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OXIDATION OF TITANIUM ALUMINIDE AND ITS XD COMPOSITE

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Air Vehicle and Crew Systems Technology Department (Code 6063)
NAVAL AIR DEVELOPMENT CENTER
Warminster, PA 18974-5000

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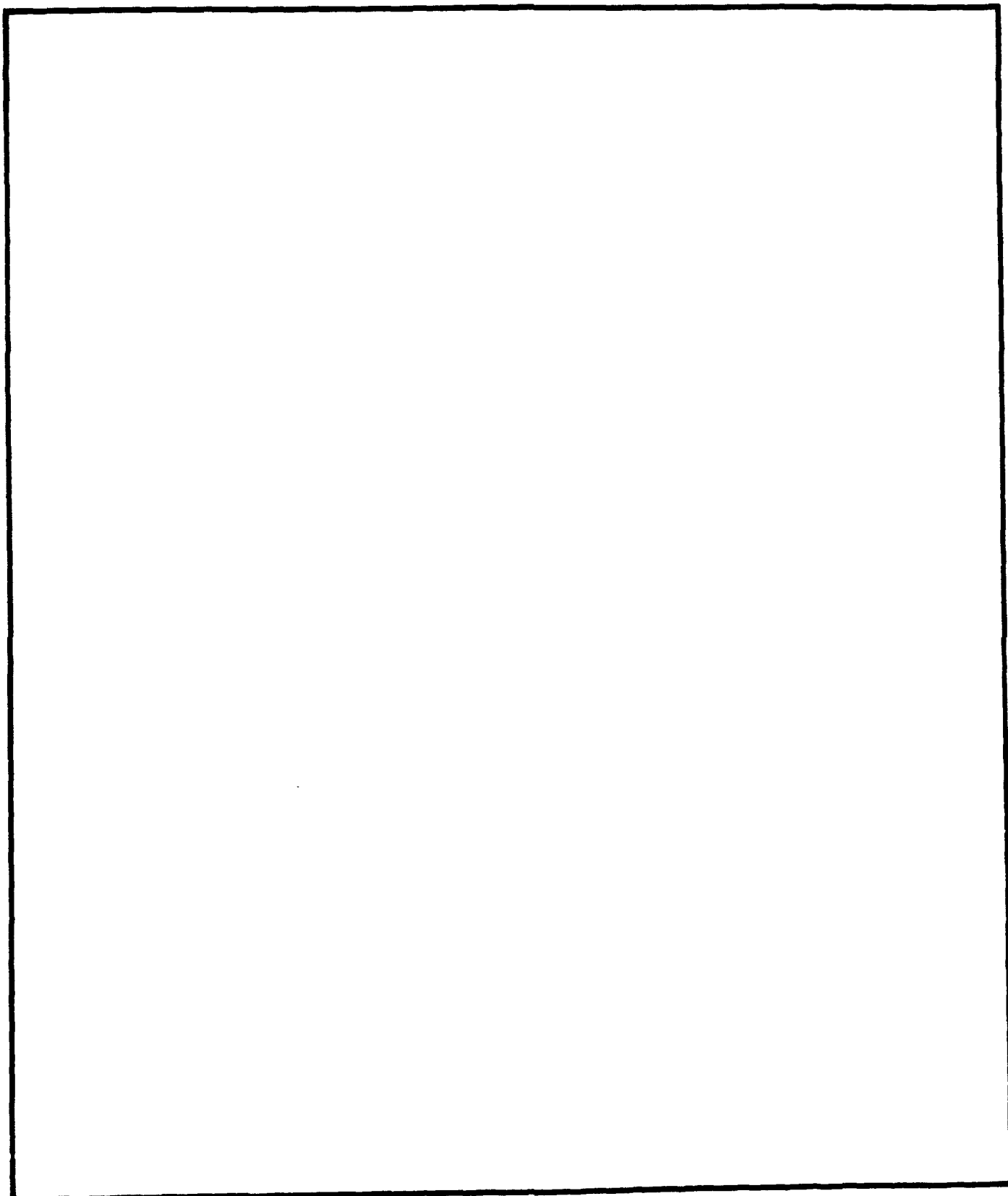
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19 ABSTRACT This study was undertaken to characterize the oxidation behavior of a Ti-45 at % Al alloy and its Exothermic Dispersion (XD) composite with TiB ₂ reinforcement particles. These materials were found to oxidize in air at temperatures ranging from 400 to 1500°C. The oxidation was evidenced by surface discoloration, formation of an oxide scale and an interfacial zone, internal oxide precipitation, internal cracking, and weight gain. The oxide scale consisted of two layers; an inner TiO ₂ and Al ₂ O ₃ mixture; and an outer TiO ₂ and Al ₂ TiO ₃ mixture. A narrow zone along the oxide scale/base metal interface was depleted in Al, but enriched with O. In the Ti-45 at % Al alloy, an Al-rich oxide, presumably Al ₂ O ₃ , was precipitated in the TiAl plate and a Ti-rich oxide, presumably TiO ₂ , was precipitated in the Ti ₃ Al plate. In the XD composite, an Al-rich oxide was precipitated along the lamellar structure colony boundary, and a Ti-rich oxide was precipitated in the interior of the colony. During oxidation, internal cracking occurred along the boundary and in the interior of the TiAl and Ti ₃ Al plates. In addition, voids were formed at the lamellar structure boundary of the XD composite. The specimen gained weight with increasing oxidation temperature. The activation energies for oxidation were determined to be 63 kcal/mol for the Ti-45 at % Al alloy, and 67 kcal/mol for the XD composite.					
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INTRODUCTION

Martin Marietta Laboratories recently developed a unique technology, whereby reinforcement dispersoids can be introduced exothermically into a metal matrix. This special alloying approach, known as Exothermic Dispersion (XD) processing, produces a metal matrix composite with a fine dispersion of a high-modulus, high-strength compound, such as TiB_2 , in titanium aluminide.

Both the matrix material, titanium aluminide, and the XD composite possess attractive properties for structural components, such as high strength-to-density ratio and high specific modulus. In addition, they retain both high strength and modulus at elevated temperatures, and have good creep resistance and non-burning characteristics. Consequently, titanium aluminides and the XD composites have potential for application at elevated temperatures, and could replace nickel and cobalt base alloys.¹ On the other hand, titanium aluminides are susceptible to oxidation.^{2,3} It was reported that double layers of oxide scale, outer TiO_2 and inner TiO_2 and Al_2O_3 mixture, were formed on the surface of $TiAl$ and Ti_3Al , and internal oxidation occurred at temperatures above 1000 degrees C in air or in oxygen^{2,4}. Similar oxidation behavior was also observed in the alloy $Ni-20Cr-2ThO_2$ ^{5,6}. In static and high-speed flowing air at 1100 and 1200 degrees C, triple layers of oxide scale (outer NiO , intermediate NiO and Cr_2O_3 mixture, and inner Cr_2O_3) were formed on the alloy surface, and a Cr-depleted zone was formed at the alloy/oxide interface.

The present study was undertaken to characterize the oxidation behavior of a mixture of two titanium aluminides, $TiAl$ and Ti_3Al , and their XD composite in air at elevated temperatures. The oxide morphology, composition change, and microstructure formed under various oxidation conditions were investigated and are presented in this report.

EXPERIMENTAL PROCEDURE

A Ti-45 at % (31.5 wt %) Al alloy and its XD composite with 7 vol % TiB_2 reinforcement were received from Martin Marietta Laboratories, Baltimore, MD. The 45 at % (31.5 wt %) is the nominal composition of Al in the matrix alloy. The actual chemical compositions of these materials were determined to be 66.8 wt % Ti and 32.4 wt % Al, and 70.1 wt % Ti, 26.2 wt % Al, and 3.1 wt % B, respectively.

The specimen materials were cut into 7 mm cubes, polished to 320 grit finish, cleaned, and weighed. Subsequently, they were heated in static air at temperatures ranging from 400 to 1500 degrees C for periods of 4 to 40 hours, cooled to room temperature, and weighed again.

Polished specimens were etched with Keller's reagent, and the microstructure was examined in an optical microscope and in an AMR 1000 scanning electron microscope, operated at an accelerating voltage of 20 kv. The chemical composition of any particular location in a specimen was determined using a KEVEX energy dispersive X-ray spectrometer and a JEOL Superprobe 733 electron microprobe analyzer, operated at an accelerating voltage of 10 kv. The surface oxide phases were identified using standard X-ray diffraction techniques, employing a copper tube and a theta-theta wide angle goniometer.

RESULTS AND DISCUSSION

The oxidation treatment resulted in surface oxidation, formation of an interfacial zone, internal oxide precipitation, internal cracking, and weight gain. These results and the specimen microstructures are presented and discussed respectively.

MICROSTRUCTURE

The micrograph of the as-received (unoxidized) matrix material, Ti-45 at % Al alloy, shows colonies of lamellar structure, consisting of alternate light and gray plates of Ti_3Al and $TiAl$, Figure 1(a). The measured colony size of the lamellar structure in the matrix is 0.024 mm. The colony size of the Ti-45 at % Al alloy is about 27 times greater than that of the XD composite. (In this report, the (Ti-45 at % Al) + 7 vol % TiB_2 XD composite is referred to as "XD composite".) The microstructural change due to oxidation in both materials will be discussed later.

SURFACE OXIDATION

The specimen surface was discolored to blue at and below 500 degrees C. The surface then was covered by blue and gray oxide scales at and above 600 degrees C and by double layers of oxide scale at and above 1000 degrees C. The outer layer oxide scale was porous and grainy, became thicker with increasing oxidation temperature and time, and tended to flake-off or spall. Its color was white in the specimens of Ti-45 at % Al alloy, and yellow in those of the XD composite. Specimen cross-sections containing the base metal and the thickened oxide scale, and SEM micrographs of the outer layer oxide scale are shown in Figure 2. The X-ray diffraction analysis of the double oxide layers shows that the inner scale is a mixture of TiO_2 and Al_2O_3 , and the outer scale is a mixture of TiO_2 and Al_2TiO_5 . The schematics of the specimen cross-section before and after oxidation are shown in Figure 3. This result agrees partially with the observations of Choudhury (2) and Mendiratta (3). They detected only TiO_2 in the outer scale, and a mixture of TiO_2 and Al_2O_3 in the inner scale on the surfaces of $TiAl$ and Ti_3Al .

INTERFACIAL ZONE

Using optical and scanning electron microscopy, a narrow zone was observed along the base metal/oxide scale interface in a polished and etched specimen cross-section, Figure 4. This zone was slightly darker than the base metal in the Ti-45 at % Al alloy, and lighter than the base metal in the XD composite. Such a zone, about 5 μm wide, was detected in the specimen oxidized at 1000 degrees C in air for four hours. Analyses with an energy dispersive X-ray spectrometer and an electron microprobe analyzer indicated that this zone was depleted in Al and enriched with O. The analysis results are given in Table 1. This is attributed to outward diffusion of Al and inward diffusion of O during the oxidation process. A similar alloying element (Cr) depletion in the interfacial zone was noticed in an oxidized Ni-20Cr-2ThO₂ alloy by Tenney et al⁵.

INTERNAL OXIDE PRECIPITATION

During oxidation at 1100 degrees C or higher, oxides were precipitated in the interfacial zone, Figures 5 and 6. In the Ti-45 at % Al alloy, the oxides appear as dark plates and fingerprint-like structures of dark and light phases, Figure 5(a). Many of the dark oxide plates are nearly parallel and extend inward from the base metal surface. The fingerprint-like structures are located along the base

metal surface between the dark oxide plates. In the XD composite, the oxides appear as dark particles, and fingerprint-like structures of dark and light plates, Figure 6. The dark oxide particles are similar in appearance to grain boundary precipitates, and they are precipitated inward from the base metal surface. The fingerprint-like structures are located along the base metal surface between the strings of the dark oxide particles. Etching attempts failed to reveal the lamellar structures of TiAl and Ti₃Al plates and their colony boundaries in the oxidized XD composite specimens. However, considering the initial unoxidized specimen microstructure, Figure 1, and the oxide particle distribution feature, it is likely that the oxide particles are precipitated along the colony boundaries.

The results of electron microprobe analyses, Table 2, indicate that the dark oxide plates and particles are enriched with Al and O, but depleted in Ti. This indication is confirmed by the X-ray mappings of Al, Ti, B, and O, as shown in Figures 7 through 9. On the other hand, the fingerprint-like structures contain high concentrations of Ti and O, but little or no Al. The dark phase's O-concentration is greater than the light phases's. The light area between the dark oxide plates has nearly equal amounts of Al and Ti, but a large amount of O in the Ti-45 at % Al alloy. In the XD composite, the light area between the dark oxide particles has a slightly greater amount of Al than Ti, but a large amount of O. From this result, it is evident that the dark oxide plates and particles are an Al-rich oxide, presumably Al₂O₃, and the dark phase of the fingerprint-like structure a Ti-rich oxide, presumably TiO₂.

The base metal microstructure of the Ti-45 at % Al alloy consists of alternating plates of TiAl and Ti₃Al, Figure 1. The lower density phase, TiAl, has a gray appearance and the higher density phase, Ti₃Al, has a light appearance in the backscattered electron image. The dark oxide plates in the Ti-45 at % Al alloy are connected to the gray plates in the interior of the base metal, as shown in an optical micrograph and a backscattered electron image, Figures 5(a) and (b). Furthermore, the X-ray mapping of O delineates the interconnected Al-rich oxide plates and gray plates of TiAl with O-reflections of high and low intensities, respectively, Figure 5(c). Therefore, it is evident that the Al-rich oxide is nucleated and grown in the gray plate of TiAl, and the Ti-rich oxide in the light plate of Ti₃Al. In addition, the oxide nucleation and growth in these aluminide plates must be due to the large colony and plate sizes in the Ti-45 at % Al alloy.

An important observation with the XD composite is the comparatively greater oxidation resistance of the reinforcement particle, TiB₂. The X-ray mappings of B and O indicate less O-concentration or less oxidation of the reinforcement particle TiB₂ than the surrounding matrix, Figures 9(c) and (d).

INTERNAL CRACKING

Cracks of various sizes were found inside the specimens of the Ti-45 at % Al alloy and the XD composite after oxidation at or above 1000 degrees C, Figure 10. The cracking is more extensive closer to the specimen surface, and its sites are the plate boundary and interior of TiAl and Ti₃Al. The cracks in the plates are parallel or nearly perpendicular to the plate boundary. Some voids are also seen along the colony boundaries of the lamellar structure in the XD composite specimens. The observed internal cracking and void formation must be associated with the inward diffusion of O, the outward diffusion of Al, and the resultant generation of vacancy and internal stress during the oxidation process.

WEIGHT GAIN

The specimens of the Ti-45 at % Al alloy and XD composite gained weight with oxidation time at a given temperature, following a parabolic rate law. A representative plot of weight gain versus oxidation time is shown in Figure 11. Moreover, the weight gain was faster with increasing oxidation temperature. This is illustrated by the plots of the square of the weight gain per unit initial surface area in a unit time ($\Delta W^2/t$) versus the reciprocal temperature of oxidation $1/T$, Figure 12. The activation energies for oxidation were determined to be 63 kcal/mol for the Ti-45 at % Al alloy and 67 kcal/mol for the XD

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composite from the slopes of the above plots and the following equation:

$$(\Delta W)^2 = b \cdot t \cdot \exp(-Q/RT)$$

where ΔW is the weight gain per unit initial surface area, b a constant, t the oxidation time, Q the activation energy, R the gas constant, and T the oxidation temperature in °K. Presumably, the activation energies correspond to the outward diffusion of Al or inward diffusion of O in respective specimen materials.

CONCLUSIONS

1. A Ti-45 at % Al alloy and its XD composite with TiB₂ reinforcement particles are highly susceptible to oxidation in air at temperatures ranging from 400 to 1500 degrees C.
2. The oxidation is evidenced by surface discoloration, formation of oxide scale and interfacial zone, internal oxide precipitation, internal cracking, and weight gain.
3. The oxide scale consists of two layers. The inner layer is a mixture of TiO₂ and Al₂O₃, and the outer one a mixture of TiO₂ and Al₂TiO₅.
4. The internal oxides are Al₂O₃ and TiO₂.
5. The internal cracking and void formation are attributable to inward diffusion of O and outward diffusion of Al, and the resultant generation of vacancy and internal stress during oxidation.
6. The activation energies for oxidation are 63 kcal/mol for the Ti-45 at % Al alloy and 67 kcal/mol for the XD composite.

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Table 1.

Composition of Base Metal, Interfacial Zone, and Oxide Scale (The Specimens were Oxidized at 1000°C in Static Air for Four Hours and Cooled to Room Temperature in Static Air.)

I. Aluminum Concentration, Indicated by X-ray Intensity Ratio, $\frac{(A1 K_{\alpha})}{(A1 K_{\alpha}) + (T1 K_{\alpha})}$

<u>Specimen</u>	<u>Inside of Base Metal</u>	<u>Interfacial Zone</u>	<u>Oxide Scale</u>
Ti-45 at % A1 Alloy	0.14	0.10	0.20
XD Composite	0.11	0.08	0.15

(The X-ray intensity was measured by an energy dispersive X-ray spectrometer, and no correction was made.)

II. Oxygen Concentration (at %)

<u>Specimen</u>	<u>Inside of Base Metal</u>	<u>Interfacial Zone</u>	<u>Oxide Scale</u>
Ti-45 at % A1 Alloy	5.22	20.56	78.06
XD Composite	5.07	20.57	66.76

(The oxygen concentration was determined by an electron microprobe analyzer, and all necessary corrections were made.)

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Table 2.

Composition of Oxides and their Vicinities in Interfacial Zone.

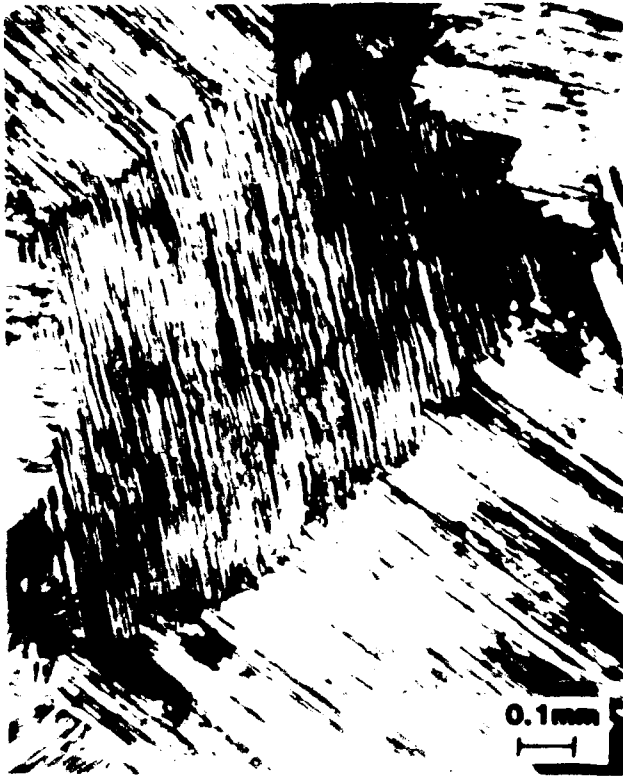
I. Composition of Oxides in Ti-45 at % Al Alloy (at %)

	<u>Al</u>	<u>Ti</u>	<u>O</u>
Dark Plate	31.27	3.60	65.14
Light Area between Dark Plates	20.98	22.13	56.83
Fingerprint-Like Structure*			
Dark Phase	0	24.63	75.37
Light Phase	0	29.92	70.08

II. Composition of Oxides in XD Composite (at %)

	<u>Al</u>	<u>Ti</u>	<u>O</u>
Dark Particle	40.53	0.69	57.53
Light Area between Dark Particles	25.97	19.49	54.31
Fingerprint-Like Structure*			
Dark Phase	5.46	22.16	71.85
Light Phase	0	40.90	56.72

* Since the fingerprint-like structure is so fine, the determined compositions of the dark and light phases cannot be accurate.



(a) Ti-45 at % Al Alloy



(b) (Ti-45% at % Al) + 7 vol % Ti P + XD Composite

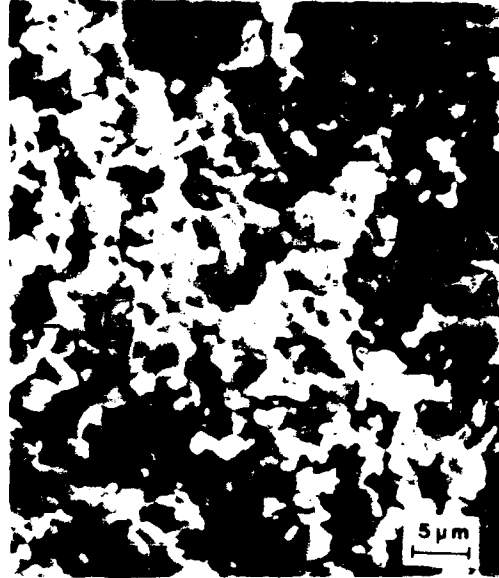
Figure 1. Micrographs of Specimen Materials

base metal

oxide scale



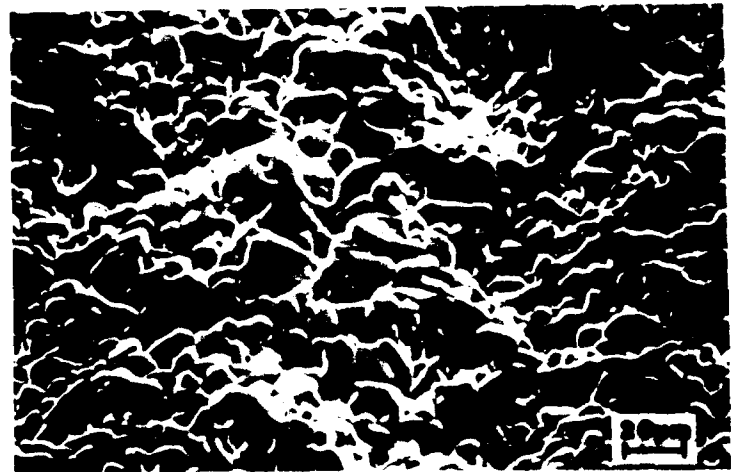
(a) Specimen Cross Section of Ti-45 at 3% Al Alloy.



(b) SEM Micrograph of Outer Layer Oxide Scale of Ti-45 at 3% Al Alloy



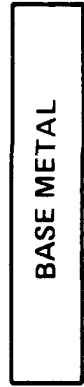
(c) Specimen Cross Section of XD Composite



(d) SEM Micrograph of Outer Layer Oxide Scale of XD Composite

(a) and (c) Specimen (Cross-Sections, Showing Base Metal and Oxide Scale),
(b) and (d) SEM Micrographs of Outer Layer Oxide Scale

BEFORE OXIDATION



BASE METAL

AFTER OXIDATION

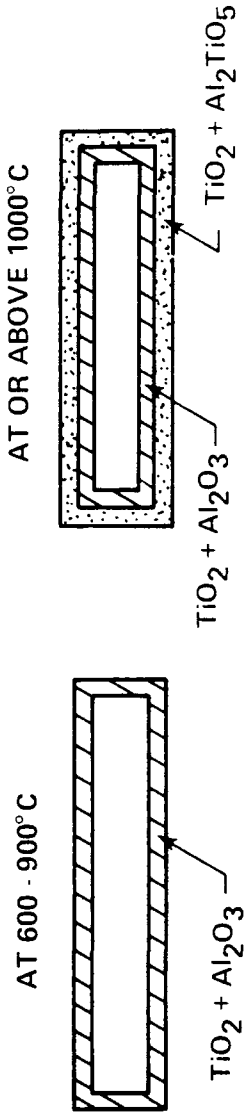
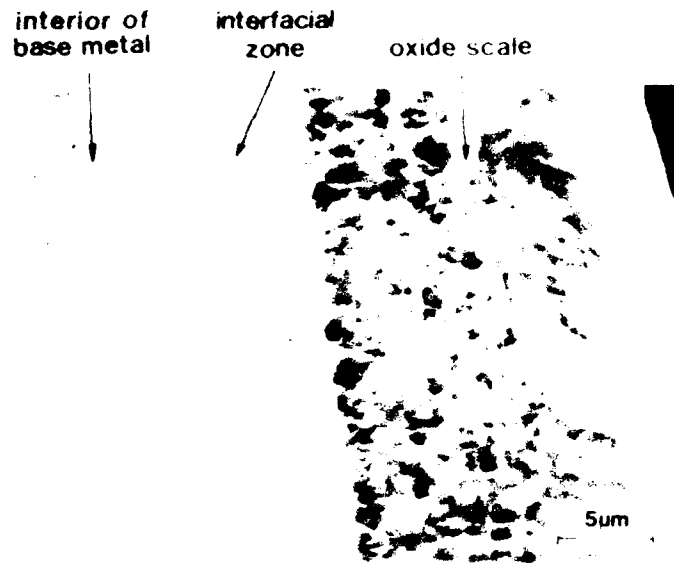


Figure 3. Schematic of Specimen (Cross-Section before and after Oxidation).

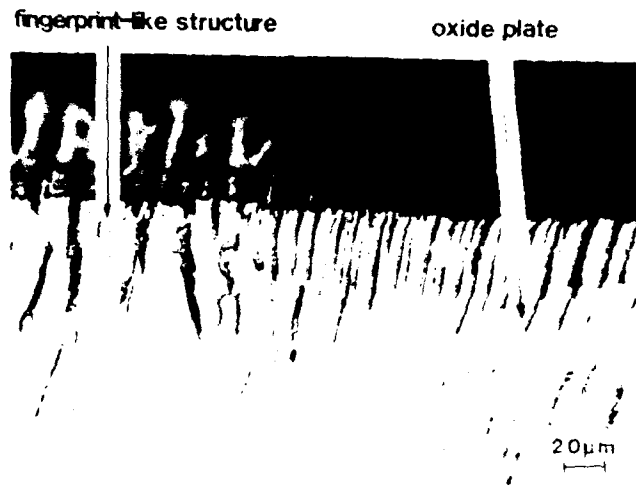


(a) Ti-45 at % Al Alloy.

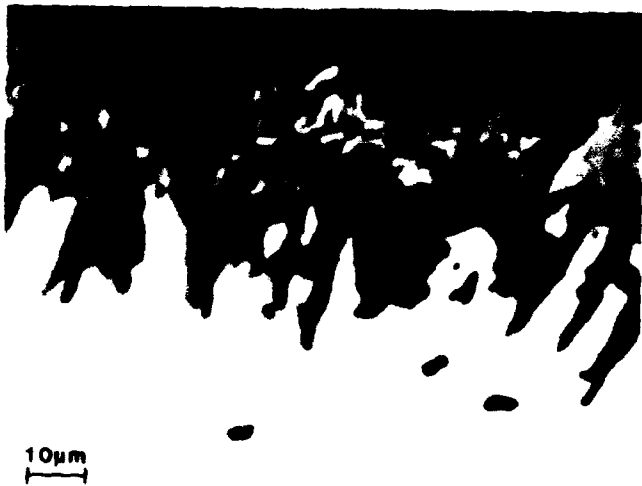


(b) XD Composite.

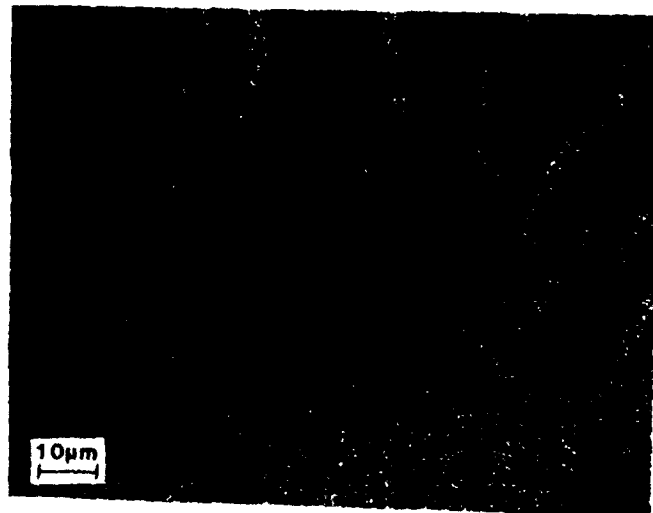
Figure 4. Interfacial Zone, Oxide Scale, and Base Metal.



(a) Optical Micrograph.



(b) Backscattered Electron Image.



(c) X-ray Mapping of O.

Figure 5. Oxides in Interfacial Zone of Ti-45 at % Al Alloy.

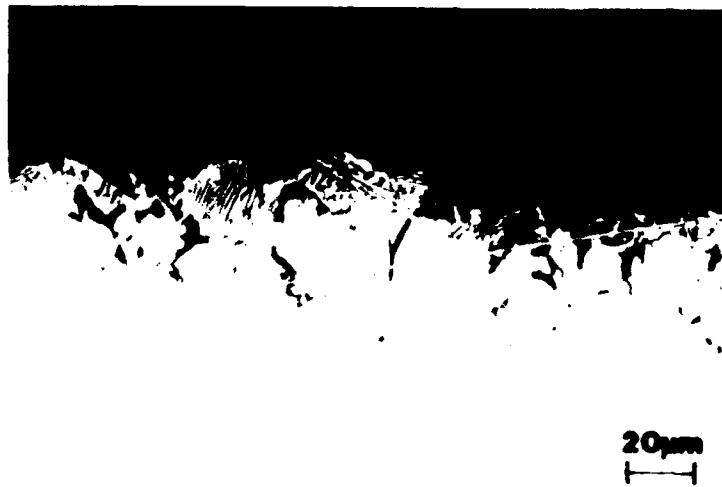
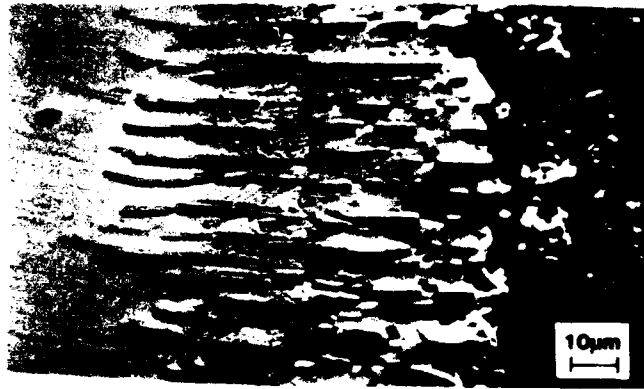
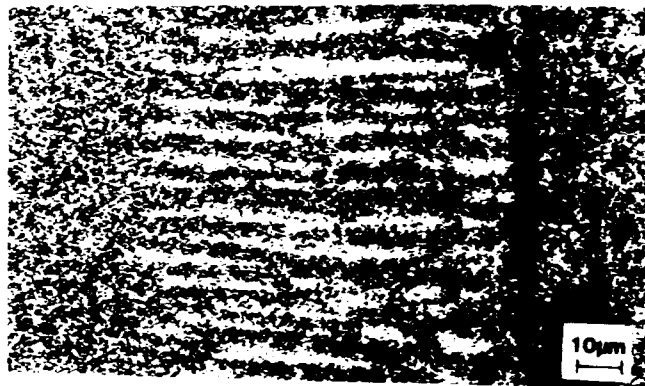


Figure 6. Oxides in Interfacial Zone of XD Composite.

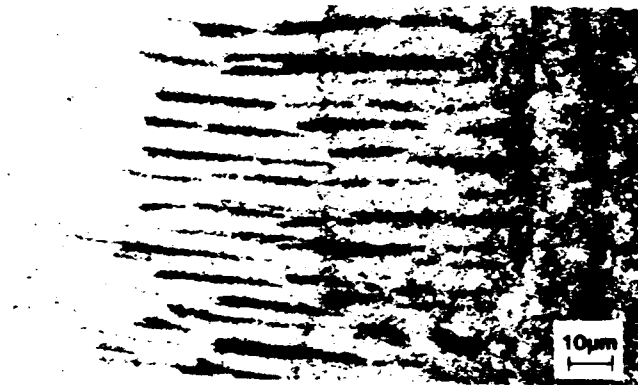
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(a) SEM Micrograph.

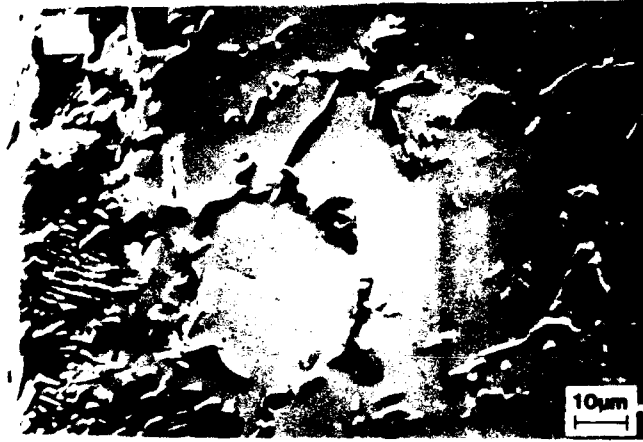


(b) X-ray Mapping of Al.

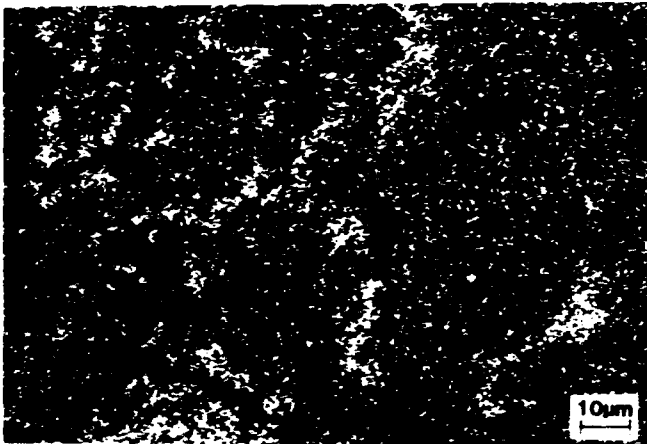


(c) X-ray Mapping of Ti.

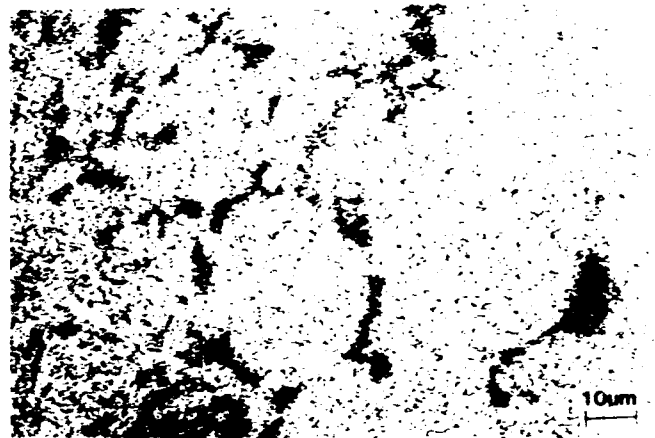
Figure 7. Oxides and X-ray Mapping of Al and Ti in Ti-45 at % Al Alloy.



(a) SEM Micrograph.



(b) X-ray Mapping of Al.



(c) X-ray Mapping of Ti.

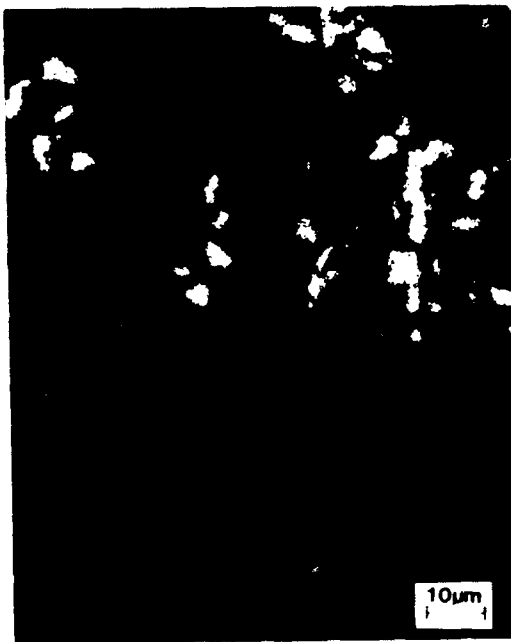
Figure 8. Oxides and X-ray Mapping of Al and Ti in XD Composite.



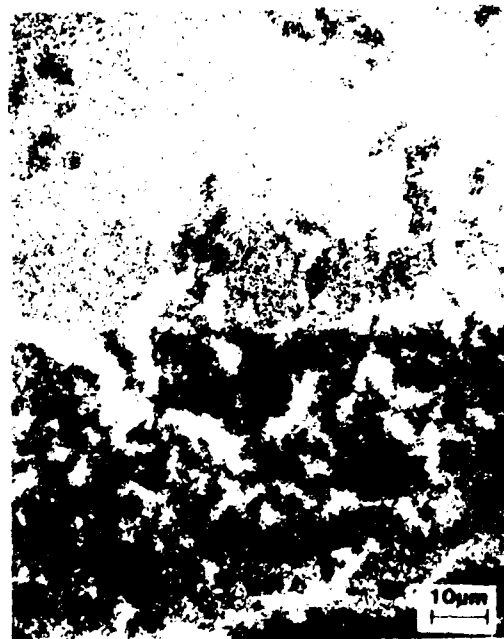
(a) Backscattered Electron Image.



(b) X-ray Mapping of Ti



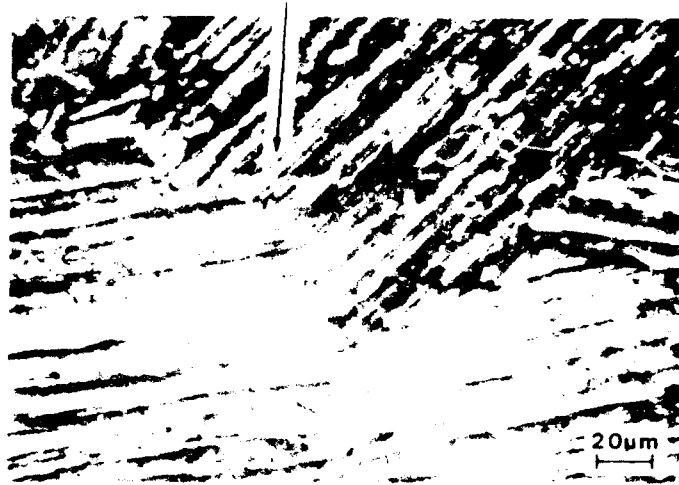
(c) X-ray Mapping of B.



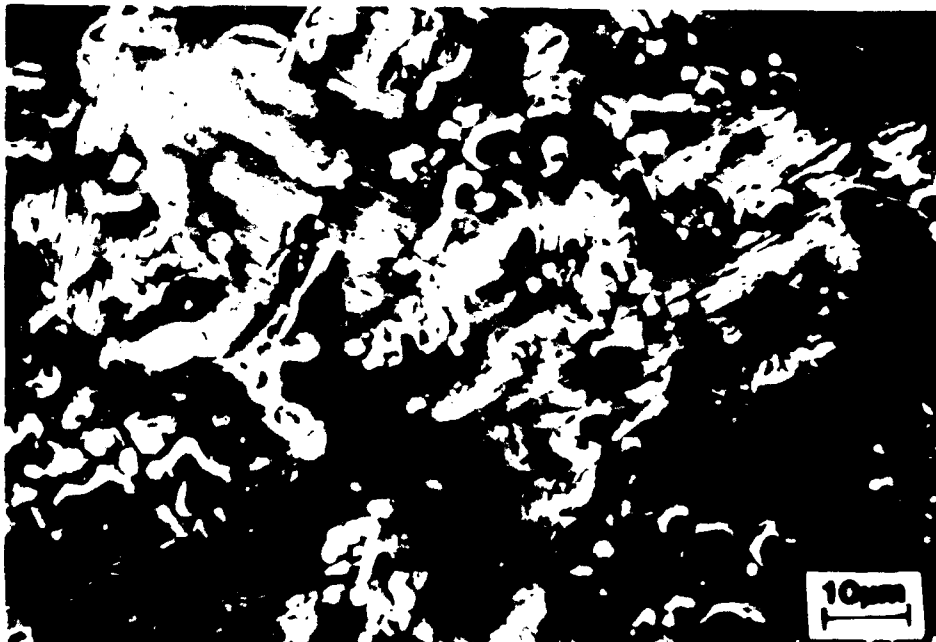
(d) X-ray Mapping of O.

Figure 9 Oxides and X-ray Mapping of Ti, B, and O in XD Composite.

internal crack



(a) Ti - 45 at % Al Alloy.



(b) XD Composite.

Figure 10. Internal Cracking.

$$\frac{(\Delta W)^2}{t} = b.t.e^{-Q/RT}$$

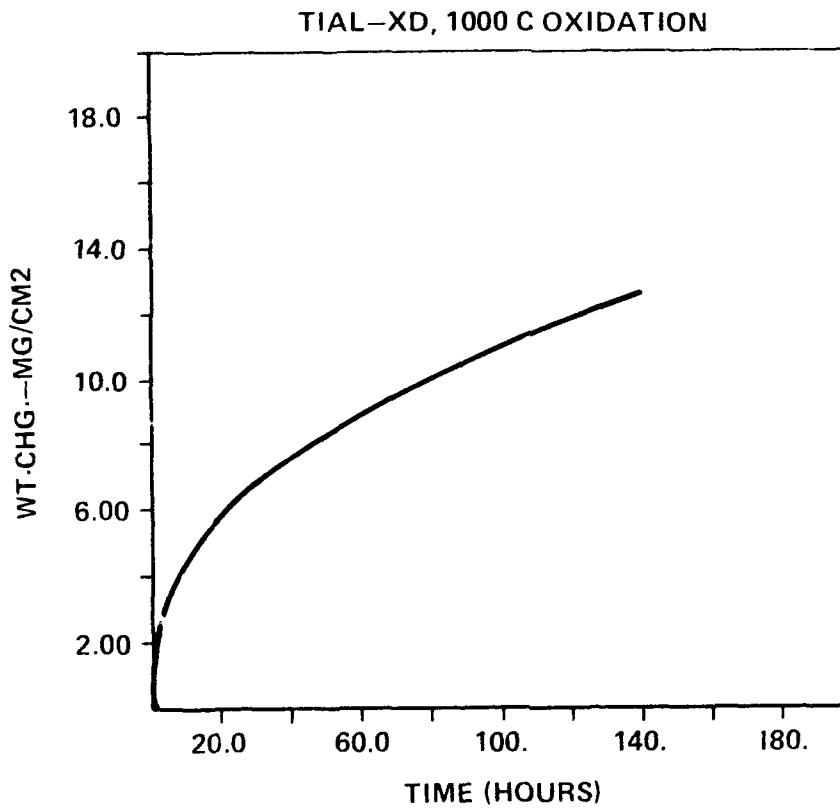


Figure 11. Weight Gain with Oxidation Time

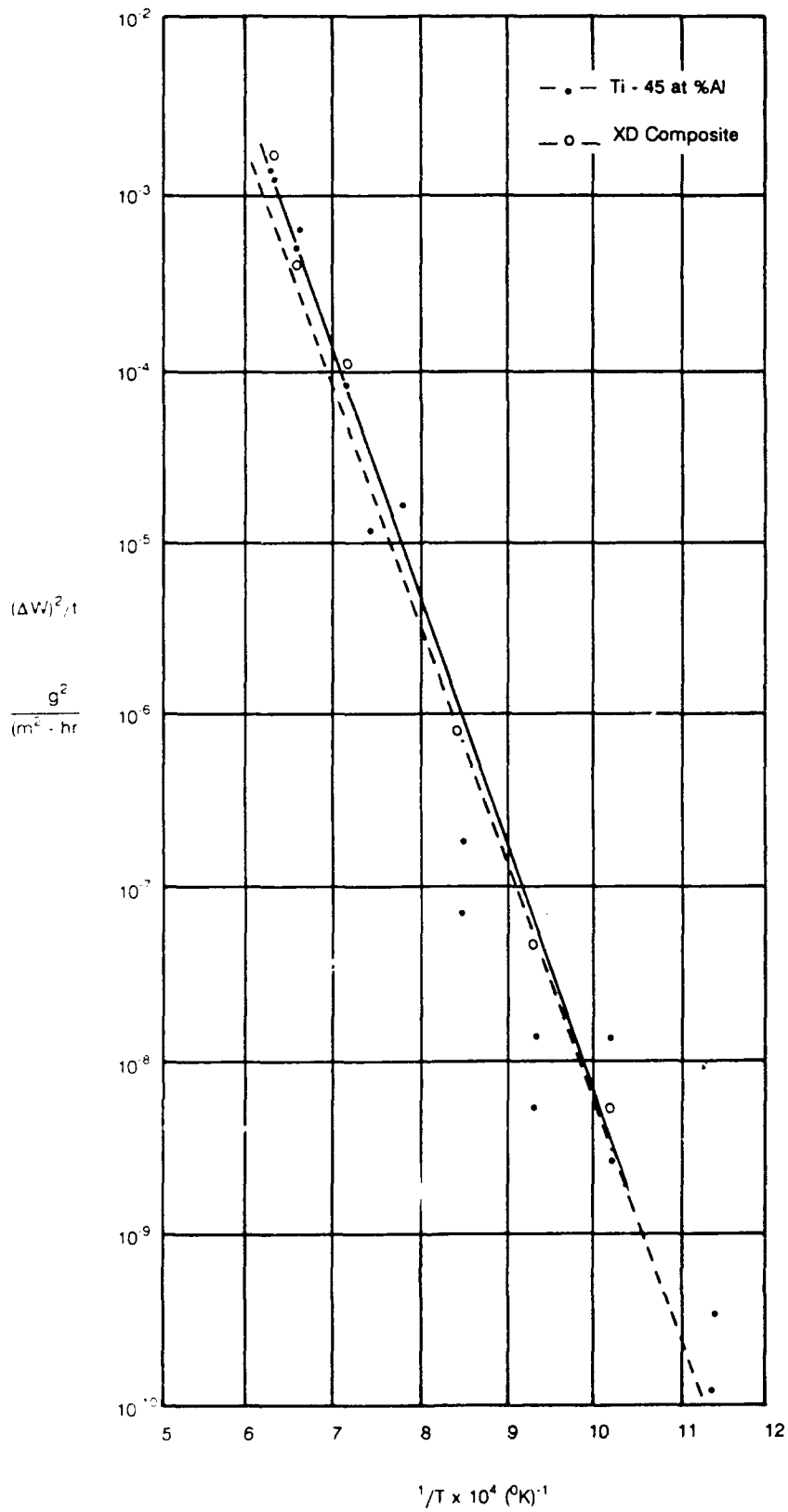


Figure 12. Weight Gain versus Reciprocal Temperature of Oxidation.