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The Development of a Ti-6Al-4V Alloy via Oxygen Solid Solution Strengthening for Aerospace and Defense Applications

by Tyrone L. Jones, Katsuyoshi Kondoh, Takanori Mimoto, Nozomi Nakanishi, and Junko Umeda

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14. ABSTRACT

The high cost of titanium has historically prevented widespread use in military ground vehicles. Two strategies to make this material more cost effective and viable are to reduce the cost of titanium armors or to improve the ballistic performance of titanium and reduce the amount of material required. This report investigates the latter strategy. Mixtures of titanium powders and TiO_2 particles were employed as starting materials and consolidated by spark-plasma sintering and hot extrusion. The content of TiO_2 particles was 0 to ~1.5% of the mass mixture. Solidification of oxygen atoms (from TiO_2 particles) into Ti matrix occurred at 1073 K for 1800 s in a vacuum. Tensile testing showed that tensile strength (TS) and yield strength (YS) increased in proportion to TiO_2 content but elongation decreased slightly with increased TiO_2 content. Extruded pure Ti powder material with 1.5% TiO_2 particles produced 1040 MPa TS, 902 MPa YS, and 25.1% elongation when tested. When using Ti-6Al-4V (Ti-64) alloy powders with 0.5% TiO_2 particles, the final extruded Ti-64 powder bars with oxygen solid solution showed 1226 MPa TS and 22.7% elongation. Initial ballistic evaluation showed the Ti-64 powder bars with 0.5% TiO_2 particles yielded a marked improvement over the conventionally rolled Ti-64 alloy plate.

15. SUBJECT TERMS

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1. Introduction

Titanium alloys are widely used in various aerospace, marine, chemical industries, medical, and military applications (*1, 2*) because these alloys have high specific strength, high Young's modulus, and excellent corrosion resistance. In particular, when a lot of organic composite materials such as carbon fiber-reinforced plastics (CFRP) are applied to aircraft components to decrease the weight reduction (*3*), titanium materials are also used because their thermal coefficient of expansion is similar to that of CFRP and titanium is not subject to damage by galvanic corrosion in contact with CFRP materials. In general, the conventional titanium alloys used in structural components contain many alloying elements of transition metals such as vanadium (V), niobium (Nb), chromium (Cr), etc. While these elements are effective in improving mechanical properties of titanium alloys, they have poor machinability, as well as decrease the alloy's ductility, and increase the alloy's cost. It is the combination of ductility and cost that challenges the employment of titanium alloys in a wider range of military applications. Powder metallurgy (P/M) process was used to fabricate titanium alloys in this study. Commercially pure (CP) Ti powders were used as the starting raw materials. From an economical standpoint, commercially pure CP Ti powders are lower cost compared to Ti alloy powders. Oxygen solid solution strengthening was mainly applied to P/M pure titanium materials by using TiO_2 additives. In the evaluation of tensile properties of the wrought pure titanium materials consolidated by sintering and hot extrusion process, a theoretical approach using the Labusch model (*4*) was carried out to understand the strengthening increment of their yield stress by oxygen atoms in solid solution. Texture analysis was also applied to clarify high ductility behavior of P/M extruded titanium materials with oxygen.

1.1 Materials Science - Experimental Preparation of Materials and Fundamental Evaluation

The elemental mixture of pure titanium (Ti) powders and TiO_2 particles with a mean particle size of 1.66 μm were employed as starting materials. A mean particle size and purity of the commercially pure Ti powders produced by hydride-dehydride (HDH) process (*5*) was 23.7 μm and 99.5%, respectively. The content of TiO_2 particles was 0 to ~1.5 mass% of the mixture. Next, the aluminum and vanadium powders were added to the mixture. The mixture powders were mechanically mixed by conventional ball milling using 10-mm-diameter ZrO_2 balls. They were consolidated in a carbon container installed in the spark-plasma sintering (SPS) equipment under vacuum condition (<6 Pa) at a temperature of 1073 K and pressure of 30 MPa for 1.8 ks. A solid solution of oxygen atoms originating from the TiO_2 particles flowed into the Ti matrix during the SPS process. After preheating the sintered Ti powder billets at 1273 K for 180 s in an argon gas atmosphere, they were immediately consolidated by hot extrusion. The extrusion ratios of 6.1, 18.5, and 37.7 were used to control the orientation texture by plastic deformation of

P/M extruded Ti materials. Optical microscope observation, Scanning Electron Microscope and Energy Dispersive X-Ray Spectrometer (SEM-EDS/EBSP) and (XRD) analysis were employed to investigate microstructures and textures of Ti materials containing oxygen atoms. Micro-hardness and tensile test at ambient temperature were carried out to evaluate the mechanical properties. Scale-up extruded materials used for ballistic evaluation were produced by the same method as the small-size specimens prepared in the laboratory. The SPSed billet dimensions were 60-mm diameter and 72-mm length, as shown in figure 1a. The plate extruded bar shown in figure 1 has a width of 42 mm and thickness of 10 mm.

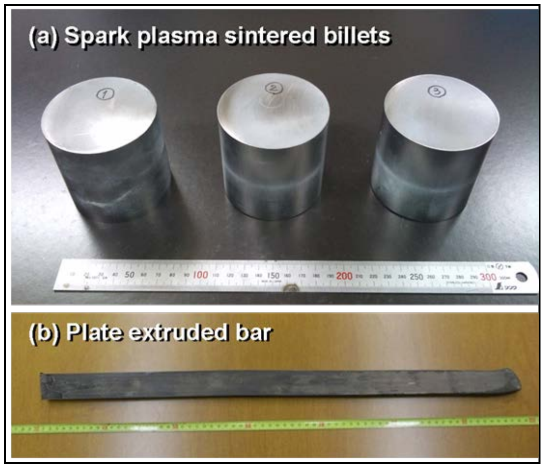

Figure 1. SPSed billets and extruded bar using $Ti64\text{-}TiO_2$ mixture powders for ballistic evaluation test.

1.2 Ballistic Science - Terminal Ballistic Methodology

Ballistic testing of the extruded, nominal 10-mm Ti-6Al-4V titanium composite bars was performed by the U.S. Army Research Laboratory (ARL) in accordance with MIL-STD-662F (*6*). Ballistic results were characterized using the standard V50 test methodology, also documented in MIL-STD-662F. The 5.56-mm ball M193 projectile (figure 2) was selected for initial ballistic evaluations of the 10-mm-thick titanium alloys bars based on previous research on thin titanium alloy plates (*7*). The 5.56-mm ball M193 projectile has a total weight of 3.56 and a lead core weight of 2.53g.

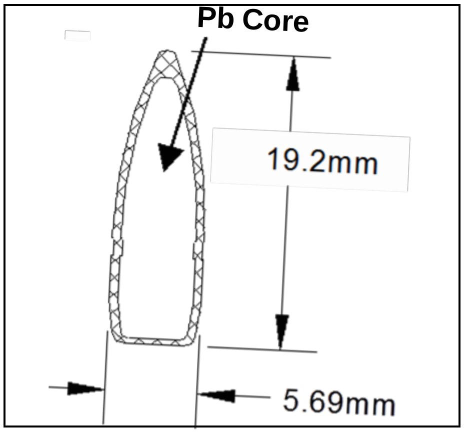

Figure 2. Illustration of 5.56-mm ball M193 projectile.

2. Results and Discussion

2.1 Microstructural Analysis

XRD profiles of Ti-1.0% TiO_2 sintered materials indicates the Ti main diffraction peak obviously shifted to a lower diffraction angle and no TiO_2 peak was detected after applying a sintering temperature over 973 K in the SPS process. Ti peaks gradually shifted to a lower and lower diffraction angle with the increase in TiO_2 content. According to the XRD profiles, the lattice constant in a-axis and c-axis was calculated by using Bragg's law (*8*). Figure 3 shows a change in each lattice constant of extruded pure Ti powder materials with various TiO_2 additives. With increase in TiO_2 content of the sintered Ti materials, the lattice in the c-axis increased proportionally to the TiO_2 content while that in the a-axis revealed a very small change. It suggests that an oxygen atom is soluted between Ti atoms as illustrated in figure 3c and the lattice spreads out in the c-axis direction.

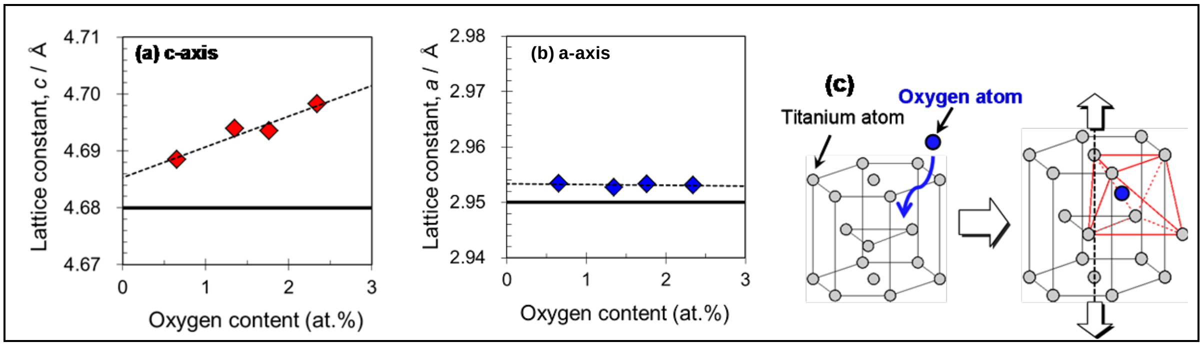

Figure 3. Dependence of lattice constant in c-axis (a) and a-axis (b) on oxygen content of extruded pure Ti powder materials with various TiO_2 particles; (c) oxygen atom is soluted between Ti atoms and the lattice spreads out in the c-axis direction.

Figure 4 shows optical microstructures of extruded pure Ti powder materials with various TiO_2 particles. The Ti matrix of all specimens consisted of equiaxed grains with no segregation of TiO_2 in the matrix. High-magnification observation by SEM shown in figure 5a–c indicates needle-like compounds are dispersed in the grains regardless of the additive TiO_2 content. On the other hand, the extruded pure Ti material using Japanese Industrial Standards grade 4 shows no needle-like dispersoid in the matrix as shown in figure 5d. Chemical composition analysis and electron probe micro-analyzer (EPMA) on the dispersoids suggest they correspond to TiH_2 compounds originated from pure Ti raw powders produced by the HDH process. Concerning oxygen dispersion in the matrix, the oxygen solubility in Ti at the ambient temperature is about 33% and all oxygen atoms originated from TiO_2 additives could be soluted in the Ti matrix. EPMA results shown in figure 6 detected the uniform dispersion of oxygen atoms in the matrix of both Ti-0% TiO_2 and Ti-1.5% TiO_2 extruded powder materials.

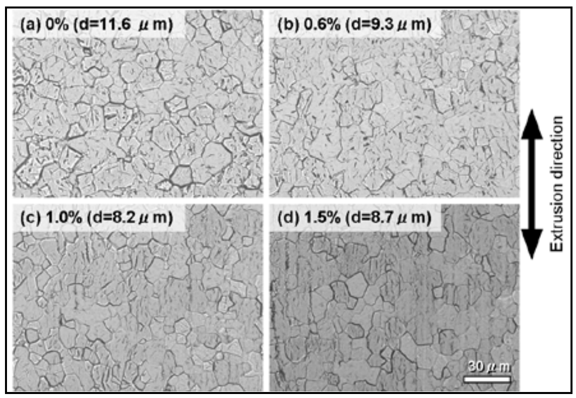

Figure 4. Optical microstructures of extruded pure Ti powder materials with various TiO_2 particles.

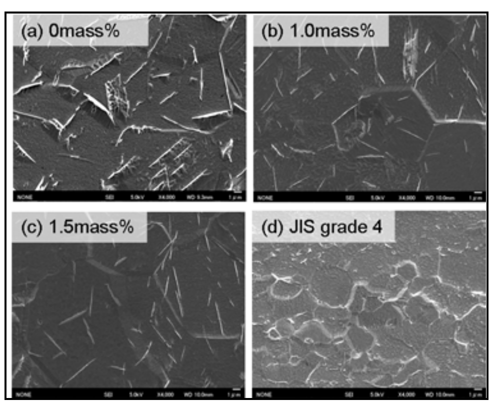

Figure 5. SEM observation on extruded Ti-TiO_2 powder materials a–c and extruded Ti using JIS grade 4 cast ingot (d).

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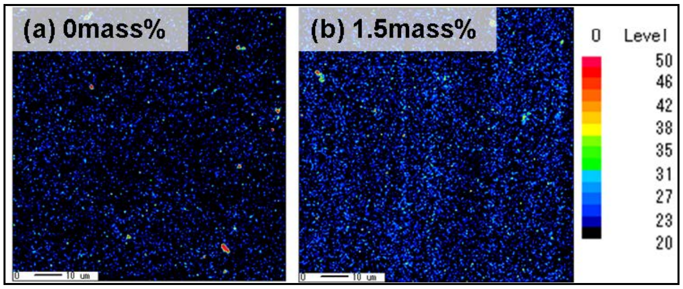


Figure 6. Oxygen mapping by EMPA: extruded pure Ti (a) and Ti-1.5%TiO_2 materials (b).

2.2 Mechanical Properties

Micro-hardness measurements by nano indenter at the grain boundary and inside grains, shown in figure 7, reveal a small difference within the scatter between two measurement points. These results and figure 3 suggest that oxygen atoms are uniformly soluted in the Ti matrix of the extruded Ti-TiO_2 powder materials via SPS process. Figure 8 shows a dependence of tensile properties on the oxygen content of wrought Ti materials. Both the UTS and YS proportionally increase with an increase in the oxygen content. As a noteworthy behavior, they show a very slight decrease of elongation while the YS of powder Ti material with 2.48 at.% O is twice that of Ti material with no TiO_2 additive (0.68 at.% O). The increase of YS is mainly due to oxygen solid solution and grain refinement effects (no effect of nitrogen and carbon solid solution because of no remarkable change of their contents of Ti materials). The latter effect on the YS increase can be estimated by Hall-Petch equation (*9, 10*) with a material constant, k value of 18 $MPa/mm^{0.5}$. The summarized YS increase is shown in table 1 and means the main strengthening factor is due to oxygen solid solution of extruded Ti powder materials. To evaluate the strengthening behavior quantitatively, the theoretical approach to calculate the YS increment with oxygen solid solution was carried out by using the Labusch model (*4*).

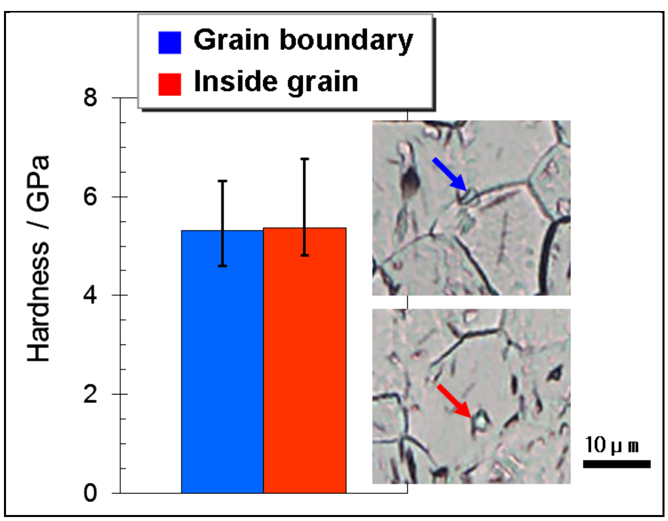


Figure 7. Micro-hardness at Ti grain boundary and inside grain measured by nano indenter.

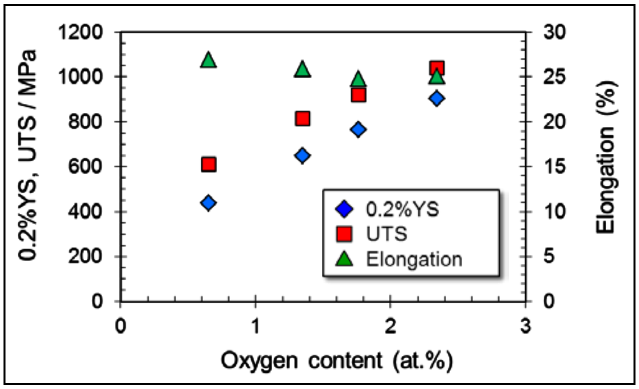


Figure 8. Dependence of tensile properties on oxygen content of extruded Ti-TiO_2 powder materials.

Table 1. YS increase by grain refinement and oxygen solid solution strengthening effect of extruded Ti materials.

Ti_2 Content (mass %)	Grain Size (per μm)	0.2% YS Increment by Grain Refinement (per MPa)	0.2% YS (revised)
0	11.6	0	437.7
0.6	9.3	19.9	627.4
1.0	8.2	31.5	732.3
1.5	8.7	25.3	876.9

As shown in figure 9, the theoretical values show a good correspondence with the experimental measurement of the YS increase shown in table 1. This comparison obviously indicates the solid solution strengthening of oxygen atoms is a main mechanism to drastically improve YS of P/M Ti materials. As previously mentioned, the extruded Ti powder materials showed both high tensile strength and high elongation.

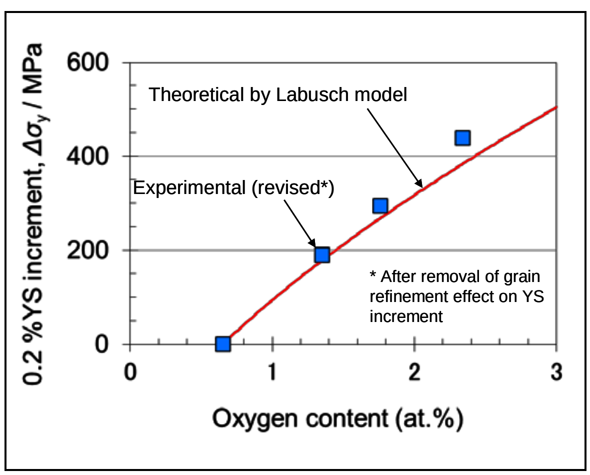


Figure 9. Comparison of YS increase by experiment and calculation by Labusch model.

To investigate the effect of the grain orientation of Ti materials on ductility, extruded Ti materials consolidated by various extrusion ratios were prepared. Heat treatment at 1073 K for 14.4 ks was applied to form the same grain size and remove the induced strains hot extrusion. The extruded Ti powder materials with no TiO_2 additive have a mean grain size of 35–40.7 μm and there is no remarkable difference between the specimens. The oxygen content of the Ti materials is 0.208–0.215 mass% and no significant difference is also detected. As shown in figure 10, UTS and YS are almost constant regardless of the extrusion ratio from 1.0 to 37.7 because there is little difference in the mean grain size and oxygen content of the extruded Ti materials. However, the elongation gradually increases with an increase in the extrusion ratio. Electron-beam plasma deposition (EBPD) analysis suggests that the Schmid factor (S_F) of a prismatic slip gradually increases with increase in the extrusion ratio as shown in figure 11 because stronger (0001) basal plane textures are formed by severe plastic deformation with a larger extrusion ratio. In general, the main slip system in hexagonal close packed (hcp) Ti (α) creates a main deformation mechanism in a prismatic plane. According to figure 11, the extruded Ti powder material with R = 37.7 showed the largest S_F value in the prismatic slip, and results in the increase of elongation because of the formation of active prismatic planes.

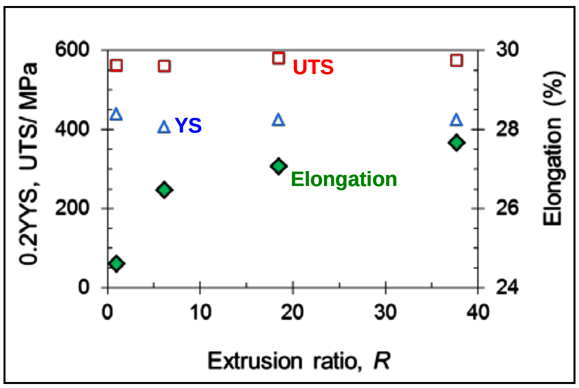


Figure 10. Tensile properties of extruded pure Ti powder materials with various extrusion ratios.

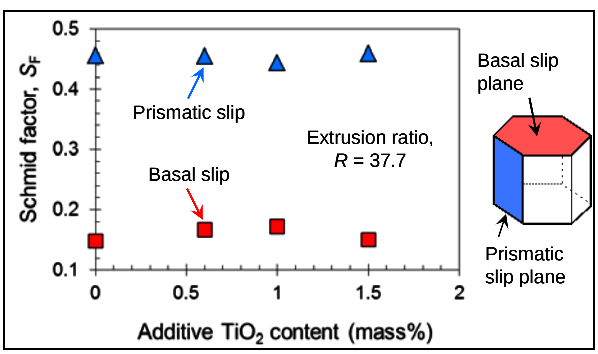


Figure 11. Dependence of Schmid factors (basal slip and prismatic slip) of extruded Ti-TiO_2 powder materials on additive TiO_2 content.

2.3 Ballistic Performance of Extruded Ti Powder Materials

In the case of Ti64-TiO_2 mixture powders, the tensile test results of the extruded bars are summarized in table 2. See figures 14–16 for the post-ballistic pictures. As the TiO_2 was increased, the ductility was reduced, which reduced the ability of the metal to dissipate the energy from the projectile.

Table 2. Tensile properties of Ti64-TiO_2 extruded plates used for ballistic evaluation test.

***θ*= 0**	**0.2%YS (MPa)**	**UTS (MPa)**	**Elongation (%)**
TiO_2; 0%	1123	1126	18.2
TiO_2; 0.5%	1133	1204	20.5
TiO_2; 0.8%	1247	1277	12.3
***θ*= 0**	**0.2%YS (MPa)**	**UTS (MPa)**	**Elongation (%)**
TiO_2; 0%	1106	1101	21.2
TiO_2; 0.5%	1075	1148	17.6
TiO_2; 0.8%	1266	1286	8.5

The anisotropic properties of the bars with the extrusion ratio of R = 37.7 are evaluated in two directions; θ = 0 (in the extrusion direction) and θ = 90 (transverse direction) as illustrated in figure 12. The number of tensile test specimens for each direction was three, and the average measurement was shown in table 2. The materials also showed an improvement of tensile strength by adding TiO_2 particles. However, when 0.8 mass% TiO_2 additives were contained, the extruded bar revealed a remarkable decrease of ductility.

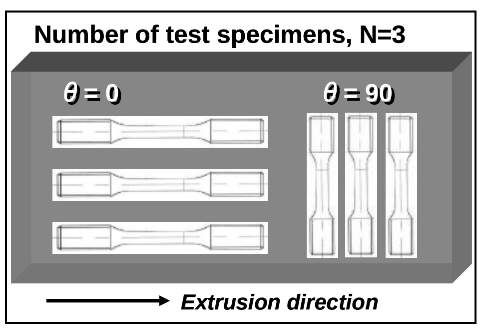


Figure 12. Schematic illustration in machining tensile test specimens of scale-up extruded plates.

These extruded bars of titanium composites were evaluated on an equivalent weight (i.e., areal density [AD]) basis and compared to conventionally rolled thin Ti-64 plate data (*7*). The ballistic limit (V_{50}) of each titanium alloy composition is displayed in table 3. The ballistic limits vs. thicknesses are displayed in figure 13.

Table 3. Ballistic evaluation results of extruded bars using Ti64-TiO_2 mixture powders.

Bar Type	Thickness (in)	Thickness (m/s)	Velocity (ft/s)	Velocity (m/s)	Comments
Ti-6Al-4V (Conventional)	—	—	1848	820	—
Ti-6Al-4V + 0%wt TiO_2	0.424	10.763	2760	841	—
Ti-6Al-4V + 0.5%wt TiO_2	0.422	10.706	2915	888	—
Ti-6Al-4V + 0.8%wt TiO_2	0.416	10.560	2884	879	Bar broke

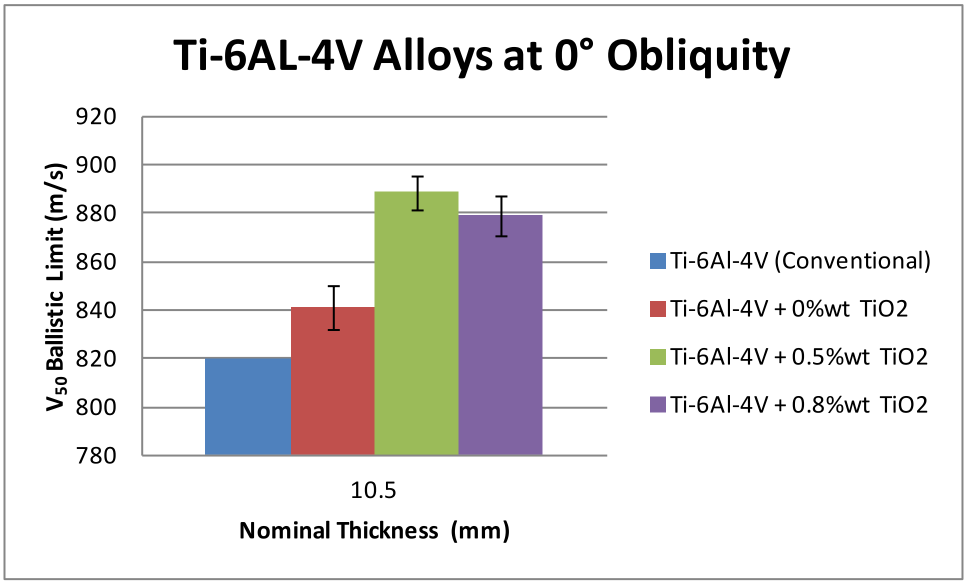
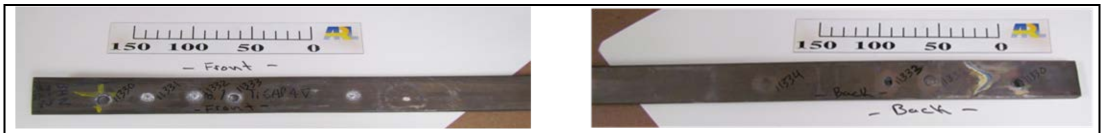


Figure 13. Comparison of ballistic capability of extruded plates using monolithic Ti64 and Ti64-TiO_2.



(a) Front (b) Back

Figure 14. Titanium composite bar: Ti-6Al-4V.

(a) Front (b) Back

Figure 15. Ti-6Al-4V + 0.5 wt% TiO_2 bar.

(a) Front (b) Back

Figure 16. Ti-6Al-4V + 0.8 wt% TiO_2 bar.

The powder alloys exhibited an improvement in ballistic performance of up to 7% over the conventionally rolled Ti64. An addition of 0.5% TiO_2 increased the V_{50}, but an addition of 0.8% TiO_2 compared to Ti64-0.5% TiO_2 did not increase the V_{50}. The reduced ductility of the Ti64-0.8% TiO_2 bar is the most reasonable explanation why the V_{50} did not increase.

3. Conclusion

The addition of TiO_2 particles to titanium powder materials significantly increase the strength of the titanium through oxygen solid solution strengthening. In the case of pure Ti powders, the extruded material showed a good balance between high strength and elongation. However, when using Ti64 alloy powders, their extruded materials revealed a decrease of ductility while increasing tensile strength. On an equivalent-weight basis, the Ti-6Al-4V + 0.5 wt%. TiO_2 bars remained intact during ballistic evaluation and provided a solid 8% ballistic improvement over conventionally rolled Ti64 plate. The Ti-6Al-4V + 0.8-wt% TiO_2 bars fractured during ballistic evaluation and provided a 7% improvement over the conventionally rolled Ti64 plate. These initial results indicate the addition of titanium dioxide powder can boost Ti-6Al-4V ballistic performance. Optimization of the Ti-6Al-4V with TiO_2 powder composition while preserving the ductility of the base material will allow more energy dissipation to occur, thus further improving its ballistic performance.

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