Vacuum Brazing of Alumina to Titanium for Implantable Feedthroughs Using Pure Gold as the Braze Metal

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***Abstract*—Pure gold brazing of 96% alumina ceramic with CP titanium using electrical resistance heating has been analyzed. Wetting studies of gold with niobium and titanium were performed. HTCC alumina ceramic was metalized with a 500nm Nb layer yielding hermetic joints with Ti ferrule having leak rate of the order of 1.6×10^{-8} atm-cc/ sec. Wetting studies of Ti with Au and EDX analysis at the Au-Ti interface revealed that all the four Ti-Au intermetallic compounds forms at the interface and to avoid the growth of these intermetallic compounds brazing time should be kept minimum. Nb also shows good wetting behavior towards molten gold. Adhesion strength of Nb thin film over polished alumina substrate was measured using nanoscratch testing using a nanoindentor on 100nm Nb thin film on alumina. Scratch testing revealed that strong and adherent thin films of Nb over polished alumina can be deposited using sputtering.**

***Index Terms*—brazing, resistance heating, wetting, adhesion nano scratch testing**

I. Introduction

Joining of engineering ceramics such as alumina, zirconia silicon nitride and silicon carbide with different metals using different techniques have been studied by various researchers in the recent past. Some of the techniques used by these researchers are: active metal brazing, diffusion bonding, ultrasonic bonding, microwave joining, Solid-state ceramic–metal joining (with and without interlayers) [1]. The role of wettability during metal/ceramic joining has been studied by several researchers [2-5] and it is observed that the key property that leads to the success of a metal/ceramic joint is the ability of the interlayer to wet, spread and react chemically with the faying surface. However most of the available literature discuss about the active metal brazing, and although noble metal gold brazing has been used previously for implantable devices but the details of the process were not revealed.

One of the many promising applications of metal/ceramic joining is in biomedical implantable devices. Brazing of alumina ceramic to titanium metal using pure gold as the filler metal is evaluated in this paper. The binary phase diagram of gold and titanium [6] indicates 4 intermetallic compounds, Ti_3Au, TiAu, $TiAu_2$, and $TiAu_4$. Brazing should therefore be time limited to minimize the intermetallic formation, since two of the intermetallics have limited solid solubility and are therefore probably brittle. The formation of intermetallic phases has been shown by previous researchers during brazing of Ti with gold [7]. Presence of large amount of these brittle phases at the brazed joint interface is very deteriorating for the performance of brazed assembly. The second reason for rapid brazing is the fact that gold melts at 1064 ℃ while Ti undergoes allotropic transformation from α (HCP) to β (BCC) at 883 °C. The grain growth in beta phase titanium is more than an order of magnitude faster than in the alpha phase. If titanium is maintained at temperatures above the allotropic transformation temperature for sufficient time, then grain growth occurs, softening the metal [8-9]. Brazing using faster heating ramp rates, such as resistance heating, should allow processing to less than 30 seconds and minimizes grain growth.

II. Experimental

A. *Brazing of Ceramic Disk to Ti Ferrule and Au-Ti Interface Analysis*

96% alumina ceramic was prepared by standard high temperature cofired ceramic (HTCC) processing. Five (5) mils thick green tape was purchased from Maryland Ceramic and Steatite Co. (Street, MD). Isostatic lamination of stacked green tape was done using a Model IL-4004 (PTC) isostatic lamination press at 3000 psi and at 70 ℃. The disc was cut to initial green dimension using laser cutting. Since the alumina exhibited a 20% x-y shrinkage, the fired diameter was 313 mils after firing. The disc was centerless ground at Ferro Ceramic Inc. (Boston, MA) to 288 mils using a 800 grit diamond wheel.

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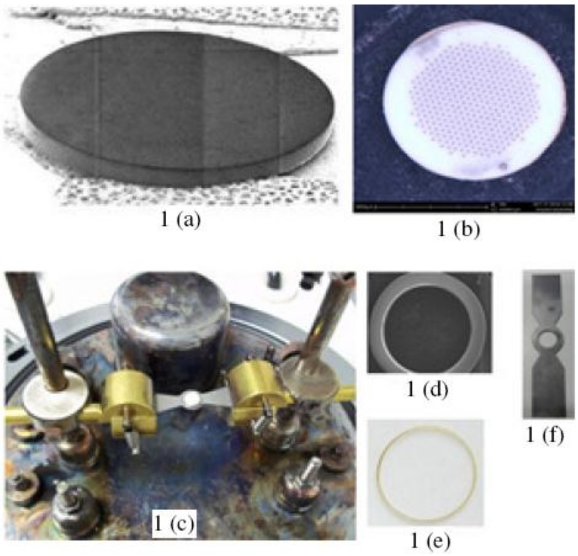


Figure 1. (a) Centerless grounded alumina disk without any vias. (b) Disk with 331 conducting vias. (c) Brazing setup inside thermal evaporator. (d) Ti ferrule. (e) Pure gold braze preform. (f) Ta electrode

A picture of the ground disk is shown above in Fig. 1 (a). Fig. 1 (b) shows an image of alumina disk with 331 conducting vias. In the present work ceramic disk without any vias was brazed to the Ti ferrule as the hermeticity of the brazed joint was tested and not of the vias. The disks were masked with kapton and 500nm thin film of Nb was sputtered on the ground edge to metallize the ceramic in order to improve the wettability of ceramic towards the pure gold braze filler metal, as pure gold does not wet alumina [10-12]. Approximately 5 mils in radial direction on each side of a diameter were left unprotected on one face, allowing a ridge of sputtered Nb along the lateral surface. After sputtering the masking is removed and the disk was cleaned.

The titanium ferrule shown in Fig. 1 (d) had a machined ledge onto which gold perform (Fig. 1 (e)) was placed. The alumina ceramic disk was placed into the titanium ferrule over the gold preform, due the tight tolerance of the centerless grinding, a gap of 2 mils was obtained (1 mil radially on both sides along any diameter). The gold preform wicked up the gap between the ceramic disk and the Ti ferrule due to capillary action when melting occurs.

Before brazing all the parts going to be brazed were ultrasonically cleaned in 3 step cleaning by isopropanol, methanol and Acetone in the same order for 5 minutes at each step. This whole unbrazed assembly is placed inside the hole of Ta electrode. An image of Ta electrode is shown in Fig. 1 (f). Ta electrode was machined out of a Ta sheet of 5 mils thickness. The hole at the center of the Ta electrode is 305 mils in diameter. Then this assembly was connected between the two high current, low voltage electrical connection posts inside the thermal evaporator as shown in Fig. 1 (c). Electrical connections were made by specially designed spring mechanism based clamps to allow some movement of the heating electrode to minimize bending while maintaining electrical contacts. Thermal evaporator was connected with an external DC power supply to control the power input using lab view software. Temperature measurements for the temperature rise and fall of the Ta electrode were made using a R type thermocouple. The tip of thermo couple was coated with high temperature alumina glue and was cured at 450 ℃ for 4 hours. This coating is required to avoid electrical connection between the Ta electrode and thermocouple. Fig. 2 shows the data obtained using lab view software was plotted with time.

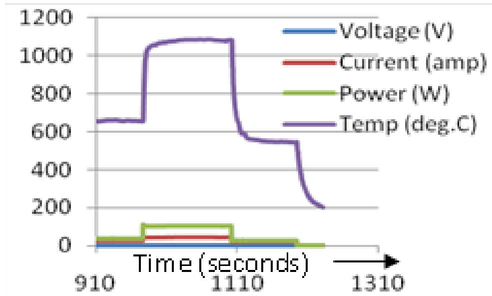


Figure 2. Variation of all the four variables with time in one plot

B. *Wetting Studies of Gold on Ti and Nb Metals*

Wetting angle determination experiment is performed using the JEOL Model JEE-4X thermal evaporator. A technique similar to sessile drop technique is utilized to determine the wetting angle of liquid gold on Ti and Nb. Experimental setup is shown in Fig. 3 (a). Ti and Nb electrodes were machined in the shape shown in Fig. 3 (b). The Ti electrode was 20 mils thick while, Nb is 4 mils thick. These electrodes are heated resistively very slowly and held at all incremental values of power for 50 seconds to stabilize the temperature until the gold melts on top of these electrodes The voltage increments was 0.1 volt each step. The experiment is performed in vacuum of 2×10^{-3} Pascal. The duration the liquid gold was kept molten at these heating electrodes were 1 seconds (just when the gold melts), 30 seconds, and 90 seconds. In case of Au-Ti system two additional data points were collected at 150 and 300 seconds to evaluate the effect of large intermetallic formation. For every individual time 3 samples are made. The samples were cross sectioned for analysis of reactions at the interface and formation of intermetallic compound which was critical for the Au-Ti interface. Temperature measurement is done by using a DFP 2000 disappearing filament optical pyrometer (Spectrodyne Inc.). Range 1 (760 ℃ to 1240 ℃) was used to determine the temperature of the electrode. The manufacturer’s claimed accuracy of the pyrometer for range 1 is 0.25% of reading.

Pure gold cylindrical samples of 99.95% purity, of approximately 1 mm diameter and 1 mm height were cut out from a wire of 1mm diameter and placed at the center of the heating elements as shown in Fig. 3 (a). The pyrometer was targeted at the center of the electrode concurrent with the gold sample. Current and power calibrations were performed first without any gold to determine the power required to increase the temperature at the center of heating element to 1100^{o}C. The purpose of wetting studies was to determine the best thin film for metallization of alumina ceramic. In the results section wetting angle θ is defined as the average of the angles θ_1 and θ_2 are which are defined as the contact angles measured at two diametrically opposite points of the solidified gold drop cross sectioned across its center.

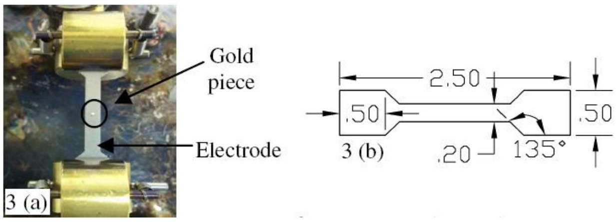


Figure 3. (a) Experimental setup for wetting studies. (b) Drawing of electrode (all dimensions are in inches).

C. Scratch Testing of Nb Thin Film over Alumina

Nanoscratch testing has been used successfully by other researchers in order to characterize the adhesion of sputtered thin film on substrates [13-15]. Nanoscratch experiment was conducted on sputtered thin films over polished alumina ceramic using Hysitron TriboIndenter TI 900 (Hysitron Inc., Minneapolis, USA). It has a horizontal capacitive transducer for applying normal load and two vertical capacitive transducers for measuring the lateral force experienced by the indenter during scratching. The Berkovich tip used for scratch testing the samples is in the form of a triangular pyramid with total included angle of 142.3 degrees and the radius of curvature of tip is 100nm. Thin film of Nb metals of thicknesses 60nm (sample number 5A) and 540nm (sample number 5D) were sputtered Alumina disks were polished before sputtering using 1 micron diamond suspension A picture of sputtered alumina disk is shown in Fig. 4.

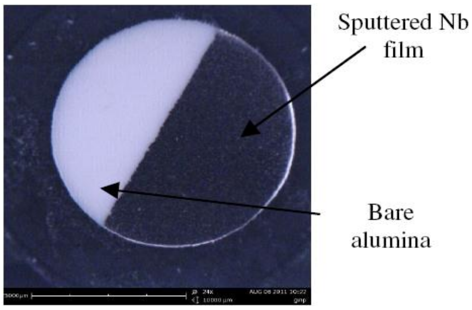


Figure 4. Scratch test disk with Nb sputtering and some bare alumina regions

Some area of the sample was masked to produce a bare surface. Scratched of lengths of 15 microns were made at across the interface. The vertical force was kept constant at 8000 μN. After the load has reached the set value, the indenter starts scratching the sample at a speed of 0.5 μm per second. When the scratch length has reached 15 μm, the load is released.

III. Results and Discussions

A. Brazed Assembly Hermeticity and Au-Ti Interface Analysis of Brazed Assembly

Fig. 5 (a, b) shows the front and back side of the brazed assembly. The brazed assembly was tested for hermeticity, a Varian vacuum technology, Model 979 series Helium Mass spectrometer leak detector shown in Fig. 5 (c, d)was used to test the leak rate of the brazed assembly hermeticity and a leak rate of the order of 1.6×10^{-8} atm-cc/sec was measured on Helium leak detector.

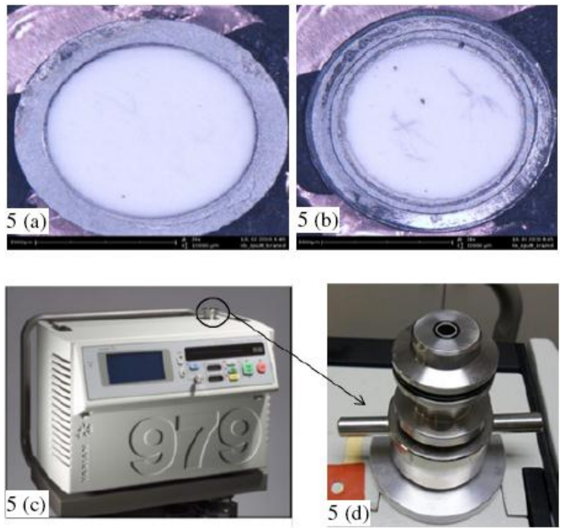
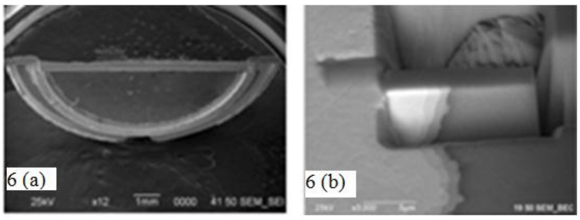


Figure 5. (a, b) Front and back side of the brazed assembly. (c, d) Leak detector and test port.

The brazed assembly was cross sectioned (Fig. 6 (a)) for microscopic analysis. High magnification SEM imaging across the Au-Ti interface shows layers at the interfaces of the intermetallic compounds between Au and Ti. Fig. 6 (b) shows SEM image of a FIB extracted sample. This sample was milled down to less than 100nm and then EDX analysis was performed at the Au-Ti interface (Fig. 6 (c)). This avoids the spreading of electron beam. Energy dispersive x-ray spectroscopic analysis shown in Table I shows a range of composition between Ti and Au and it suggest the presence of all the 4 intermetallic compounds that form in the binary phase diagram of Ti and Au as shown in Fig. 7.



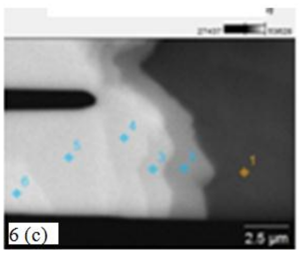


TABLE I.

	Ti-K	Au-M
Pt 1	93.36	6.64
Pt 2	75.50	24.50
Pt 3	54.28	45.72
Pt 4	34.97	65.03
Pt 5	20.63	79.37
Pt 6	7.92	92.08

Figure 6. (a) Sectioned brazed assembly. (b) FIB extraction of lamella at Au-Ti interface. (c) FIB extracted lamella and EDX point analysis shown in Table I.

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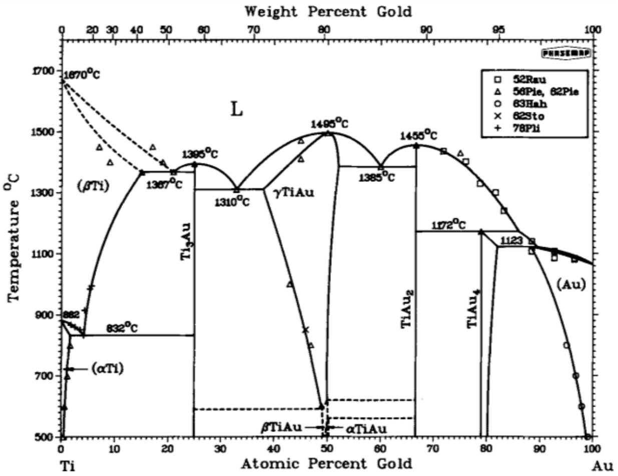


Figure 7. Au-Ti binary phase diagram.

A major problem was minimizing the thermal impedance between the titanium ferrule and the Ta heating element. The use of pressure on alumina ceramic disk while brazing was also tried. This technique reduced the brazing time considerably. The experimental setup is shown in Fig. 8. In this brazing cycle gold remain molten for only 39 seconds during brazing.

Figure 8. Use of pressure to reduce the brazing time

B. *Wetting Studies of Gold with Ti and Nb*

1) Au-Ti wetting studies

Gold exhibit very good wetting and spreading on Ti surface as can be seen from the measured value of wetting angle shown in Fig. 9 below.

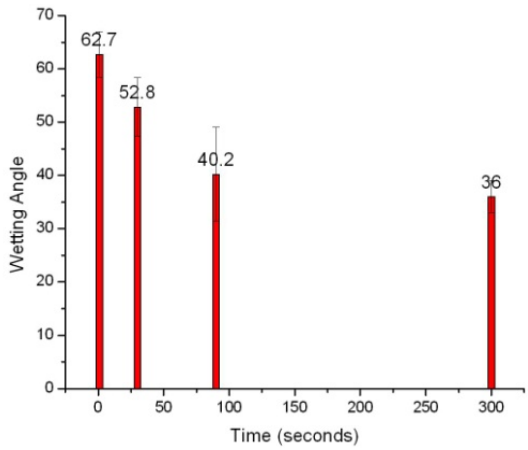
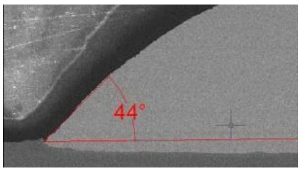


Figure 9. Change in the wetting angle with time at 1075±10 ℃

Au-Ti system shows reactive wetting [16] and dissolution of Ti substrate as shown in Fig. 10 (a) and Fig. 10 (b), along with the formation of intermetallic compounds of Ti and gold at the interface as shown in Fig. 11. Fig. 12 shows the Ti dissolution depth as a function of time of interaction of molten gold with Ti.



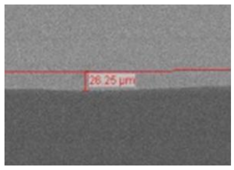


Figure 10. (a) Solidified drop end. (b). Solidified drop center

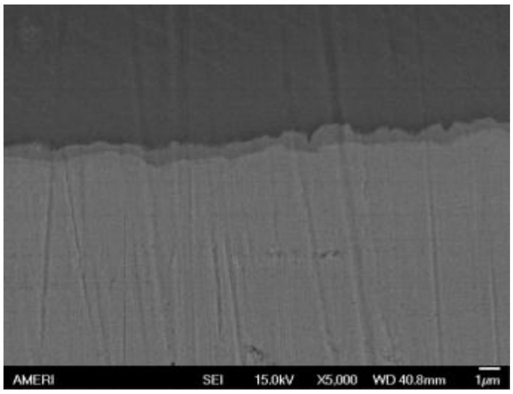


Figure 11. Sample Ti-90 sec-1st showing IMC formation.

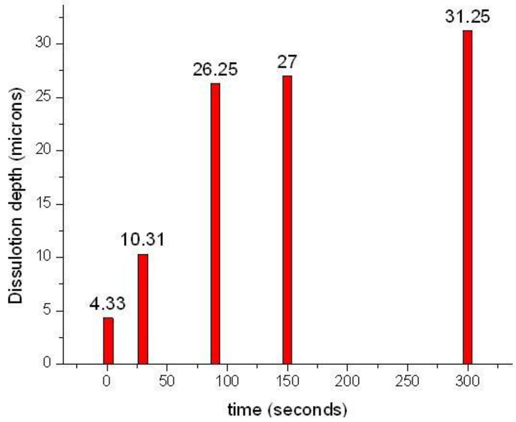


Figure 12. Graph showing variation of Ti substrate dissolution depth with time

a) EDX analysis at the Au-Ti interface of wetting sample

When the Au-Ti interface obtained during wetting studies were studied under SEM and EDX it was observed that some or all of these Intermetallic compounds shown in the binary phase diagram formed at the interface. Fig. 13 shows an image of the Au-Ti interface at which the EDX is performed. This sample corresponds to wetting studies for molten gold on titanium held for 287 seconds. The data obtained are shown in the Table II.

The Ti-300 sec samples where gold was kept molten over Ti substrate for 5 minutes were observed to fracture at the interface of gold and Ti and solidified gold drop popped out. An image of the popped out solidified gold drop and its impression over the Ti substrate are shown in the Fig. 14 (a) and Fig. 14 (b) respectively.

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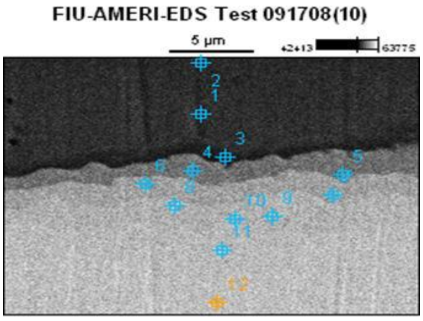


Figure 13. SEM image at 5000x where EDX point scan is performed

TABLE II. EDX ANALYSIS AT THE DIFFERENT POINTS SHOWN IN FIG. 12 SHOWING ATOMIC PERCENTAGES OF AU AND TI AT DIFFERENT POINT

	Ti-K	Au-M
pt1	93.73	6.27
pt2	95.37	4.63
pt3	91.32	8.68
pt4	74.35	25.65
pt5	72.19	27.81
pt6	55.92	44.08
pt7	48.76	51.24
pt8	44.25	55.75
pt9	32.64	67.36
pt10	37.09	62.91
pt11	18.78	81.22
pt12	8.97	91.03

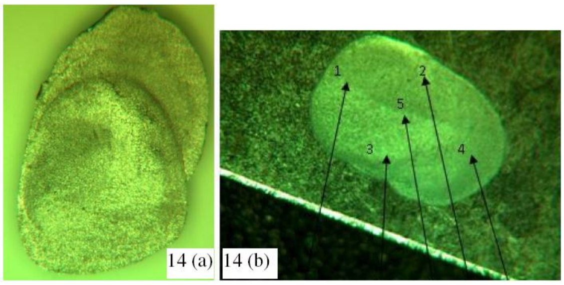
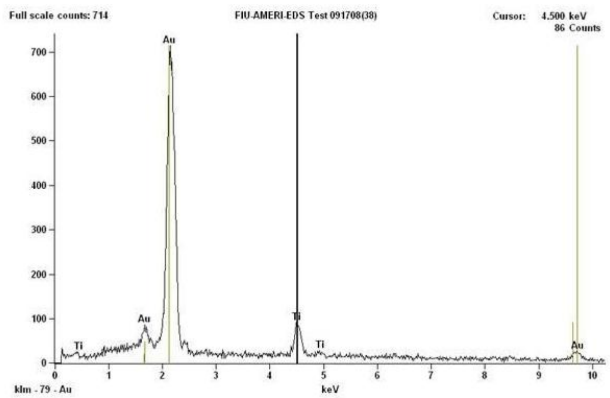


Figure 14. (a) Fractured gold drop. (b) Impression of fractured gold drop on Ti substrate and points where EDX is performed.



Quantitative Results FIU-AMERI-EDS Test 091708(38)

Element Line	Net Counts	Weight %	Atom %
Ti K	1149	10.46	32.45
Ti L	122	---	---
Au L	529	---	---
Au M	13711	89.54	67.55
Total		100.00	100.00

Figure 15. EDX spectra at five points

Fig. 15 shows the EDX spectra obtained at point 5 shown on Fig. 14 (b). The EDX data consistently shows the atomic ratio of Ti to Au as 1:2, suggesting the presence of $TiAu_2$ compound at the interface causing crack propagation and failure at the interface. All the five points showed the presence of only $TiAu_2$ compound with the atomic percentages in the range of ±5%.

2) Au-Nb wetting studies

Gold shows excellent wetting and spreading on Nb metal substrate as can be seen from the measurements of the wetting angle with time plot in Fig. 16. Gold also shows the Nb metal substrate dissolution and formation of layered structure probably of intermetallic phases at the interface of Au and Nb as shown in Fig. 17. Au-Nb binary phase diagram also shows formation of intermetallic compounds between these two [17]. Formations of intermetallic compounds like Au_2Nb_3 have been reported by Masahiro Kitada during heating of sputter deposited thin film of gold over Nb [18].

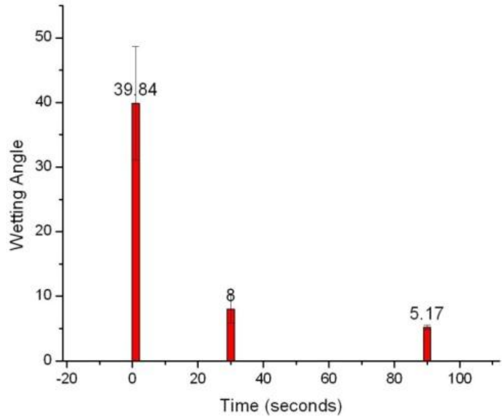


Figure 16. Change in the wetting angle with time for Au-Nb system

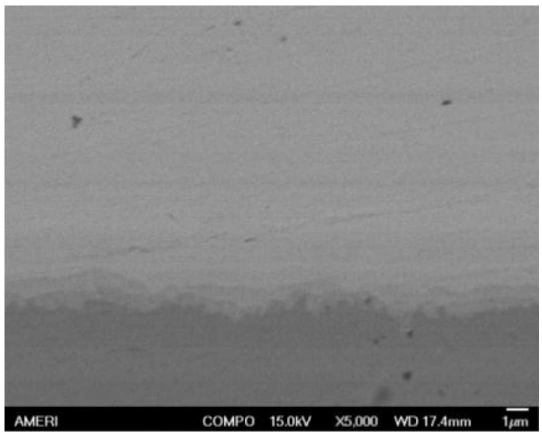


Figure 17. Au-Nb interface for Nb-90 secs-2nd sample

C. Scratch Testing of Sputtered Thin Nb Films over Alumina Substrate

Fig. 18 shows the lateral force vs. displacement plot corresponding to scratch 2 on the disk.

Table III shows adhesion strengths values obtained for the three samples tested.

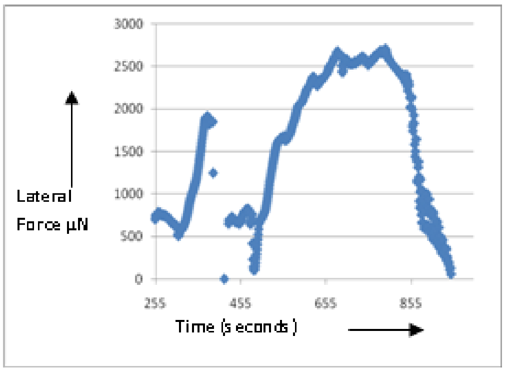


Figure 18. Lateral force (µN) vs. Lateral Displacement (µm) plot for scratch 2

TABLE III. ADHESION STRENGTHS VALUES OBTAINED FOR SAMPLE 5A

Sl. No	Indentor depth at the point of delamination (nm)	Lateral Area (nm^2)	Lateral Force for delamination (µN)	Adhesion Strength (MPa)
1	308	2456029	1550	631
2	177	811108	1950	2404
3	182	857580	1450	1691

The large variation in the adhesion strength observed which could be due to two reasons.

- There is variation in the thickness of sputtered thin film coating at different regions on the substrate.
- The surface of the alumina substrate is not perfectly smooth and there could be a scratch going underneath the coating which could have changed the adhesion strength.

The coefficient of thermal expansion (CTE) mismatch is another critical aspect which should be examined to achieve a successful ceramic/metal joint. CTE of some of the materials of interest for the present work are mentioned below in table IV. CTE mismatch of the mating surfaces should always be minimum as this can lead to stresses when the assembly is heated to the brazing temperature and also during subsequent cooling of the brazed structure. These stresses can make the joint weak and failure can occur at the interface [19].

TABLE IV. COEFFICIENTS OF THERMAL EXPANSION OF MATERIALS OF INTEREST

Element	Coefficient of thermal expansion ($X10^{-6}$) K^{-1}
96% Alumina	7
Nb	7.2-7.3
Ti	8.4-8.6
Ta	6.5
W	4.5-4.6
Au	14

IV. CONCLUSIONS

Resistance brazing of alumina ceramic to titanium metal using pure gold as filler metal was successfully achieved. Strong and hermetic brazed joints are obtained. Alumina was metallized to increase the wetting towards gold by sputtering a thin film of Nb metal of 500nm thickness.

The brazed assembly was tested for hermeticity and a leak rate of the order of 1.6×10^{-8} atm-cc/sec was detected.

Cross sectioning the brazed assembly confirmed that there are intermediate phase present at the interface of Au and Ti. EDX analysis of FIB extracted sample which was thinned down to avoid spreading of electron beam gave the elemental composition as a range of compositions between Au and Ti as per the binary phase diagram. Probably all the 4 intermetallic compounds present in the binary phase diagram of Ti-Au are present at the Au-Ti interface.

Wetting studies of gold with all the elements Ti and Nb were observed to be reactive wetting and dissolution of substrate was observed. Gold exhibit good wetting tendency towards Nb and Ti both. Since Nb exhibited very good wetting tendency towards gold and also the difference between the coefficients of thermal expansion (CTE) of alumina ceramic and Nb is least among the 4 metals mentioned in table IV, making Nb the right choice for metallization of ceramic.

In the Au-Ti system the sample in which gold was kept molten over Ti substrate for 300 sec. and air quenched showed fracture at the interface of Ti and Au. The facture surface was analyzed by EDX technique and $TiAu_2$ compound was detected at the fractured surface over Ti substrate. Effect of joint strength with the variation of brazing time on Ti-Au-Ti was also analyzed by the author and the results were published in ASM conference [20]. It was observed that the joint strength decreases drastically with increase in the brazing time.

Scratch testing at the polished Alumina/Nb interface yielded the adhesion strength values of sputtered thin film of Nb to alumina ceramic. It can be concluded that adherent sputtered Nb films over polished alumina surface can be obtained.

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