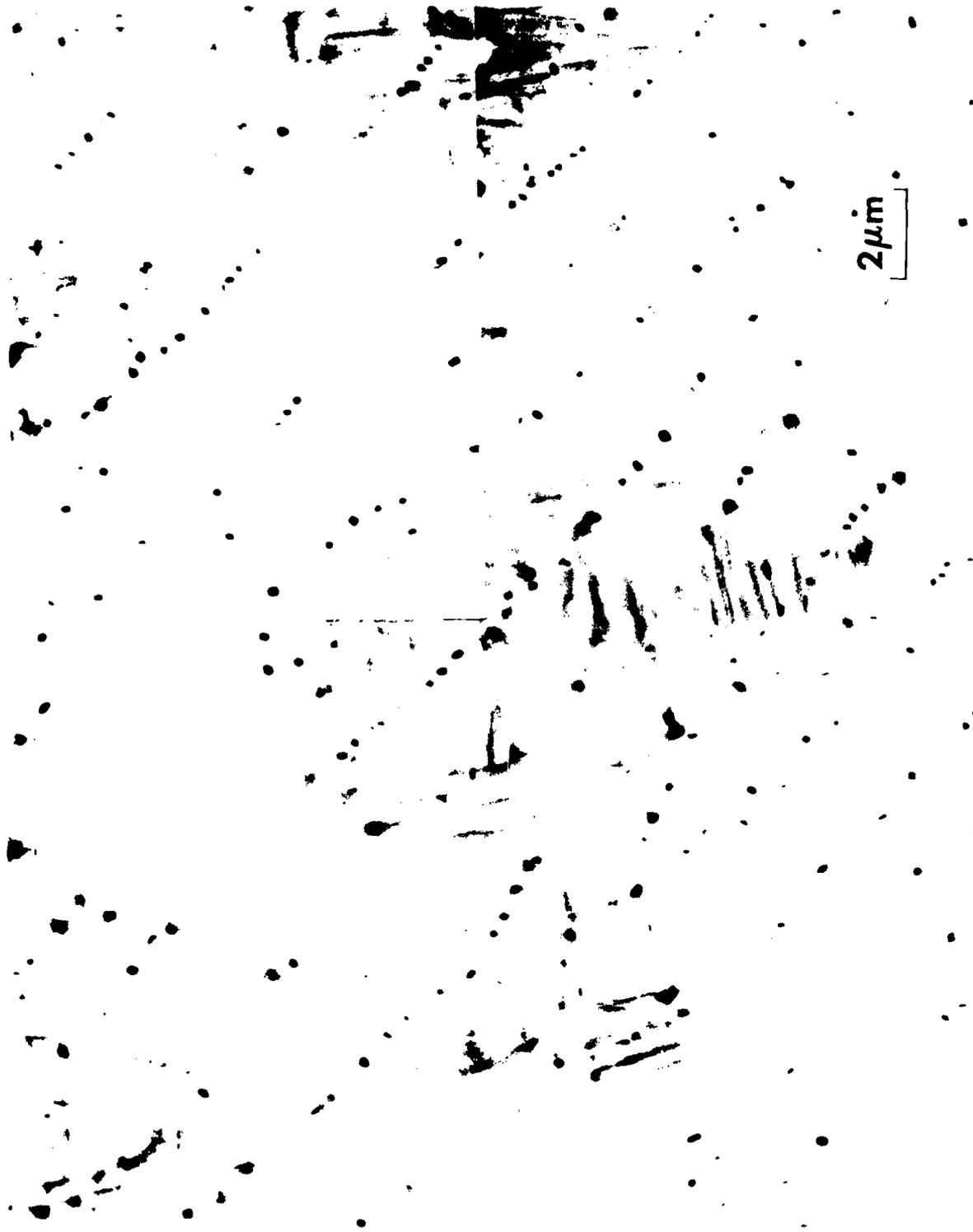
2 μm

DISTRIBUTION OF Y_2O_3 IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Y POWDER

BRIGHT FIELD CTEM
AREA AT LOWER RIGHT OF FIG. 34, MIRROR IMAGE

1μm

84-2-40-23

Y$_2$O$_3$ DISTRIBUTION IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Y POWDER

ANNULAR DARK FIELD STEM STEREO IMAGE
AREA IN LOWER RIGHT, FIG. 34, ROTATED 90°

2μm

84–2–40–22

FIG. 37
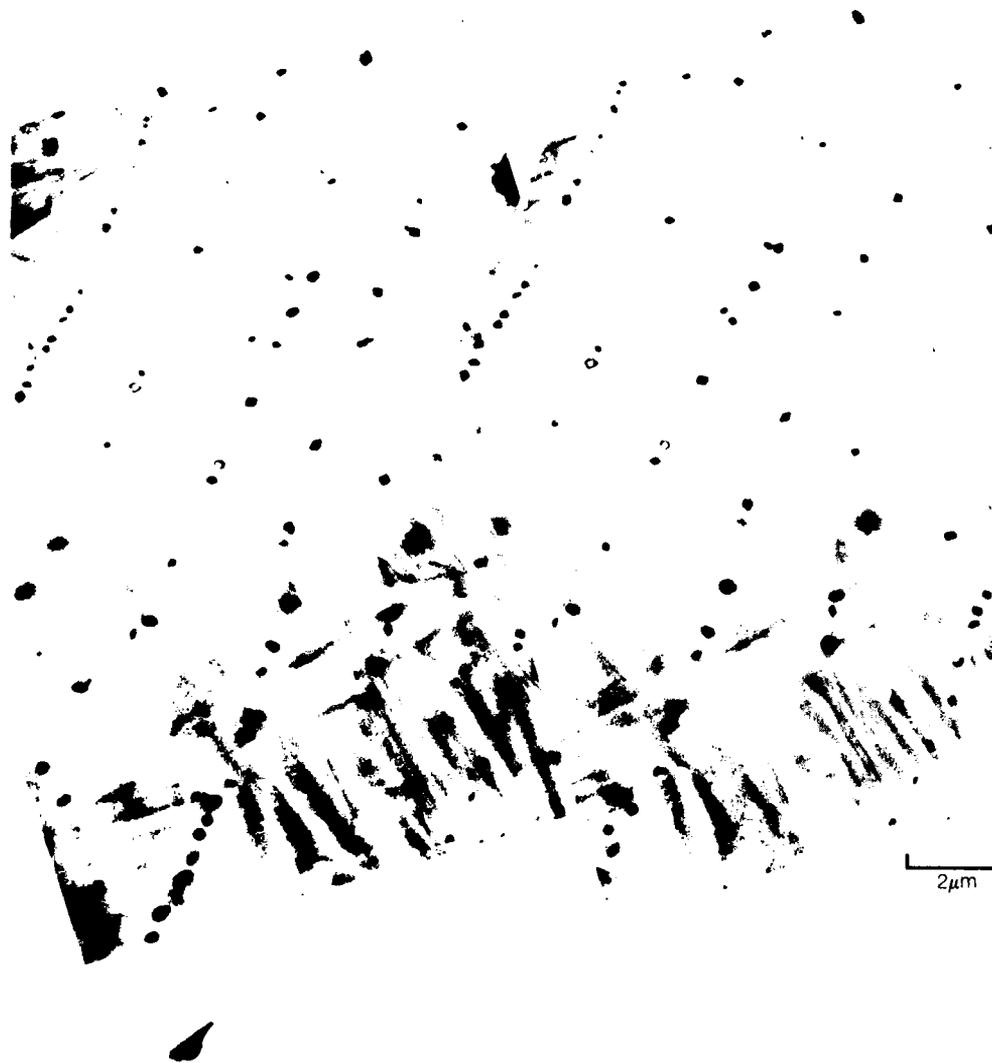
STEM IMAGE AND ENERGY DISPERSIVE X-RAY SPECTRUM, Y_2O_3 IN SPECIMEN
AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Y POWDER

INTERDENDRITIC Y₂O₃ IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-1.5Y POWDER

(A) DARK FIELD CTEM, g = 400. (B) SELECTED AREA DIFFRACTION PATTERN FROM (A). [001] ZONE AXIS, POINTER MARKS 400. (C) [001] ZONE AXIS CONVERGENT BEAM DIFFRACTION PATTERN FROM (A), 40 nm BEAM SIZE

FIG. 39

DISTRIBUTION OF Nd_2O_3 IN SPECIMEN
AS-LAYERGLAZED FROM Ti-6Al-4V-3Nd POWDER

PREDOMINATELY INTERDENDRICTIC Nd_2O_3 PRECIPITATION
ANNULAR DARK FIELD STEM IMAGES

GRAIN
BOUNDARY

$5\mu m$
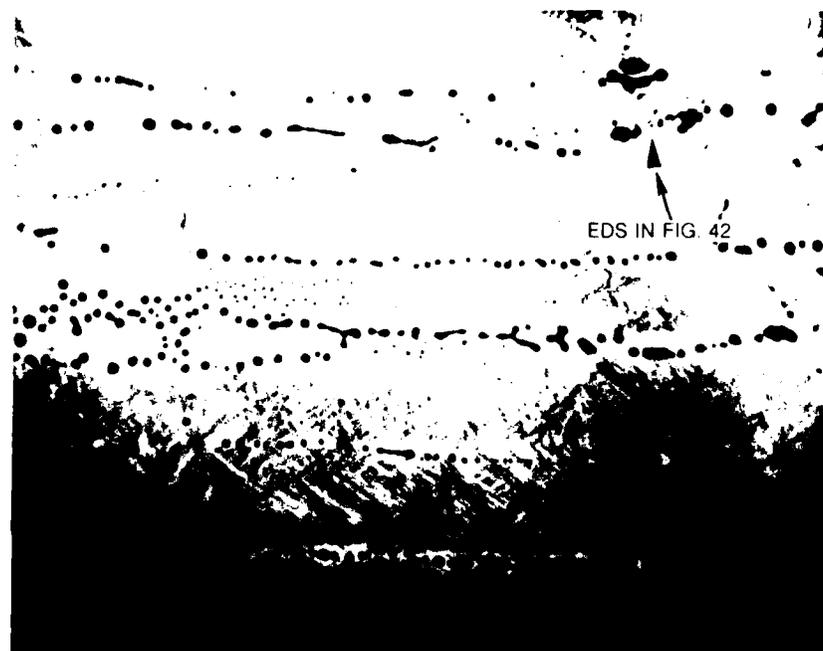
EDS IN FIG. 42

$5\mu m$

84-2-40-40

R84-915797-4

FIG. 40

DISTRIBUTION OF Nd_2O_3 IN SPECIMEN
AS-LAYERGLAZED FROM Ti-6Al-4V-3Nd POWDER

BRIGHT FIELD CTEM

MIRROR IMAGE OF AREA IN UPPER PHOTOMICROGRAPH, FIG. 39

GRAIN
BOUNDARY

5μm

84-2-40-39

FIG 41

DISTRIBUTION OF Nd_2O_3 IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-3Nd POWDER

BRIGHT FIELD CTEM.   AREA IN RIGHT OF FIG. 40

GRAIN BOUNDRY

GRAIN BOUNDRY

1 µm

84-2-40-38

FIG. 42

ANNULAR DARK FIELD STEM IMAGE AND ENERGY DISPERSIVE X-RAY SPECTRUM,
Nd_2O_3 IN SPECIMEN AS-LAYERGLAGED FROM Ti-6Al-4V-3Nd

LOCATION OF Nd_2O_3 SHOWN BY ARROW IN LOWER PHOTOMICROGRPAH OF FIG. 39.

120kV, 5nm BEAM SIZE

84-2-40-37
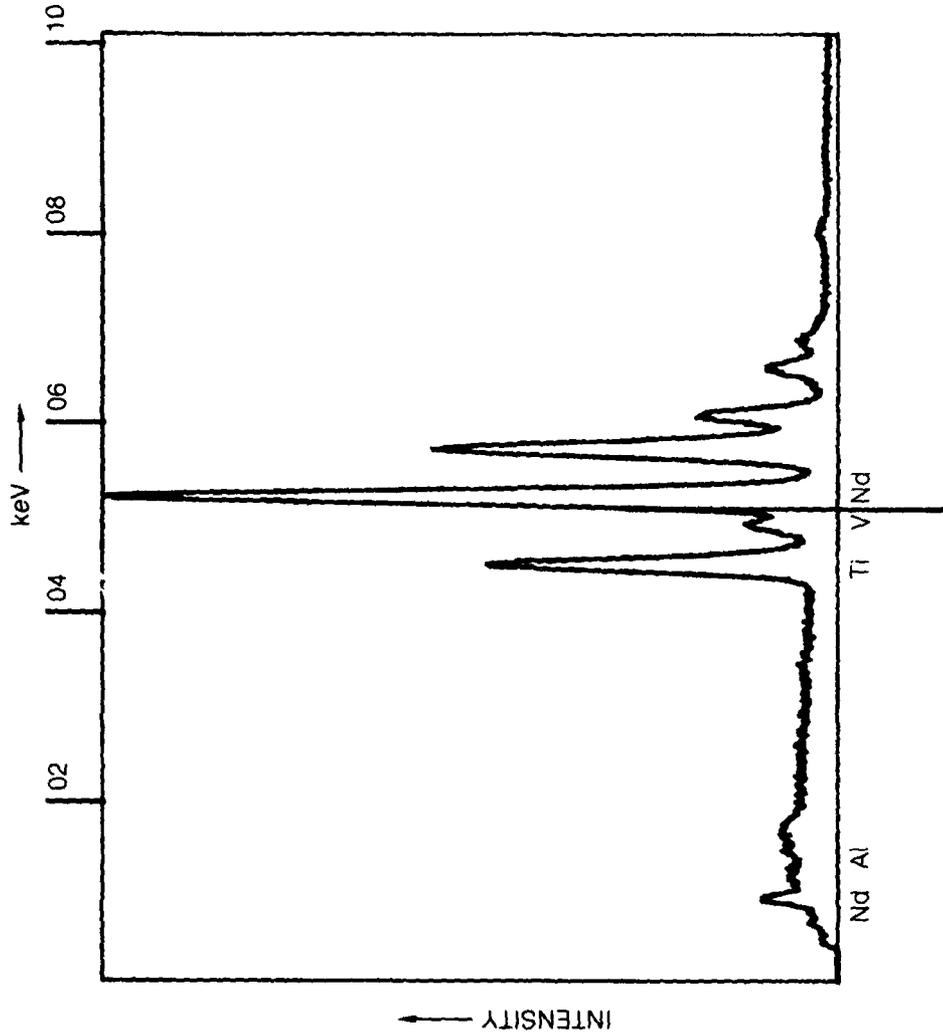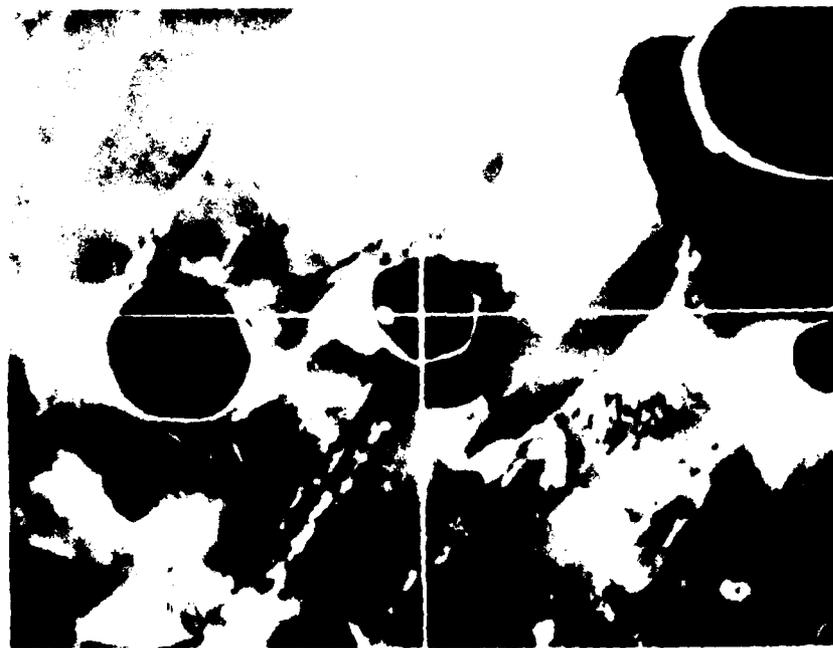
INTERDENDRITC Nd_2O_3 IN SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-4V-3Nd POWDER

(A) BRIGHT FIELD CTEM. PARTICLE SHOWN IN FIG. 42 (B) SELECTED AREA DIFFRACTION PATTERN FROM (A). $[1\bar{2}1\bar{3}]$ ZONE AXIS, POINTER COVERS 0000.

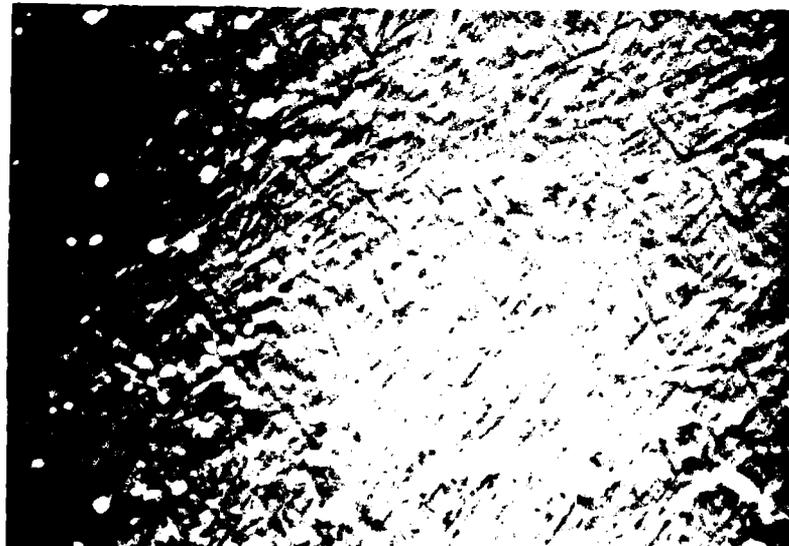(C) AND (D) $[1\bar{2}1\bar{3}]$ ZONE AXIS CONVERGENT BEAM DIFFRACTION PATTERNS FROM PARTICLE IN (A). 40mm BEAM SIZE.
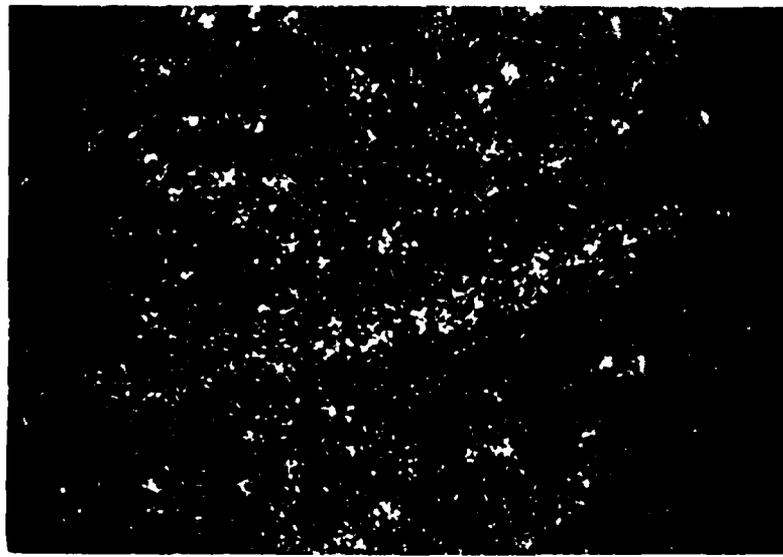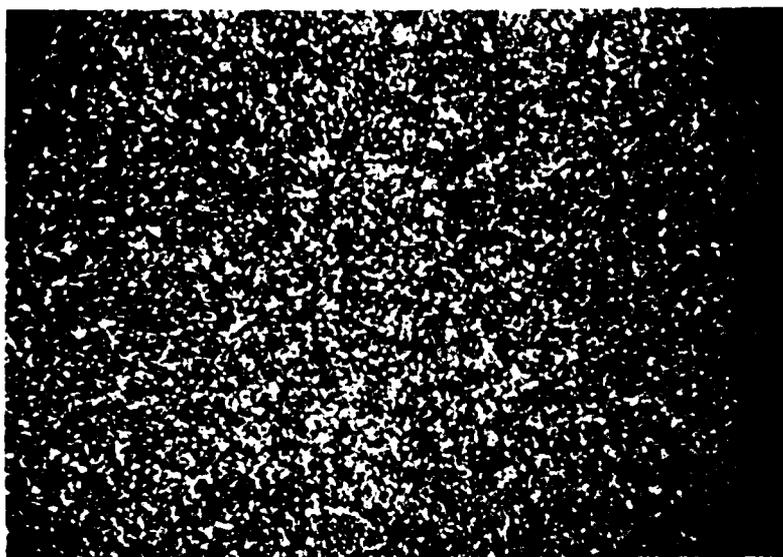

(B)

100mm

(A)
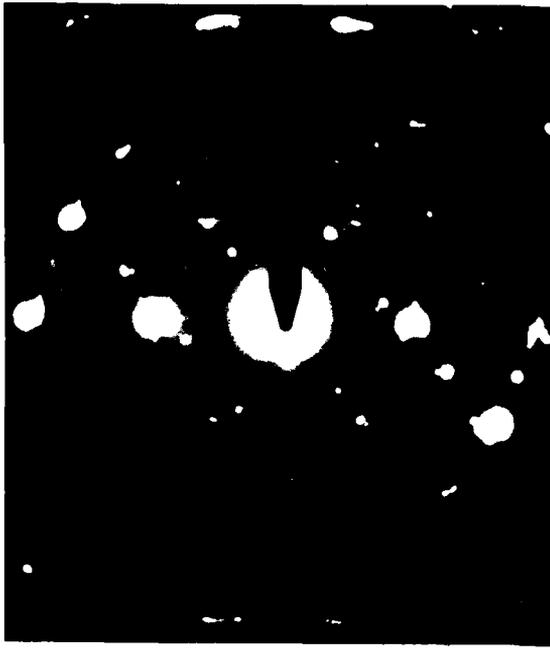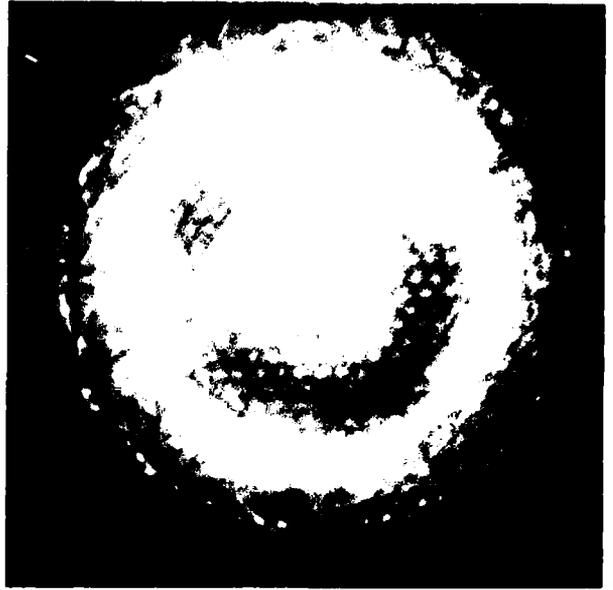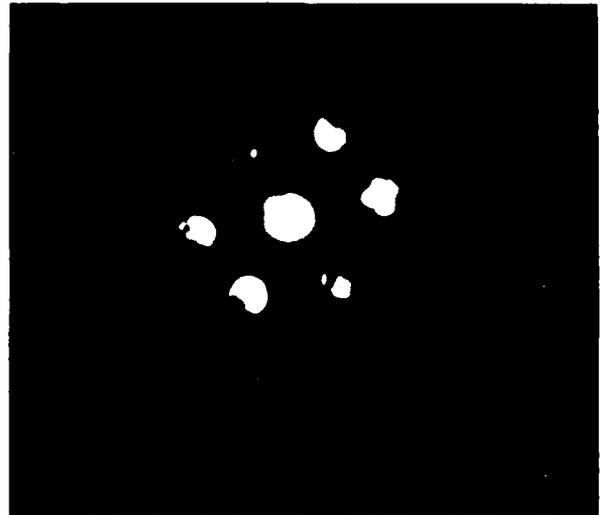
FIG. 44

ELECTRON MICROPROBE IMAGES OF SPECIMEN AS-LAYERGLAZED FROM Ti-6Al-2Sn-4Zr-6Mo-1Er

(C) Mo Lα X-RAY IMAGE. AREA OF (A)

(B) Er Lα X-RAY IMAGE. AREA OF (A)

(A) BACKSCATTERED ELECTRON IMAGE.
ARROWS SHOW Er_2O_3 PRECIPITATION
AT GRAIN BOUNDARY.

2μm

84--2--40--41

FIG. 45

DISTRIBUTION AND EDS DATA, Er₂O₃ IN SPECIMEN
AS-LAYERGLAZED FROM Ti-6Al-2Sn-4Zr-6Mo POWDER
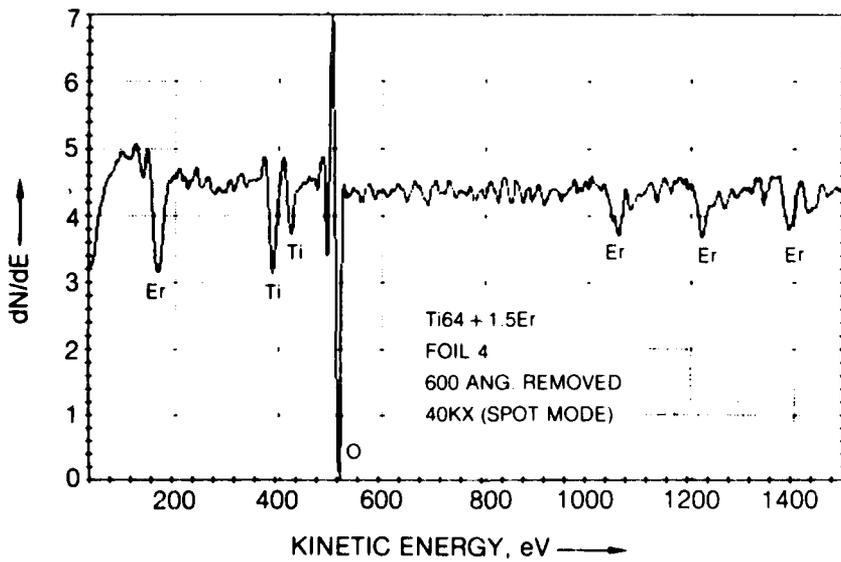

(B) ENERGY DISPERSIVE X-RAY SPECTRUM FROM INTERDENDRITIC Er₂O₃ PARTICLE SHOWN BY ARROW IN (A). 120 kV. 5nm BEAM SPOT DIAMETER. Cu AND S PEAKS ARE ARTIFACTS
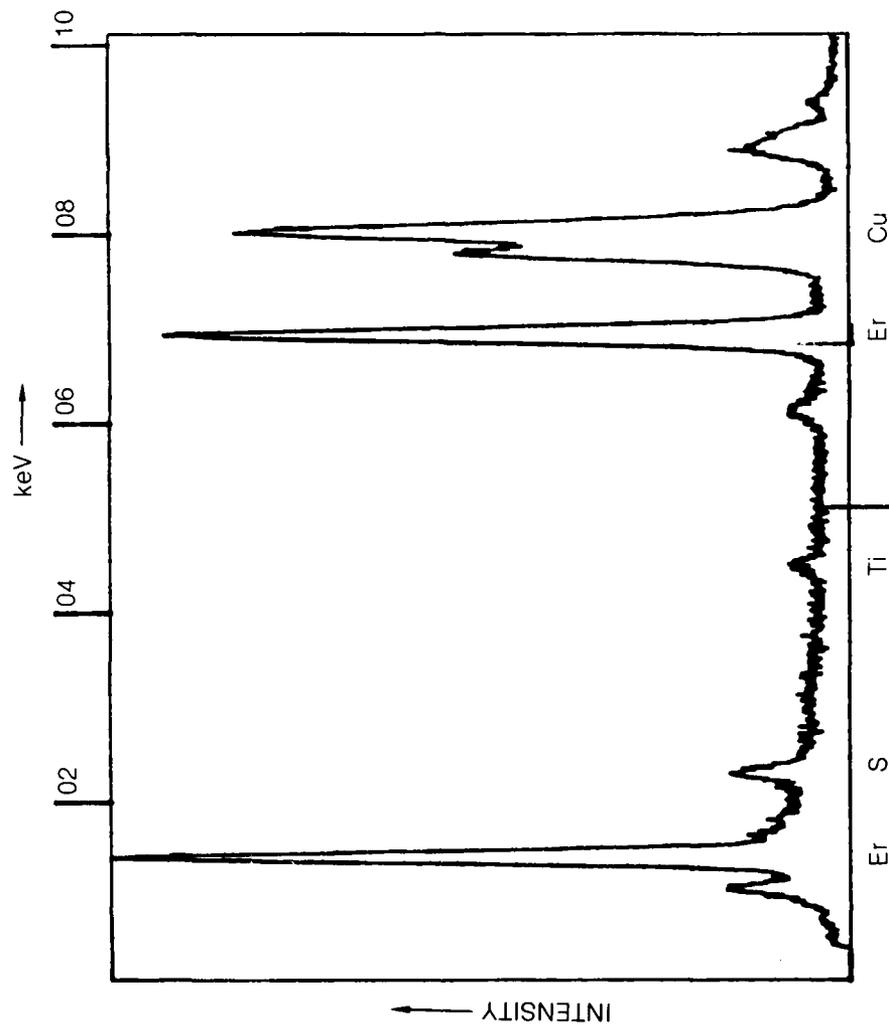
(A) CARBON EXTRACTION REPLICA. PARTICLE GIVING EDS DATA IN (B) MARKED BY ARROW

84-2-40-42

AUGER ELECTRON SPECTRA FROM SPECIMEN AS-LAYERGLAZED FROM
Ti-6Al-4V-1.5Er, 100 nm BEAM DIAMETER

Ti-6-4 + 1.5Er
SAMPLE #3
SPUTTER RATE 0.01
500 ANG.

A) α' HCP MARTENSITE
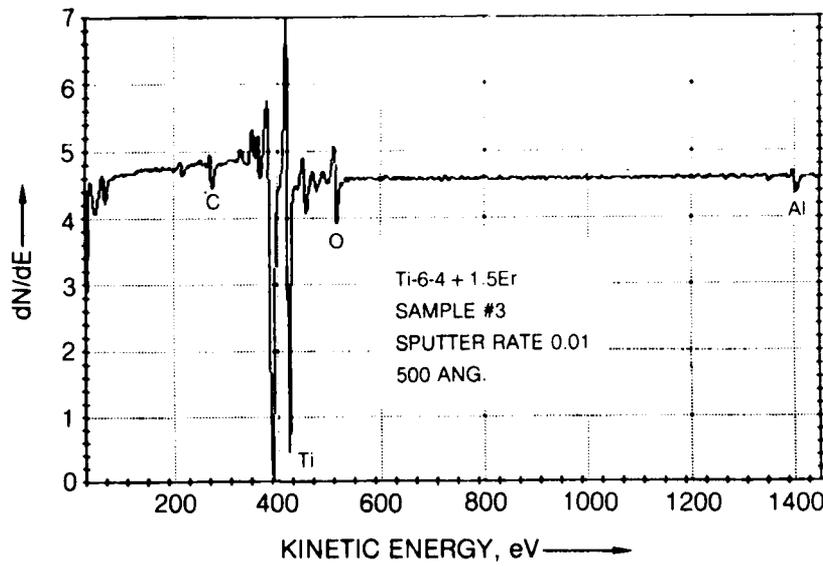
Ti64 + 1.5Er
FOIL 4
600 ANG. REMOVED
40KX (SPOT MODE)

B) INTERDENDRITIC Er_2O_3 PARTICLE

84-2-40-44

FIG. 47

AUGER ELECTRON SPECTRUM FROM CARBON EXTRACTION REPLICA, SPECIMEN AS-LAYERGLAGED FROM Ti-6Al-2Sn-4Zr-6Mo-1Er POWER.

100nm BEAM DIAMETER

Ti-6246 + 1 Er
40.000X SPOT MODE
BEAM DIA. = 0.1 N AMP

dN/dE

KINETIC ENERGY, EV

END

FILMED

DTIC