Effects of Post Processing Heat Treatments on the Bond Quality and Mechanical Strength of Ti/Al3003 Dual Materials Fabricated using Ultrasonic Consolidation

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Abstract

The interface between layers in ultrasonically fabricated parts is often poor for desirable material combinations, resulting in relatively low bond strength. This makes these fabrications unsuitable for structural applications. This work discusses a study of the effects of post processing heat treatment of ultrasonically consolidated titanium and aluminum dual-material specimens. The shear strengths of as-deposited specimens as well as heat treated ones were tested. The results show that there is significant improvement of the strengths of post processed specimens over the as-fabricated ones. The improvement is as a result of interactions of the base materials across the interfacial boundaries at elevated temperatures, leading to stronger bonds. The study highlights the role of post processing for improving the mechanical properties of ultrasonically consolidated structures.

1. Introduction

Ultrasonic consolidation is a solid-state fabrication process that combines ultrasonic metal welding and layered manufacturing techniques to produce three-dimensional freeform objects. The process uses the power of high frequency ultrasonic vibration at low amplitude to bond thin foils of materials to form solid objects. It combines normal and oscillating shear forces on mating foils and the resulting friction forces between the materials to fracture and displace surface oxides from the materials. The exposed atomically cleaned surfaces are then brought into direct contact under modest pressure and temperatures that are less than half of the melting point of the materials. The materials are thus metallurgically bonded [1]. Fractured oxides and surface

impurities in the materials are distributed in the bond zone. The process combines the layer-by-layer addition of foils with contour milling using the integrated 3-axis CNC machining facilities to produce desired component geometry. It is therefore both an additive and subtractive process. Notable advantages of the solid state nature of the UC process are discussed by White [1].

The UC machine consists of a welding horn, also known as a sonotrode, that exerts a normal force and the oscillatory high-frequency vibration on the materials to be welded. Welding takes plate on a substrate fixed on a heated plate or anvil. The UC machine is designed for automatic foil material feeding, but materials can also be fed manually. Figure 1 shows the schematic view of the ultrasonic consolidation process. The primary process parameters are vibration amplitude, temperature, welding speed, and normal force [2]. Other parameters that can affect weld qualities include sonotrode roughness, materials surface finish [3], and side-by-side foil positioning accuracy with respect to the automated material feed system [4].

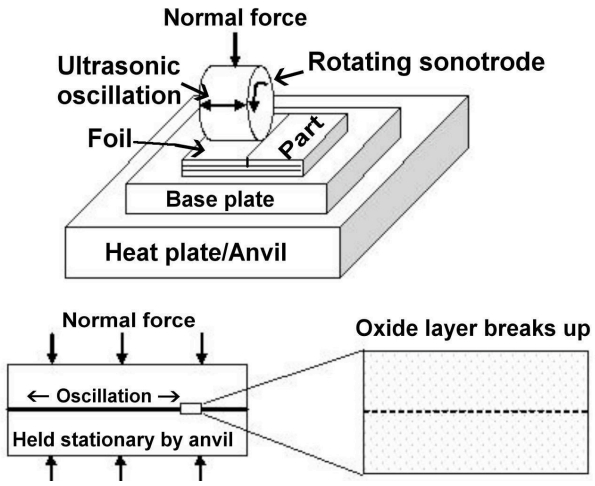


Figure 1: Schematic of UC process

Ultrasonic consolidation technique is applicable for rapid tooling for injection molding, extrusion, vacuum forming tools and others. It is also used for fabricating tools with conformal

cooling channels [1]. Previous works have demonstrated other potential applications for UC. These include honeycomb structures [5], embedding shape memory alloy (SMA) fibers and silicon carbide fibers in aluminum matrices [6-9], and embedded electronics structures [10]. While the process has been widely used for single material fabrications using aluminum alloys, a few researchers have demonstrated its capabilities for multiple material fabrications. The multi-material capabilities of UC was demonstrated by Janaki Ram *et al.* [11] in their work in which copper, brass, nickel, inconel 600, AISI 347 stainless steel, stainless steel AISI 304 wire mesh, MetPreg, and aluminum alloy 2024 were individually welded to aluminum 3003 H18 materials. Domack *et al.*, [12], demonstrated the capability of UC for graded materials composition fabrications using titanium and nickel alloys. Obielodan et al. [13] have also demonstrated UC multi-material capabilities by welding different combinations of molybdenum, tantalum, titanium, copper, silver, nickel aluminum alloys 1100, 3003, 6061 and boron powder.

The bond strengths of UC fabricated structures is a major concern in attempts to use them for mechanically stressed structural applications. Some earlier works have determined inter-layer bond related mechanical properties of ultrasonically consolidated structures. Kong *et al.* [14] determined the peel strength of Al alloy 6061 and Tuttle R.B. [15] determined the peel strength between stainless steel 316L foils welded on a stainless steel plate. Also, Yanzhe Yang *et al.* [8] determined the peel strength of aluminum alloy 3003-H18 with embedded silicon carbide fibers in aluminum matrix, and Obielodan *et al.* [4] determined the conditions for optimum transverse tensile strength of ultrasonically consolidated structures made with automatically fed aluminum alloy 3003-H18 foils. While most of the previous works were on similar materials, there is a growing interest in multi-material structures fabrication by ultrasonic consolidation. A major limiting factor is that many materials are not easily joined at the current limits of operating

parameters of available UC machines. Some material combinations will require higher values of parameters that are beyond the upper bounds of the machines currently available. The present work seeks to apply post process heat treatment as a way to improve the bond strength between ultrasonically consolidated titanium and aluminum alloy 3003. Although the low temperature operating conditions of UC is a major advantage as stated earlier, it is postulated that by applying post processing treatments such as heat and pressure at optimized levels, significant inter-layer material diffusion across the consolidated foil interface will be achieved resulting in better bond strengths. It is a well known fact that high temperature treatment of some dual materials lead to the formation of brittle intermetallic phases; this study seeks to avoid or minimize their formation.

The UC post process heat treatment in this work is limited to elevating UC fabricated specimens to higher temperatures without pressure application. The improved strength advantages derivable from the post process heat treatments will most probably offset the additional costs incurred. The study seeks to create a synergy by combining the freeform fabrication capabilities of UC additive manufacturing with the inherent advantages of diffusion bonding. Improved diffusion based interlayer bond strengths and achievable complex geometries will expand the scope of applications for UC structures. The success of this work with titanium and aluminum 3003 will lay the ground work for further investigations and eventual commercialization for aerospace applications and in other industries.

2. Experimental Work

2.1 Material Preparation

A Solidica Formation™ machine was used for all the UC fabrications in this work. The materials used were annealed commercial pure (CP) Titanium foils of 75 microns thickness and Aluminum alloy 3003-H18 of 150 microns thickness with chemical compositions shown in Table 1. Deposits were made on Aluminum 3003-H14 substrates. Aluminum substrates and foils were procured from Solidica. The titanium and aluminum foils were welded on the substrate in alternate layers for four layers, with titanium foils being the first to be welded onto the substrate. Different welding parameters were applied for the two materials as listed in Table 2. The welding temperature was maintained at 300°F (150°C) for the two materials.

Table 1: Chemical compositions of the materials used and their form

Material	Nominal Composition	Tensile Strength (MPa)	Shear Strength (MPa)	Thickness (µm)
CP Titanium	Ti-0.6Fe-0.38Mn	430	380	75
Al 3003-H18	Al-1.2Mn-0.12Cu	200	110	150

Table 2: Ultrasonic welding parameters used for the different materials

Material	Amplitude (µm)	Speed (mm/s)	Normal Force (N)	Temperature (°F)
CP Titanium	28	10.58	2000	300
Al 3003-H18	16	23.70	1750	300

Titanium foils were manually placed on the substrate for welding while the aluminum foils were automatically fed. Other layers of materials were welded on top of the first four layers in order to strengthen the welded foils and avoid tensile failure outside the joints as reported for

two single aluminum 3003 foils tested for lap shear tests by Kong *et al.* [2].The shear test specimens, described in Fig.2 were designed to fail in shear along lapping surface between the first titanium foil and the aluminum 3003-H14 substrate material. During initial specimen trials,

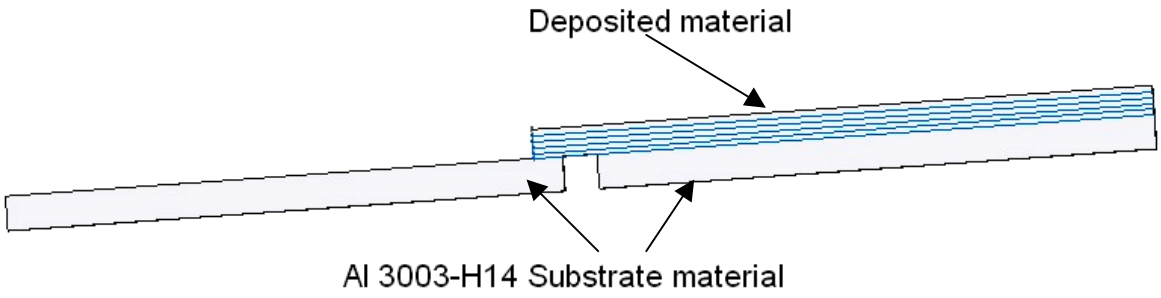


Figure 2: Lap shear strength design

slots of 3.2mm width and 3mm depth were machined into the Al 3003 substrate with the integrated CNC facilities in the UC machine, before the first titanium foil deposit was made. The initial slot was to provide a precise separation between the ends of the tensile shear specimens at predetermined location. It was found that the foils deposited above the slots were not bonded to each other as there was no support material underneath to resist the normal force of the sonotrode for proper welding. The titanium foils exposed by the cut slots were thus highly oxidized during heating in the oven. These specimens failed prematurely because the tensile loads applied were not evenly distributed, and the exposed titanium foils were weakened by oxidation. With subsequent specimen trials, the slots were not cut into the substrate before the foil welding. They were cut using a milling machine after material deposition and post process heat treatment were completed.

Eighteen specimens were fabricated and randomly grouped into six groups of three specimens each. Each group was then randomly assigned to a post process annealing at 480^{o}C

for the following length of time: 0 (control), 30, 60, 120, 180 and 270 minutes, after which they were oven cooled to room temperature. The treatments are respectively labeled A, B, C, D, E and F for the purpose of analysis. Those assigned to zero minutes (treatment A) were not heat treated, and served as the control specimens for comparison with the groups that were subjected to post process annealing. A Lindberg BlueM laboratory table top oven without atmospheric control was used for the post process annealing treatments. The foil lapping surfaces were not exposed to the oven atmospheric conditions, only the edges (which are very small compared to the total surface area of the titanium foils) were exposed, and as such minimal lapped surface oxidation could occur. The samples were loaded into the oven for annealing without the substrates machined off in order to avoid heat induced distortion in the specimens. After removal from the oven, the substrate materials were machined down to 3mm after which the slot was cut to separate the two ends of the tensile shear specimens, which were then only joined at the overlapping surface of the welded foils as shown in Figure 2. Because the preparations for the slot machining were manually done, the slot cuts resulted in different overlap lengths for the specimens. The resulting overlap lengths of the specimens ranged from 2.32 to 3.3mm. It is assumed that the differences does not significantly affect the lap shear strength measurements obtained.

A 50KN capacity Tinius Olson tensile testing machine was used for the lap tensile shear test. The specimens were held in the flat grips of the testing machine and pulled in tension at a speed of 1mm/min until fracture. The maximum fracture load was divided by the area of overlapping surface to arrive at the failure shear stress for each specimen.

2.2 Metallography

Small samples cut from the unstrained grip ends of the tensile shear strength specimens were mounted and polished according to standard metallographic procedures. They were then etched with Kellers reagent (90ml H_2O, 5ml HNO_3, 3ml HCl and 2ml HF) and observed in optical microscope (OM) and scanning electron microscope (SEM). Line scan Electro Dispersed X-ray (EDX) analysis across the interface of the titanium and Al 3003-H18 deposits were undertaken for one representative specimen randomly selected from each of the five specimen groups that were annealed. This was done to verify if post process heat treatment-induced diffusion took place (and the distribution of diffusing elements, if it occurred) across the interface of the consolidated materials. The lack of atomic diffusion across the interface of ultrasonically consolidated dual material foils in previous experiments indicated that it was unnecessary to carry out EDX analysis on any of the control specimens. It is assumed that detectable diffusion will not occur at the UC operating parameters for the as-deposited samples.

3 Results and Discussion

3.1 Microscopy

The optical and SEM micrographs of the specimens are shown in Figures 2-8. The arrows in Fig.2a show the titanium foils.

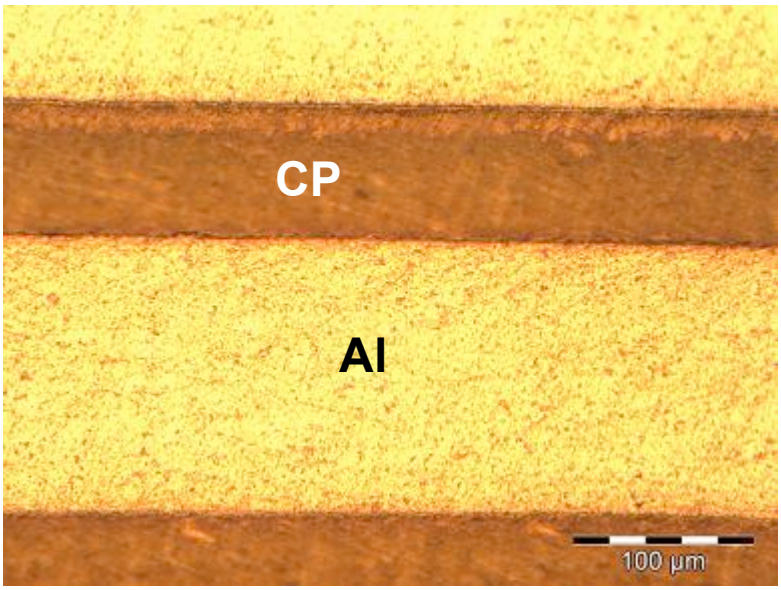
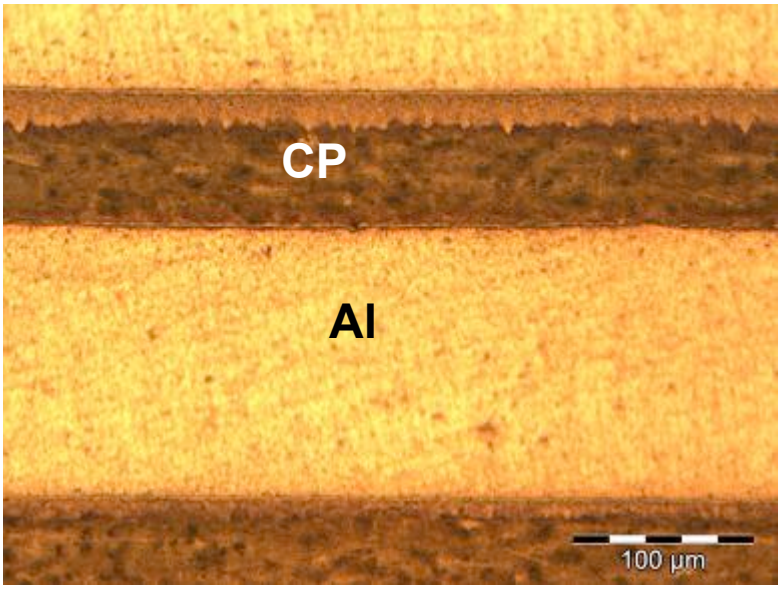
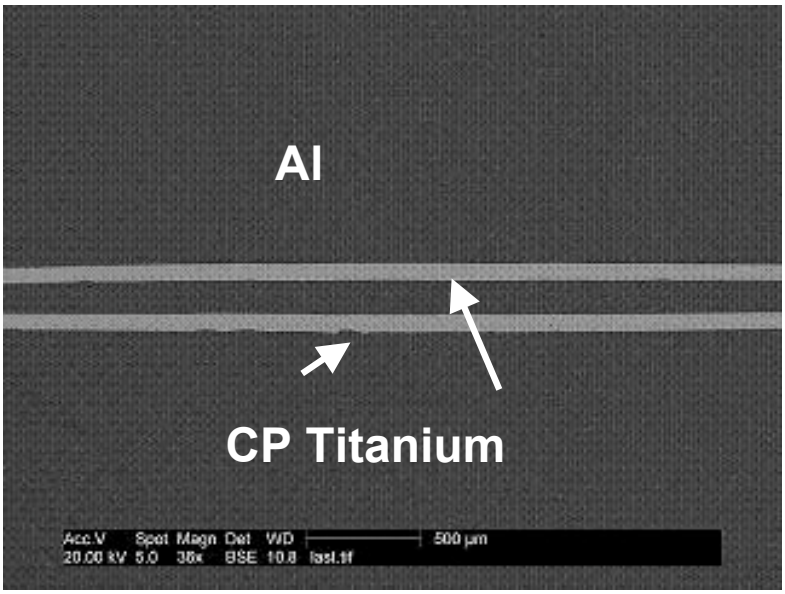


Figure 2: OM micrograph of a specimen without post processing



a: OM micrograph of a 30 minutes annealed specimen



b: SEM micrograph of a 30 minutes annealed specimen

Figure 3: Sample OM and SEM micrographs of 30 minutes annealed specimens

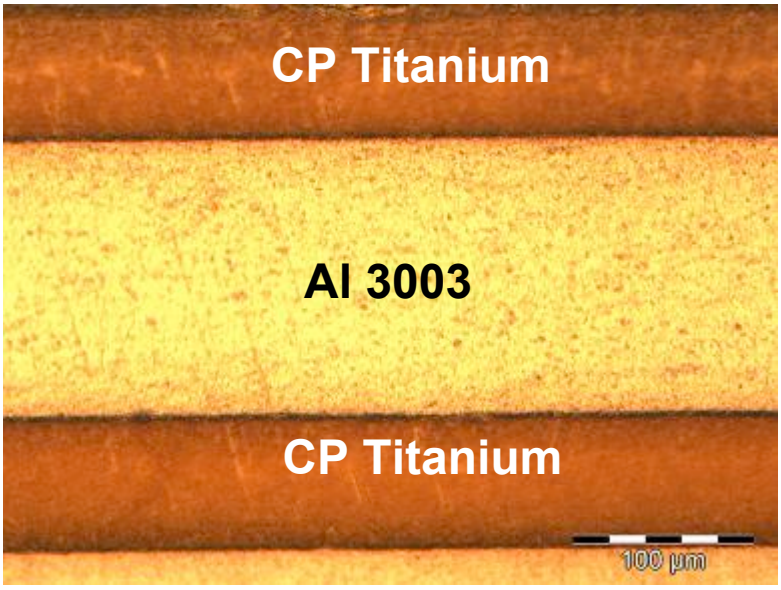


Figure 4: OM micrograph of a 60 minutes annealed specimen

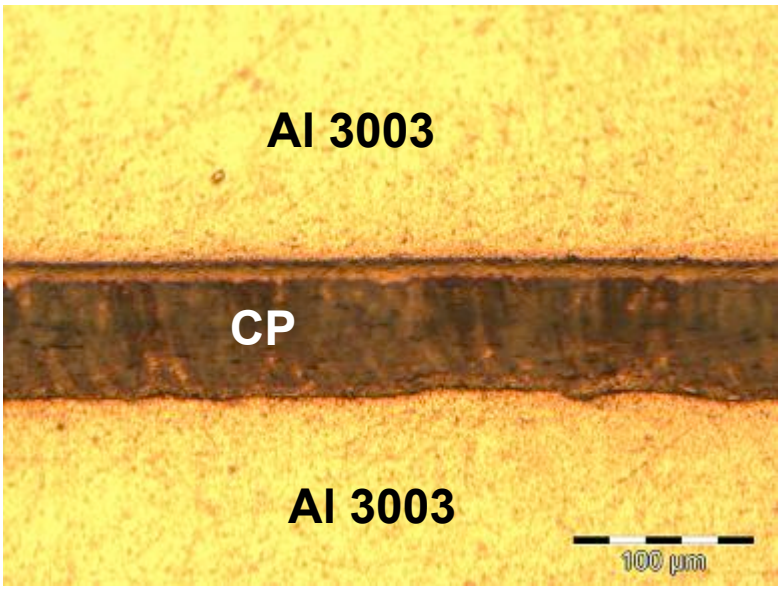


Figure 5: OM micrograph of a 120 minutes annealed specimen

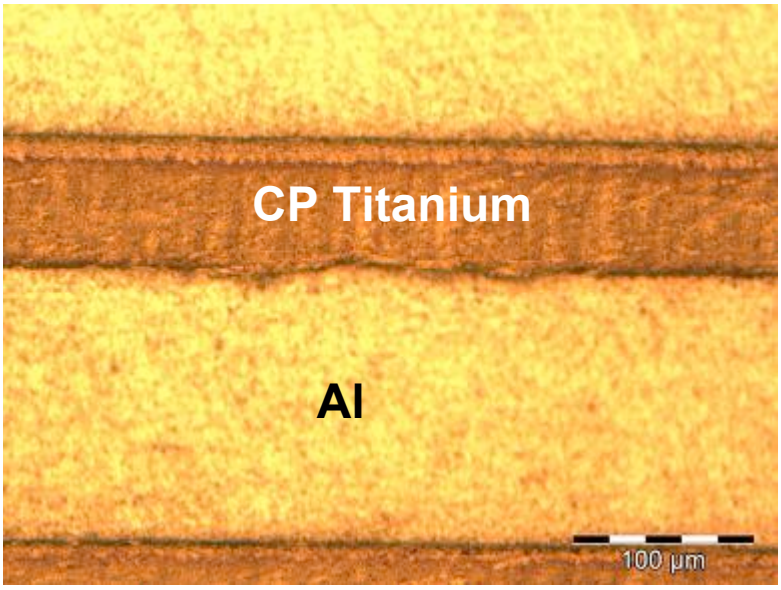


Figure 6: OM micrograph of a 180 minutes annealed specimen

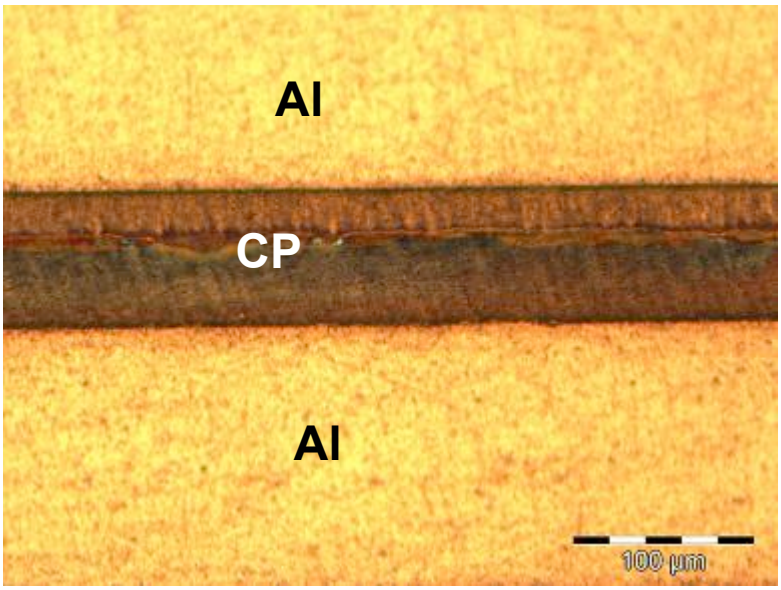
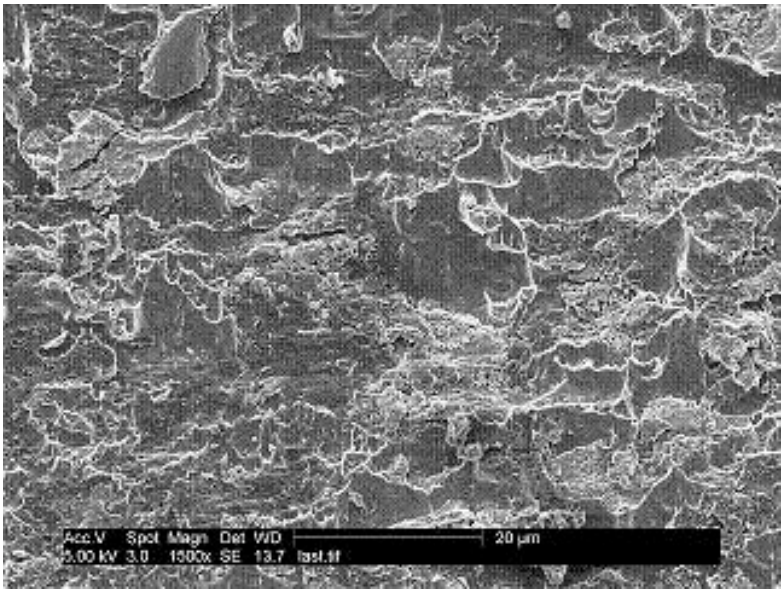
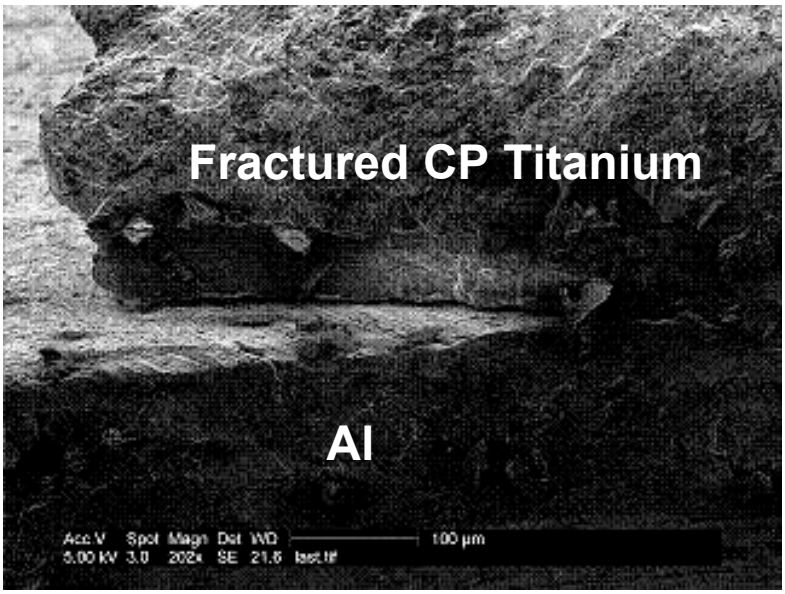


Figure 7: OM micrograph of a 270 minutes annealed specimen



a: SEM micrograph showing the fracture surface of a 30 minute annealed specimen



b: SEM show one of the fracture mode of a 30 minute annealed specimen

Figure 8: SEM of the fracture features of the specimens

The micrographs in Figures 2 to 7 shows that the bonds between consolidated titanium and aluminum alloy foils were generally good. There are no visible bond defects between the layers. Figure 8 shows the fracture features of the specimens. The fracture morphology at the lapped surfaces is shown in Fig.8a. Figure 8b shows how in some of the specimens, the first consolidated titanium layer that bridge the two halves of the lap shear specimens failed in tension. The tensile failure of the first consolidated titanium layer is most probably due to the delamination of that layer that occur as a result of induced bending moment about the center of the lapping surface as the applied tensile shear load reaches a particular level (Fig.9 shows the picture of the bent profile of a 30 minute annealed specimens). The delamination causes differential strain between the separated foil and the undelaminated ones. The delaminated foil fails early because the applied stress soon exceeds its tensile strength as its thickness is relatively small. In those cases, the lapping foil fraction is completely separated from the longer side

causing the immediate consolidated aluminum foil to fracture by shear as the load increases. The output data obtained from the lap shear strength tests are shown in Table 3 and Figure 10.

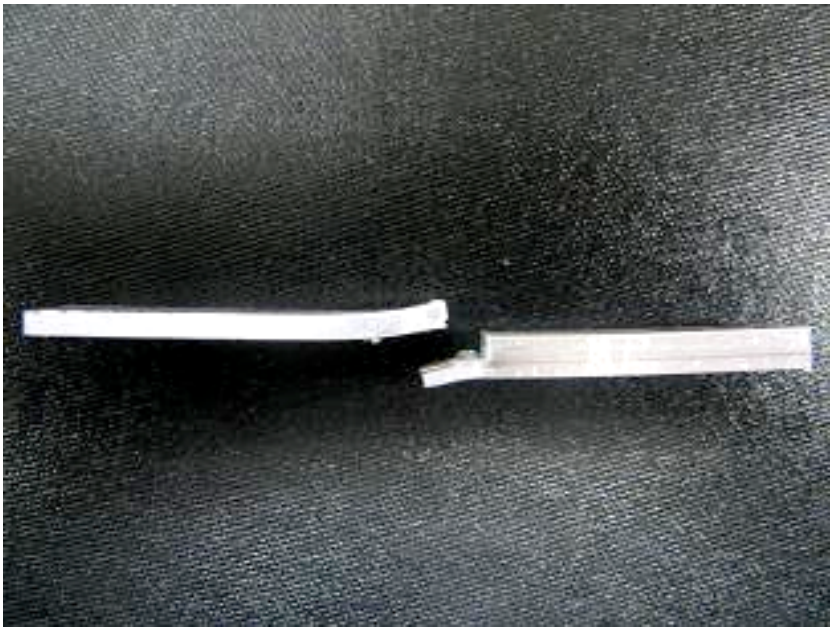


Figure 9: A lap specimen with bent lapped section

Table 3: Lap Shear Strength (MPa) data

Annealing Time (minutes)	Samples 1	2	3	Average
0	44.30	31.48	37.56	37.78
30	69.34	62.73	86.82	72.96
60	51.17	43.06	70.73	54.99
120	53.81	33.53	41.96	43.10
180	46.25	32.26	53.84	44.12
270	53.61	69.96	50.87	58.15

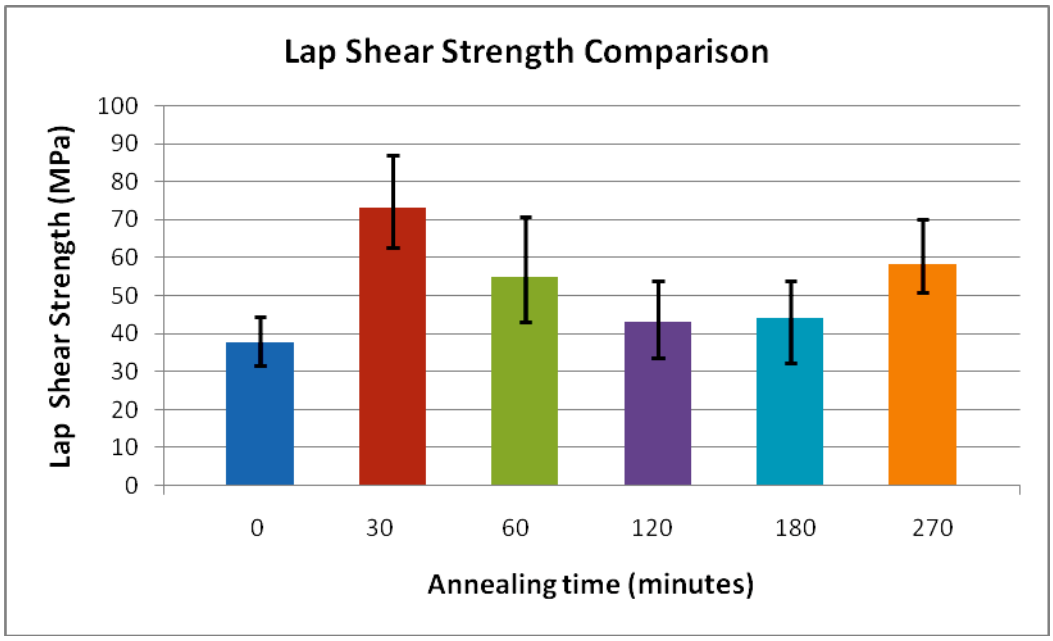


Figure 10: Bar chart of the lap shear strengths of the different groups of specimens.

SAS 9.1 software was used for the statistical analysis of the data to verify the effect of the post process annealing as a single factor with six treatment levels on the shear strengths of the specimens. The boxplot from the analyses did not show any outlier and the normal quantile plot is close to a straight line. Also, all the tests for normality have high P-values, so the assumption of approximate normality of the data is satisfied. The results of the analysis also show that there is a uniform spread of the errors, which means the data satisfies the homoscedasticity assumption. The analysis of variance (ANOVA) in Table 4 with a P-value of 0.0210 shows that post process annealing has a significant effect on the lap shear strengths for the different groups of specimens. A post hoc mean comparison using the Ryan-Einot-Gabriel-Welsch multiple range test (REGWQ) method is shown in Table 5. The REGWQ controls the maximum experiment-wise error rate under any complete or partial null hypothesis. The table shows that the specimens with 30 minutes post process annealing (treatment B) yield the highest mean shear strength of 72.963MPa. Since treatment B shares the same REGWQ group A with specimens that were

annealed for 270 minutes (treatment F) with mean shear strength of 58.147MPa and with those annealed for 60 minutes (treatment C) with mean shear strength of 54.987MPa, it shows that its differences in result with those two treatments groups are not statistically significant. It (that is, treatment group B) however, have statistically significant higher mean shear strength than specimens that were annealed for 120 minutes (treatment D), 180 minutes (treatment E), and the control specimens (treatment A).

Table 4: Result of Analysis of Variance (ANOVA) for the data

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	5	2488.774111	497.754822	4.10	0.0210
Error	12	1457.120067	121.426672		
Corrected Total	17	3945.894178			

Table 5: REGWQ post hoc means comparison

Means with the same letter are not significantly different.				
REGWQ Grouping		Mean	N	treatment
	A	72.963	3	B
	A			
B	A	58.147	3	F
B	A			
B	A	54.987	3	C
B				
B		44.117	3	E
B				
B		43.100	3	D
B				
B		37.780	3	A

The result of the line scan EDX analysis across the interface of the consolidated dual materials reveals that diffusion took place at the 480^{o}C annealing temperature. Figure 11 shows the diffusion trend of aluminum into the titanium foil at the interface. It can be seen that the extent of diffusion of aluminum into titanium was different for the different post process annealing durations. The Figure shows that the specimens that were annealed for 30 minutes had about 13 wt.% composition of aluminum at the surface of the titanium foil. At a depth of 0.2 microns into the titanium surface, the aluminum composition is about 7 wt.%. From the titanium-aluminum phase diagram shown in Fig. 12, alpha titanium (α-Ti) can hold up to 7 wt% aluminum in solid solution at 480^{o}C, beyond which apha2 titanium (α_2-Ti_3Al), an intermetallic phase, gradually begins to precipitate. It therefore means that within a depth of 0.2 microns from the surface of the titanium foil, α-Ti and α_2-Ti_3Al coexist after annealing. There is the posibility of these materials interfacing with a very narow band of other titanium aluminide intermetallics on the aluminum side of the interface. Figure 11 shows higher weight percent concentrations of aluminum diffused deeper into titanium foils for longer annealing times than 30 minutes. Using the titanium-aluminum phase diagram, it can be seen that thicker layers of titanium aluminide intermetallic phases must have formed at the interface. The specimen group F (with 270 minutes annealing) recorded the highest depth of high concentration of aluminum diffusion into the titanium foils.

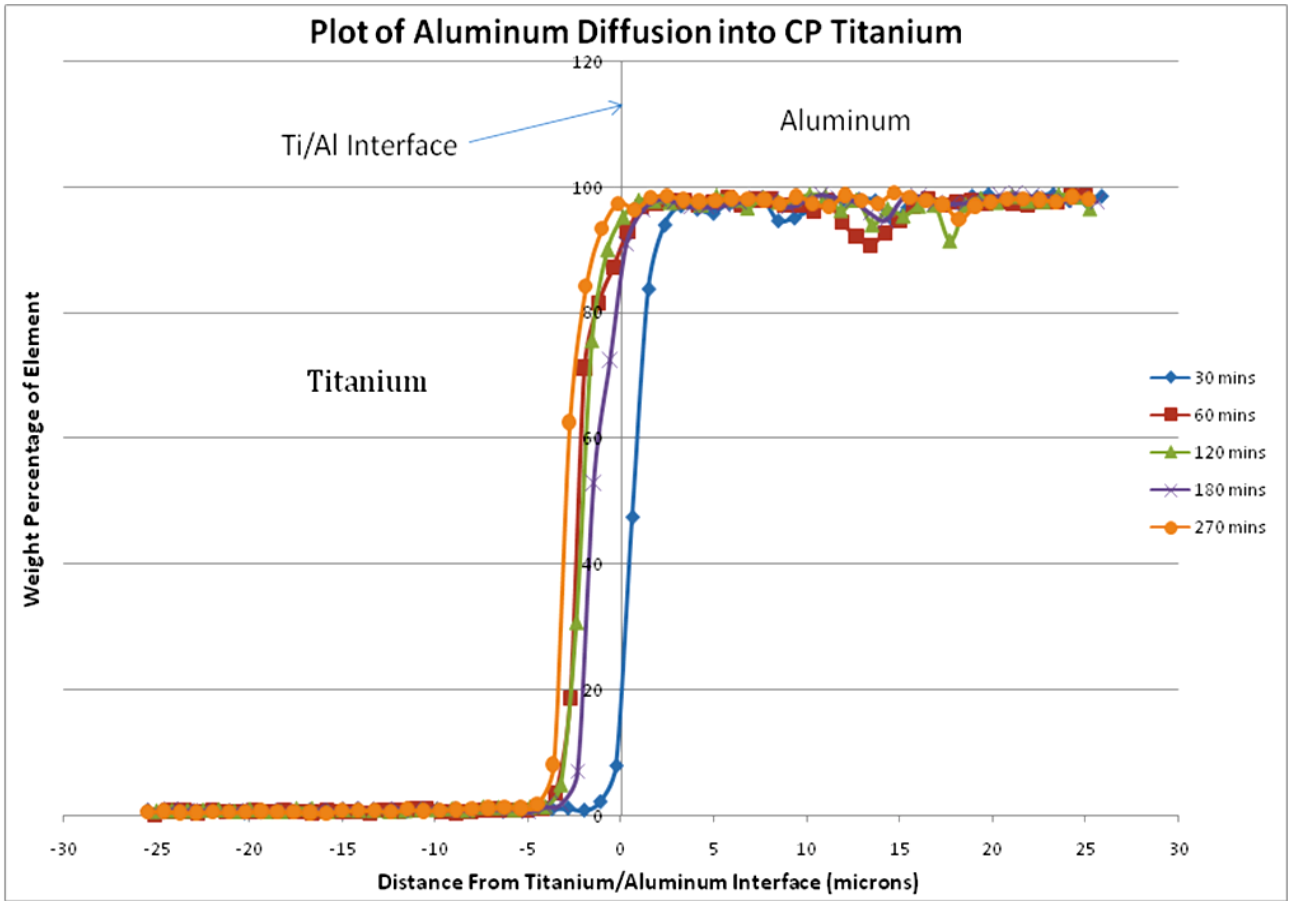


Figure 11: A plot of line scan EDX analysis result showing diffusion trend of aluminum into titanium at the interface at different annealing times

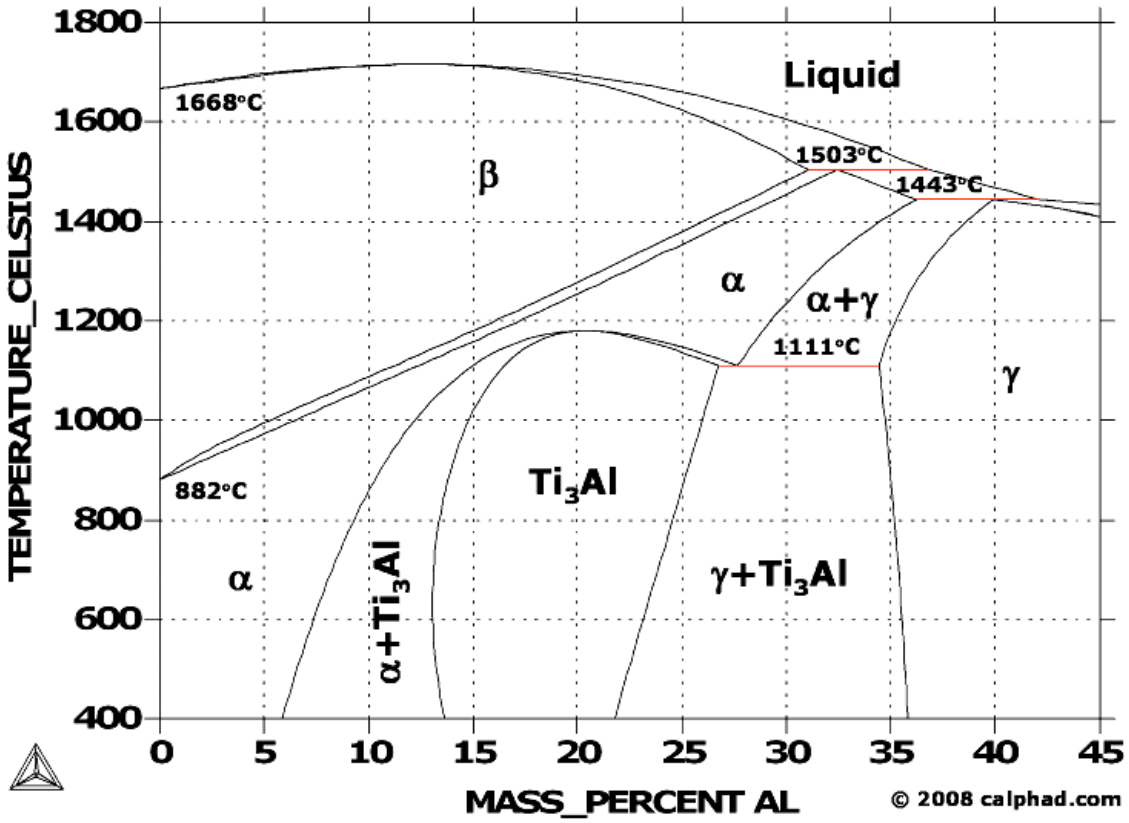


Figure 12: Titanium-aluminum (Ti-Al) phase diagram [16]

It can be infered that the group B specimens yielded the highest average tensile shear strength because of the significant aluminum in solid solution of the alpha titanium at the interface, which co-existed with the narrowest intermetallic phases that may have formed. Specimen groups C, D and E must have failed at relatively lower shear stresses as a result of the higher depths of brittle intermetallics occassioned by the higher depth of penetration of aluminum below the titanium surface. Because the intermetallics are hard, their thicker layers produced in the group F specimens must have offered some resistance to early failure thereby yielding relatively higher shear strengths than the C, D and E groups. The shear strengths recorded for the thicker intermetallic layer is however not reliable under service conditions. It is always better to avoid or minimize the formation of the intermetallic phases for reliability under service conditions. This makes the group B treatment prefered over the other treatments.

Although the mechanical properties of the CP titanium used is yet to be experimentally verified, it is generally known to be superior to those listed for aluminum 3003-H18 in Table 1. It is also known that the shear strengths of the consolidated titanium-alumnium dual materials can not exceed that for Al 3003-H18 base material of 110MPa. Despite falling short when compared to the shear strength of the aluminum alloy 3003-H18 shear strength, the highest average of 72.96MPa for the post conslidation annealed titanium/Al 3003-H18 is a significant improvement over the 37.78MPa average shear strengths obtained for the as-consolidated specimens. The effects of annealing must have also contributed to the reduced shear strengths obtained, as the base materials will undergo softening in the process. It is assumed that if heat treatable aluminum alloys are used, better shear strength values can be obtained. Also, the as-consolidated specimens may not have uniformly perfect bonding, which can affect the ultimate strength values of the post processed specimens. Application of pressure as another factor is expected to offer improved results.

4 Conclusions

This experiment has shown that post consolidation heat treatment by annealing at 480^{o}C has significant effects on the lap shear strengths of UC specimens. The best average lap shear strength of 72.96MPa was obtained for specimens heat treated for 30 minutes compared to 37.79MPa for as-consolidated ones. Higher heat treatment times result in the formation of thick layers of brittle titanium aluminide intermetallic phases that causes less reliability and early failure under service conditions.

Acknowledgement

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