International Journal of Materials Science and Applications
2019; 8(3): 47-55
http://www.sciencepublishinggroup.com/j/ijmsa
doi: 10.11648/j.ijmsa.20190803.13
ISSN: 2327-2635 (Print); ISSN: 2327-2643 (Online)

Microstructure and Strength of Titanium After Heat Treatment at Different Temperatures in the Range of 680-1000°C

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To cite this article:
Xingyu Zhang, Benjamin Hanes, Daniel Brooks, Steve Niezgoda. Microstructure and Strength of Titanium After Heat Treatment at Different Temperatures in the Range of 680-1000°C. *International Journal of Materials Science and Applications*. Vol. 8, No. 3, 2019, pp. 47-55. doi: 10.11648/j.ijmsa.20190803.13

Received: July 18, 2019; **Accepted**: August 26, 2019; **Published**: August 27, 2019

Abstract: The main purpose of this research is to determine the effect of brazing treatment on mechanical properties of both titanium Grade 2 and titanium Grade 5 alloys. The research group obtained Grade 2 and Grade 5 titanium alloys and brazing-treated them at temperatures of 680, 800, 850, 900, 920, 950, and 1000°C. Afterward, each sample was tensile tested, mounted, hardness tested, and observed by optical microscope to investigate corresponding microstructures. Based on the result sheets, it was revealed that the yield strength and tensile strength and ultimate strength of Ti Grade 2 alloys showed drastic fall after heating to 680°C, then no change up to 850°C, fall again up to 950°C, and remained unchanged strength to 1000°C However, the Ti Grade 5 samples showed completely different behavior. The yield strength was unchanged after heating to different temperatures. When heating to 680°C. It didn't affect the strength at all, then after heating to 800°C, the strength decreased about 100MPa. But after this, higher temperatures didn't change strength anymore. The Ultimate strength however showed a different trend as it continuously went down at elevated temperature. Meanwhile, the hardness of both alloys decreased constantly when temperature increased. Regarding Ti Grade 2 alloys, the initial drop in strength was due to annealing. Around 800°C, alpha laths started to form and that caused strength to increase. When the temperature reached at 850°C, the basketweave alpha laths were formed. Over that temperature, the grain sizes were significantly large which caused the strength to decrease. However, there was not much of change in alpha/beta ratio for Ti Grade 5 alloys. EBSD could be a helpful method since the alpha grain size can be determined from that.

Keywords: Titanium Alloys, Brazing, Microstructures, Mechanical Properties

1. Introduction

Brazing is a metal-joining process that puts two or more metals together by melting and flowing a filler metal into the joint. Brazing has many advantages over other techniques. For instance, brazing could provide a clean joint without the need of secondary finishing. Meanwhile, it produces less thermal distortion than welding. An example of brazed interface of titanium is presented at Figure 1. Also, there would be a thermal cycle for brazed area. The following Figure (Figure 2) shows an example of brazing thermal cycles [1]. Braze alloy is generally available as rod, powder, paste, cream and so on [2]. Some common types of filler metals used are Aluminum-silicon, Copper-silver, Copper-zinc (brass), Copper-tin (bronze), Gold-Silver, Nickel alloy, Silver [3]. Among all types of base alloys, titanium alloys are a hot metallic material because of its high strength and toughness. It can be classified into four categories, which are alpha alloys, near-alpha alloys, alpha and beta alloys and beta and near beta alloys [4, 5]. By working as well as heat treated them below or above the alpha-beta transition temperature, a large change in microstructure can be achieved. Therefore, this would significantly improve the strength of material. The transition temperature is different for distinctive titanium grades. Microstructure of Titanium Grade 2 joint brazed in vacuum at 1180°C, Ni-27 Ti-10Al filler metal can be seen in Figure 1, and Figure 2 shows an example of Titanium Brazing thermal cycle [1]. It is important to mention that the most commonly used titanium alloy is Grade 5 which is known as Ti6Al4V. It has a chemical composition of 6% aluminum, 4% vanadium, 0.25% (maximum) iron, 0.2% (maximum) oxygen, and the remainder titanium. The application of this kind of alloys includes blades, discs, rings, airframes, fasteners, components, vessels, cases, hub [6, 7].

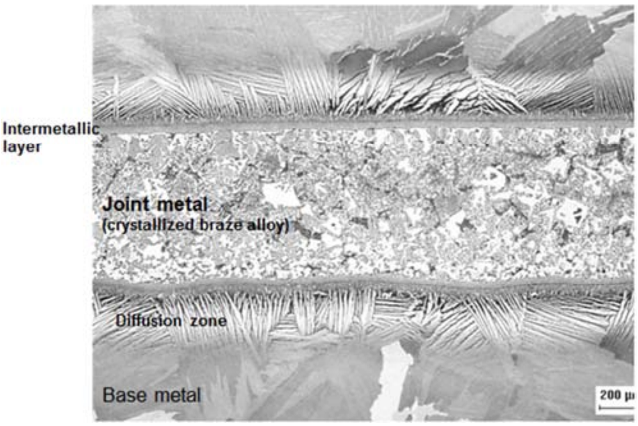


Figure 1. *Microstructure of Titanium Grade 2 joint brazed in vacuum at 1180°C, Ni-27 Ti-10Al filler metal [1].*

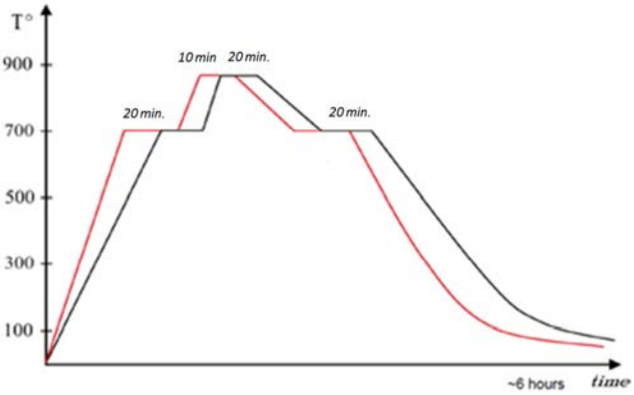


Figure 2. *An example of Titanium Brazing thermal cycle [1].*

In previous research, Scientists tried to investigate several titanium related brazing systems. Some notable publishes included the correlation between the mechanical properties and the microstructural behavior of Al_2O_3-(Ag-Cu-Ti) brazed joint [8]. A range of temperature from 800°C to 1200°C was tested. Also, Scientist tried to reveal the mechanism of Ti/Al dissimilar alloys. Their brazing zone was examined under transmission electron microscope (TEM). A composition difference in different zone and a clear stacking fault structure in intermetallic phase $TiAl_3$ was discovered [9]. Also, it was demonstrated Grade 7 and Grade 5 titanium alloys which were brazed by 25Ti-25Zr-50Cu brazing foil. The mechanical properties around brazing zone were tested and it was found out that fast cooling rate after brazing could suppress formation of the brittle phase, thus resulting in high mechanical properties of the brazed joint [10]. Speaking of more recent studies about titanium alloys brazing, a study revealed a novel multicomponent Ti50-Zr27-Cu8-Ni4-Co3-Fe2-Al3-Sn2-Si1 (at. %) amorphous brazing filler metals with low content of Cu and Ni and low liquidus temperature was designed and synthesized by melt spinning. When brazed with Grade 5 titanium alloys, high shear strength was exhibited around brazing zone [11]. A. Winiowski and D. Majewski also presented a study by brazing of Grade 2 titanium with 6082 aluminum alloy using B-Ag72-Cu28 grade silver brazing metal as an interlayer. Then shear strength and microstructures of brazed joints were studied and it was obtained that the highest quality of the sample was that brazed at 530°C with a 30-minute hold [12].

From those literature reviews, it was discovered that majority of papers focused on the properties at brazing zones, and none of them focus on the metal alloys as a whole. It is significant to know the change of titanium strength due to the effect of a brazing thermal cycle in order to evaluate its effect on reliability of brazed structures. However, a systematic study of the strength of titanium base metals after brazing and auxiliary heat treatment was not done yet. Therefore, our research group investigated how heat treatment in terms of brazing will affect the mechanical properties of titanium alloys and how they were changed at different temperatures (from 680°C to 1000°C).

The main objective of this project is to determine the effects the brazing process would have on the mechanical properties and microstructures of both Grade 2 and Grade 5 Titanium alloys. The group conducts mechanical testing of two types of alloys after heat treatment at 680°C, 850°C, 900°C, 950°C and 1000°C accordingly. Then, each of the samples is mounted and polished so that microstructures can be observed. Finally, the group finds the correlation between strength and microstructures.

2. Materials and Methods

2.1. Materials

In this project, only Grade 2 and Grade 5 were used. Grade 2 represents unalloyed titanium, standard oxygen, while Grade 5 is also known as Ti6Al4V, which is the most commonly used alloy. For Grade 2, the beta transus temperature would be 913°C while it would be 980°C for Grade 5 [13, 14].

2.2. Methods

All heat-treated samples were first tensile tested, then polished and investigated under optical microscope. Afterwards, hardness test was conducted. The specific experimental procedures were as follows:

2.2.1. Tensile Testing

The dimensions of the samples were measured, and one side of the sample was colored in with marker. Laser tape was then added so the laser could measure the elongation in the region between the tape. The samples were loaded in the MTS 810 load frame, and they were subject to a strain rate of 2 mm/min.

2.2.2. Mounting/Polishing

The samples were cut using an abrasive saw, and then they were mounted in conductive bakelite. The samples were polished from 240-600 grit paper and 6-.05µm alumina.

2.2.3. Optical Microscopy

The samples were etched using a Kroll's reagent (6% nitric acid, 2% hydrofluoric acid, 92% water) and imaged at 100, 200, and 1000x magnification. All of the imaging was done using PAXit.

2.2.4. Hardness Testing

The machine was used to measure macrohardness of the samples. Since titanium Grade 5 is harder than Grade 2, Grade 5 was measured on the Rockwell C scale using a 1/16 steel ball indenter and a load of 100 kgf, and Grade 2 was measured on the Rockwell B scale using a pyramidal diamond indenter and a load of 150 kg. After these measurements were taken, the hardness values were converted to the Vicker's scale for comparison.

3. Results

The effects of different alpha grain composition are caused by different treated temperature on the strength of Grade 2 titanium alloys. Data from (Figures 3-5) shows the strength of Grade 2 titanium alloys at different temperature treatment and their respective microstructure under optical microscope. From the micrographs, when the Grade 2 Ti is heat treated for 10 minutes at 680°C, a fully equiaxed alpha grain structure is formed (Figures 3-5). These equiaxed alpha grain can be seen to have secondary alpha laths that have formed inside the grain boundaries. The secondary alpha laths are in a colony microstructure. For the heat treatment at 850°C, the grain size of the equiaxed alpha became marginally larger and the density of secondary alpha laths had increased. The effects of the increase in alpha laths density can be seen by the higher strength of samples heat treated at 850°C compared to those heat treated at 680°C. The Grade 2 Ti heat treated at 1000°C shows a different microstructure than those heat treated at lower temperatures. When treated at 913°C, the beta transus for Grade 2 Ti is well. The treatment itself, only being 10 minutes long, allows little time for the recrystallization process to be completed. During the cooling of the titanium, secondary alpha laths were formed in a basketweave formation.

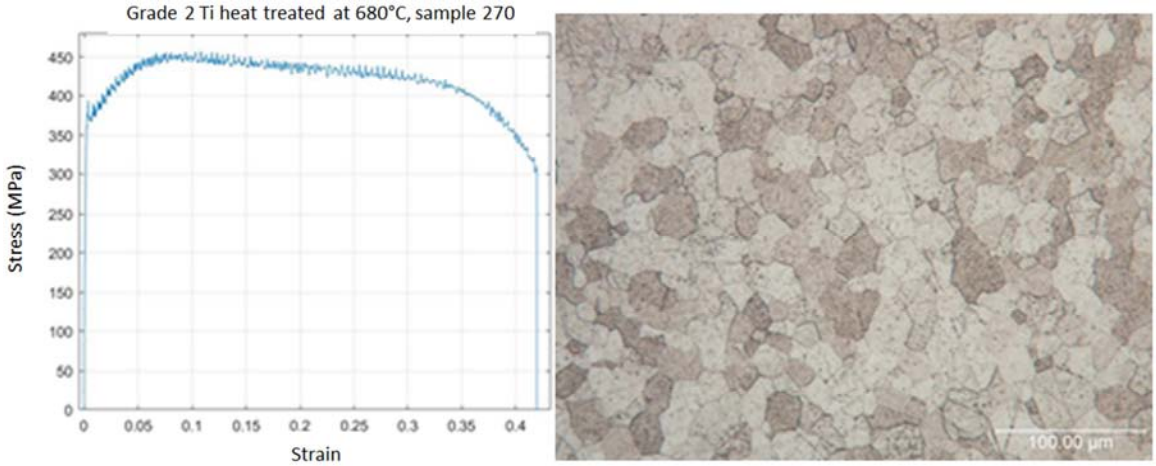


Figure 3. *Stress-Strain curve and micrograph (200x) of Ti Grade 2 treated at 680°C for 10 min.*

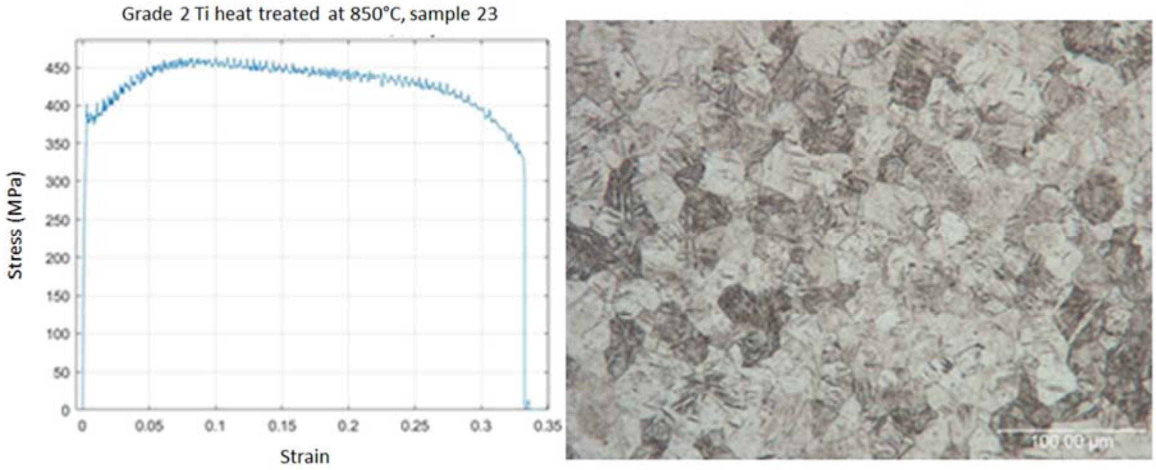


Figure 4. *Stress-Strain curve and micrograph (200x) of Ti Grade 2 treated at 850°C for 10 min.*

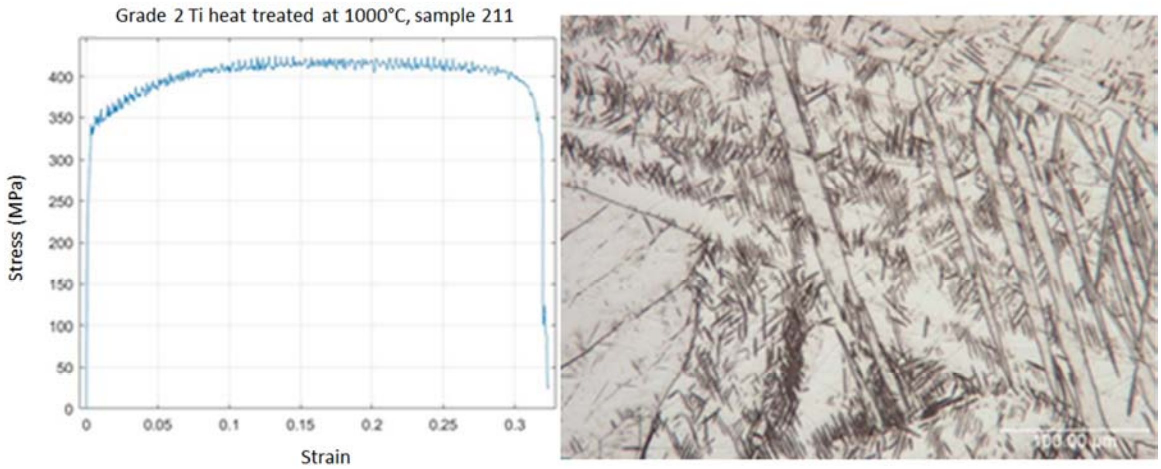
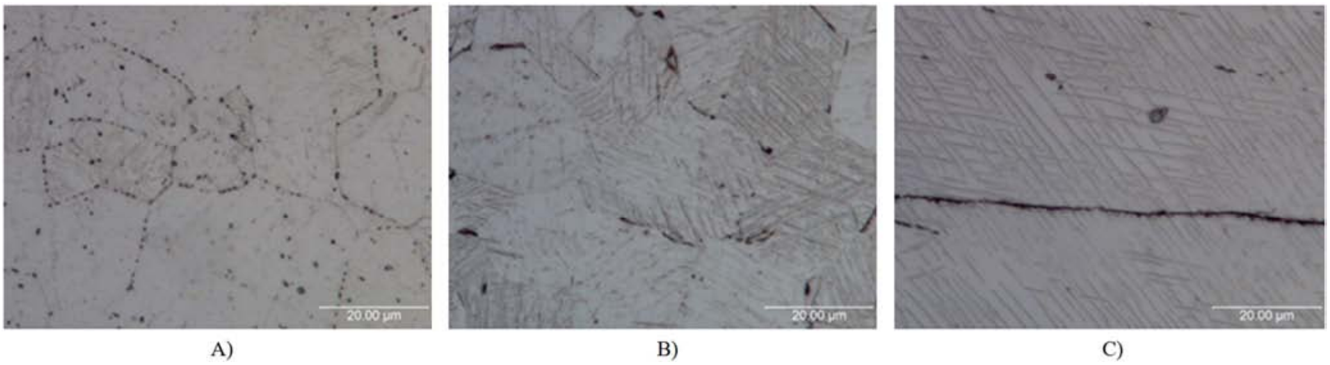


Figure 5. *Stress-Strain curve and micrograph (200x) of Ti Grade 2 treated at 1000°C for 10 min.*

The further analysis on alpha and basketweave laths composition was conducted. Grade 2 titanium alloys at different temperature of treatment were investigated under 1000x magnification (Figure 6). For Grade 2 Ti heat treated at 680°C, some of the grains contained alpha laths in a colony formation. When heat treated at 850°C there were a larger number of grains containing these alpha laths and the basketweave laths formation was also observable. The large grain size caused by the 1000°C heat treatment could be observed, where the average grain size increases 572% compared to that of 850°C.



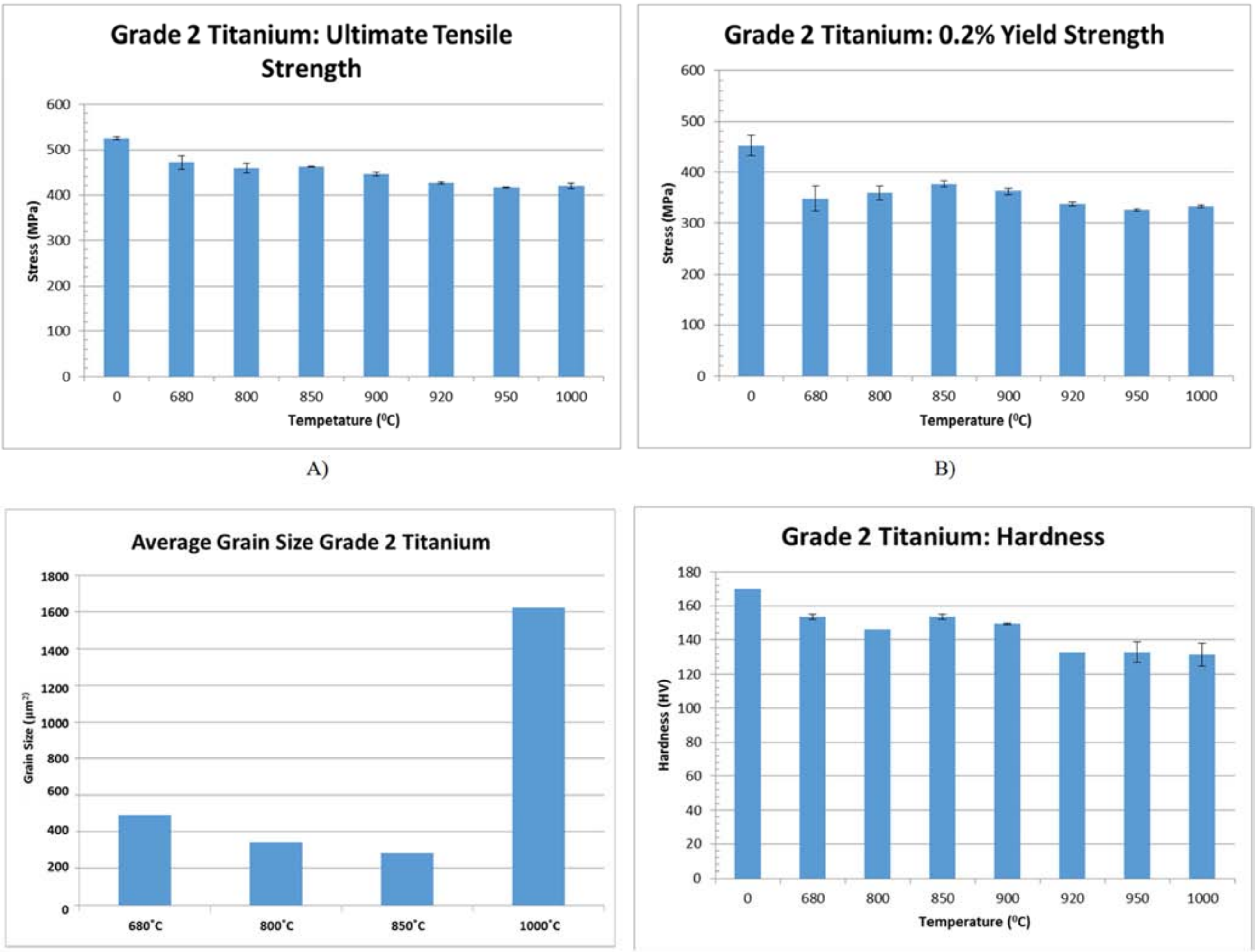
A) B) C)

Figure 6. *Micrographs of Ti Grade 2 treated at (A) 680, (B) 850, and (C) 1000°C at 1000x.*

The mechanical properties comparison at different brazing temperature can be seen in Figure 7. The properties of Ti Grade 2 change as heat-treatment temperature increases as shown. Upon heating, the ultimate tensile strength and yield strength decreases. The As-received strengths are highest, with UTS of 525 MPa and a yield strength of 452 MPa. There is then a 12.7% drop of strength from the as-received to 800°C. This is believed due to annealing at this temperature, reducing residual stresses of the as-received sample. Past 800°C there is an increase in strength. This is attributed to the formation of secondary alpha lath. The strength then peaks at around 850°C at 88% of the as-received strength; here the basketweave lath formation can be seen. Past 900°C, the strength plateaus at about 80% of the as-received strength. There is an extreme growth in grain size that is seen at these temperatures shown at Figure 8, tabulates the data shown in Table 1.

Table 1. *Ti Grade 2 Ultimate Tensile Strength, Yield Strength, Elongation, and Hardness Data.*

Temperature (°C)	UTS (MPa)	UTS Average	YS (MPa)	YS Average	Elongation (%)	Elongation Average	Hardness (Vickers)	Hardness Average
680	456.7	471.7	371.7	347.7	42.0	37.0	152.0	153.5
680	486.6		323.8		32.0		155.0	
850	463.0	462.0	381.7	376.0	33.3	35.6	155.0	153.5
850	461.0		370.4		37.9		152.0	
900	450.1	445.9	368.2	362.2	35.1	35.4	150.0	149.5
900	441.7		356.1		35.8		149.0	
950	416.9	416.6	328.6	326.2	32.5	32.7	139.0	133.0
950	416.3		323.7		32.9		127.0	
1000	424.6	418.8	330.8	333.2	31.9	33.3	138.0	131.5
1000	413.0		335.5		34.6		125.0	



A) B)

C) D)

Figure 7. *Ti Grade 2 (A) Ultimate Tensile Strength, (B) Yield Strength, (C) Grain Size, and (D) Hardness Graphs.*

Figure 8. *Ti Grade 2 Ultimate Tensile Strength and Yield Strength.*

The effects of fully equiaxed alpha with intergranular beta grain structure are caused by different treated temperature on mechanical strength of Grade 5 titanium alloys. The strength of Grade 5 titanium alloys at different temperature treatment and their respective microstructure under optical microscope are shown (Figures 9-11). From the micrographs, when the Grade 5 Ti is heat treated for 10 minutes at 680°C, a fully equiaxed alpha with intergranular beta grain structure is formed. For the heat treatment at 850°C, the equiaxed alpha with intergranular beta grains have coarsened. In the micrograph for the 1000°C heat treatment, acicular alpha and intergranular beta can be seen to have formed.

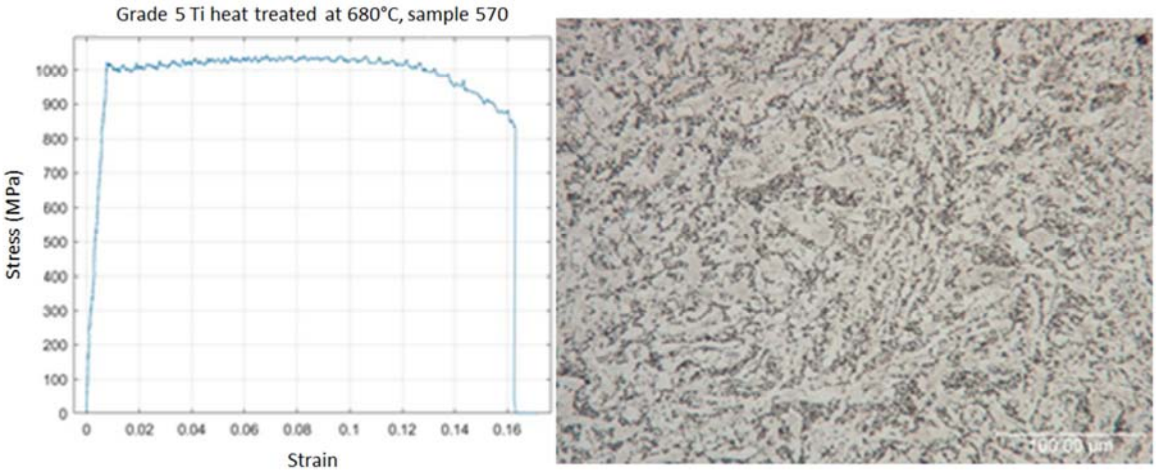


Figure 9. *Ti Grade 2 Ultimate Tensile Strength and Yield Strength.*

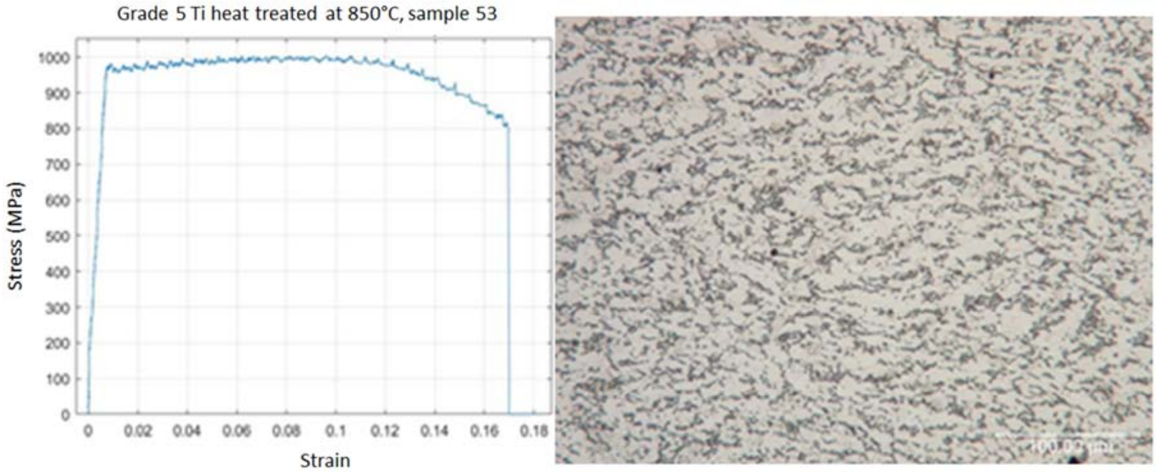


Figure 10. *Stress-Strain curve and micrograph (200x) of Ti Grade 5 treated at 850°C for 10 min.*

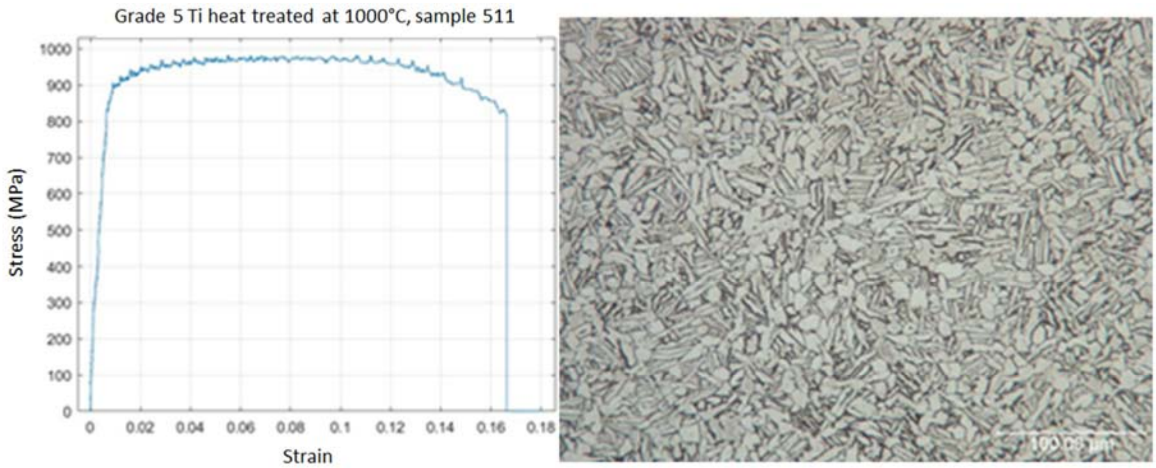
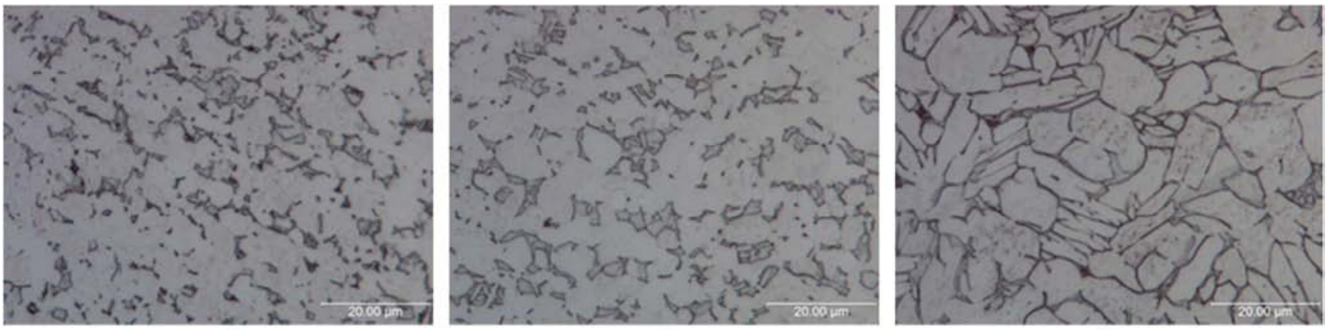


Figure 11. *Stress-Strain curve and micrograph (200x) of Ti Grade 5 treated at 1000°C for 10 min.*

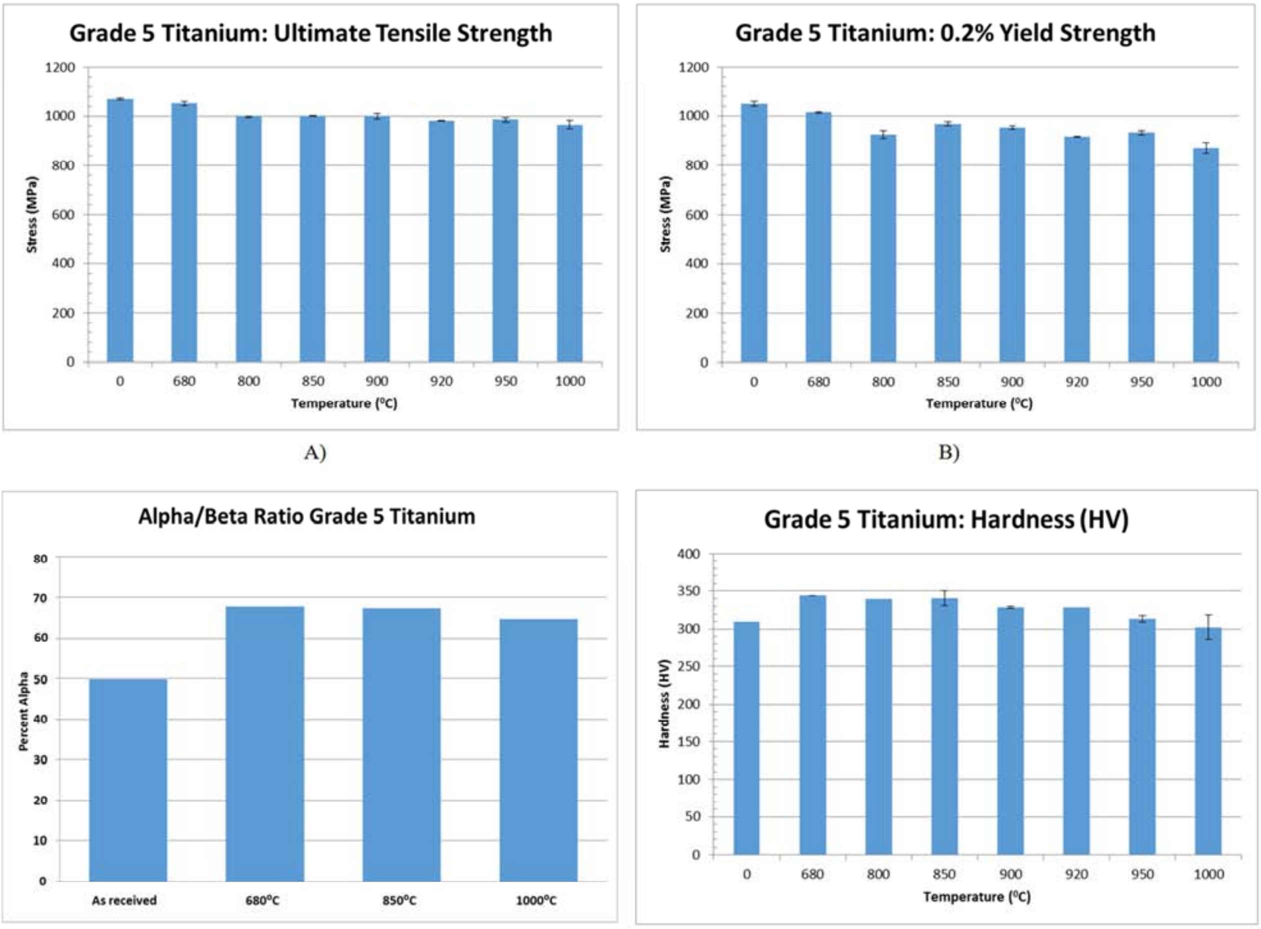
The Beta and alpha grain concentration were further investigated at a magnification of 1000x (Figure 12). The fully equiaxed alpha with intergranular beta grain structure can be seen in both samples from 680°C and 850°C heat treatment. It is also seen that there is some acicular alpha that is starting to form in the 850°C heat treatment sample. The samples heat treated at 1000°C can be seen to have a fully acicular alpha and intergranular beta microstructure.



A) B) C)

Figure 12. *Micrographs of Ti Grade 5 treated at (A) 680, (B) 850, and (C) 1000°C at 1000x.*

The change in properties of Ti Grade 5 subjected to varying heat-treatments is reflected in Figure 13. The ultimate tensile strength and yield strength follows a similar trend (Figure 14). The As-received sample has the highest ultimate tensile strength with a value of 1051.6 MPa and yield strength of 1013.8 MPa. However, for ultimate strength curve, it is noticed that the value is stable at 1000 MPa between 850°C-900°C. The hardness has a drastic increase with the initial temperature increase, and it steadily decreases above 680°C as grain size getting larger at elevated temperature. Table 2 shows the data from Ti Grade 5 (A) Ultimate Tensile Strength, (B) Yield Strength, (C) Alpha/Beta, and (D) Hardness in a numerical manner.



A) B)

C) D)

Figure 13. *Ti Grade 5 (A) Ultimate Tensile Strength, (B) Yield Strength, (C) Alpha/Beta, and (D) Hardness Graphs.*

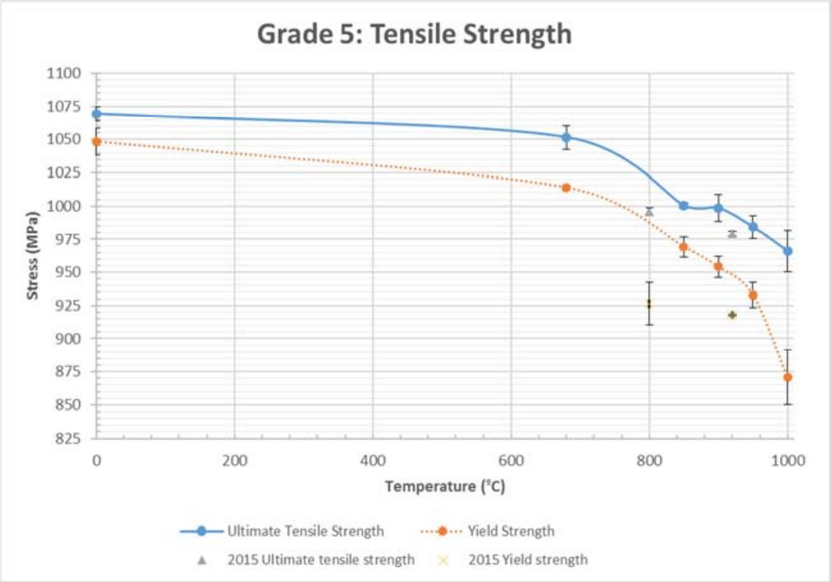


Figure 14. *Ti Grade 5 Ultimate Tensile Strength and Yield Strength.*

Table 2. *Ti Grade 5 Ultimate Tensile Strength, Yield Strength, Elongation, and Hardness Data.*

Temperature (°C)	UTS (MPa)	UTS Average	YS (MPa)	YS Average	Elongation (%)	Elongation Average	Hardness (Vickers)	Hardness Average
680	1042.7	1051.6	1016.0	1013.8	16.3	15.8	344.0	344.0
680	1060.4		1011.6		15.4		344.0	
850	1002.5	1000.5	976.8	969.2	17.0	16.7	331.0	340.5
850	998.5		961.6		16.4		350.0	
900	988.5	996.7	946.2	954.2	16.9	16.5	327.0	328.5
900	1008.8		962.1		16.0		330.0	
950	992.9	984.4	942.6	933.0	16.3	16.4	309.0	313.5
950	975.8		923.3		16.5		318.0	
1000	981.9	966.2	891.7	870.9	16.6	15.5	319.0	302.5
1000	950.5		850.1		14.4		286.0	

4. Discussion

Acicular alpha is when there is a high-volume fraction of primary alpha that the alpha grains start to interconnect [15]. It is suggested that the formation of acicular alpha is the cause for the decrease in strength when compared to other heat treatments.

In this project experiment, the beta transus for Grade 2 Ti was well below the 1000°C heat treatment in 10 minutes and little time for the recrystallization process was allows to be completed. Ultimately, the titanium was cooled during this recrystallization process causing it to form extremely large primary alpha grains. During the cooling, secondary alpha laths were formed in a basketweave formation. The large increase in primary alpha grain size is seen to have a negative effect on the strength of the titanium.

Regarding the Grade 5 titanium alloys, the coarsening is a suggested reason as to why there was a decrease in strength, when compared to the 680°C heat treatment. The As-received sample had the highest ultimate tensile strength and yield strength. Generally, they both decrease with an increase in temperature and can be explained by the same theory as mentioned for Ti Grade 2. It is still unsure that the exact reason of the value is stable at 1000 MPa between 850°C-900°C. The group determines that it can be resulted from grain size and alpha percentage change. Nevertheless, the reason for the jump is unsure as well and more data points falling in that region seem needed.

In the current research, the group didn't see much of change in alpha/beta ratio. Therefore, the exact reason for decreasing strength is unknown. If the group applies EBSD analysis on Titanium Grade 5 alloys, the average alpha grain size in the Titanium Grade 5 samples can be determined. Therefore, the reason behind strength decreasing can be better discussed. Also, the samples in current experiment were all heat treated for 10 minutes. If the group changes the brazing processing time, such as 30 seconds, the mechanical properties of samples treated in much shorter time could be determined. Furthermore, other brazing processes can also be explored such as thermal cycles using induction brazing or torch brazing. In that case, the effect of different brazing methods on Titanium samples could be seen.

5. Conclusion

Titanium alloys are widely utilized in industrial world because of its promising mechanical properties. Thus, it can be applied in aircraft frame, blade, vessel and so on. In this study, The effect of brazing on Grade 2 and Grade 5 titanium alloys were determined and the microstructures and mechanical properties of them after brazing were investigated by microscopy, tensile test. There was a very significant relationship between microstructures and mechanical properties because mechanical properties are normally determined by corresponding microstructures. Through our study, for the Titanium Grade 2 samples, the initial drop in strength is due to annealing. Around 800°C, alpha laths begin forming, and the strength begin increasing. The strength peaked at 850°C where the basketweave alpha laths are formed. Above that temperature, the grains sizes grew significantly which reflect a decrease in strength. For the Titanium Grade 5 samples, there is not much of a change in the alpha/beta ratio, so the decrease in strength cannot stem from that. It is expected to root from the increasing alpha grain size.

Controlling brazing temperature is a relatively easy way to control the microstructures of materials and doesn't need additional steps or processing which could potentially mess up the materials themselves because of potential unprofessional operation. Also, brazing treatment is not a complicated method and can be very easily done in industry and factory. Moreover, it is considered to be an economic method because less steps and processing are involved. Based on this study, people could produce titanium alloys with desired properties according to the requirement of performance.

Acknowledgements

This research used all of titanium alloys provided by Dr. Alex Shapiro.

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