**35th Annual International Conference of the IEEE EMBS**
**Osaka, Japan, 3 - 7 July, 2013**

# Materials design considerations involved in the fabrication of implantable bionics by metallization of ceramic substrates

Sunil Patel[1], Thomas Guenther[1] *Member IEEE*, Christopher W.D. Dodds[1] *Member IEEE*, Sergej Kolke[1],
Karen L. Privat, Paul B. Matteucci[1] *Member IEEE* and Gregg J. Suaning[1] *Senior Member IEEE*

*Abstract*— **The Pt metallization of co-fired $Al_2O_3/SiO_2$ substrates containing Pt feedthroughs was shown to be a suitable means to construct implantable bionics. The use of forge welding to join an electrode to such a metallized feedthrough was demonstrated and subsequently evaluated through the use of metallography and electron microscopy. Metallurgical phenomena involved in forge welding relevant to the fabrication of all types of biomedical implants are discussed within this paper. The affect of thermal profiles used in brazing or welding to build implantable devices from metal components is analysed and the case for considered selection of alloys in implant design is put forward.**

## I. INTRODUCTION

Many commercially produced implantable bionics designed for use as an audible or visual prosthetic are constructed from electronics that are hermetically encapsulated in a metal and ceramic box. In such devices wires leading out from the electronics connect to the neurostimulating electrodes by passing through a dielectric feedthrough, which is typically ceramic. Therefore, the joining of ceramic and metal materials is of key interest in the development of next generation implantable bionics. Metallization of ceramic substrates by uni-axial hot press and forge welding (a solid state method for joining metals) were developed as methods to join ceramic and metal materials together for this purpose. The results have been metallurgically evaluated for the first time.

It is critical to consider thermal effects during brazing or welding in any manufacturing process. The same technique used to achieve forge welding can be used to join metals and ceramics [1]. This type of welding has advantages and disadvantages versus other methods such as arc, resistance or laser welding. For example, because melting is not necessary, forge welding can be carried out at a lower temperature than other techniques [2]. However, because the application of heat to the workpiece is much less localised than arc, resistance or laser welding [2], it is important to consider the affect of the thermal cycle on the other components in the assembly.

In the case of forge welding of small devices such as implantable bionics, usually the entire assembly is heated to the welding temperature. This means that any components in the assembly which are cold worked to attain an anisotropic micro-structure in their final shape, such as wires, foils or forgings could be annealed during the welding process. Annealing involves the nucleation and growth of new grains and the growth of existing grains. This process follows sigmoidal type kinetics described by the Johnson-Mehl-Avrami-Kolmogorov (JMAK) equation [3].

$$\chi = 1 - e^{\frac{-\pi}{3} N G^3 t^4} \tag{1}$$

Where $\chi$ is the fraction of material which has recrystallised, $N$ and $G$ are the nucleation and growth rate of new grains respectively and $t$ is time.

An annealed micro-structure is 'coarser' than the original, the result of which is a decrease in the yield strength, a phenomena known as the Hall-Petch relationship [3]. In an extreme case, annealing may remove micro-structural texture within such components, and the mechanical advantages of using cold worked materials in the design of the bionic device may be eliminated by the selection of forge welding in the manufacturing process. Consideration of this fact during process design and materials selection may help to identify and reduce the recurrence of failure modes in an implantable bionic device.

This paper presents an evaluation of the characteristics of forge welded materials suitable for building implantable bionics. Forge welding was utilised as a method to join a metal/ceramic electrical PCB type substrate containing high density feedthroughs to a large piece of foil. The foil was then laser micromachined into structures onto which an ASIC may be mounted, or into lead wires and tips forming a neurostimulating electrode array.

It is established that the inclusion of alloying elements in metals can increase the temperature at which recrystallization occurs and the slow the rate of grain growth during annealing [4]. Therefore, platinum-iridium alloy PtIr was investigated as an alternative to the use of pure platinum (Pt) for the electrode workpiece. This alloy also has better intrinsic mechanical[4] and neurostimulating[5] properties compared to pure Pt.

## II. MATERIALS AND METHODS

### A. Metallization and Welding

In this study 16mm diameter, 650$\mu$m thick $Al_2O_3/SiO_2$ (Keral 96, Kerafol, Germany) ceramic discs were used as test structures to represent ceramic/metal feed-throughs. The discs were metallized with 12.5$\mu$m thick platinum foil (99.95%, Goodfellow, UK) in dense graphite tooling with

---

*This research was supported by the Australian Research Council (ARC) through its Special Research Initiative (SRI) in Bionic Vision Science and Technology grant to Bionic Vision Australia (BVA)

[1] Graduate School of Biomedical Engineering, University of New South Wales, Sydney, Australia. `sunil.patel@unsw.edu.au`

978-1-4577-0216-7/13/$26.00 ©2013 IEEE

graphite foil (Permafoil 200HF, Toyo Tanso, Japan) placed between the sample and tooling as a sacrificial contact material. The samples were then heated to 1200°C under uni-axial compression of 5MPa in a vacuum furnace (FR210, OxyGon Industries Inc., USA) for a period of 4 hours. After metallization, residual graphite on the surfaces of the components was first removed by mechanical abrasion before finishing by combustion in air whilst the sample was heated to 800°C for 15 minutes in a muffle furnace (102C, Scientific Equipment Manufacturers South Australia, Australia).

The metallized discs were then forge welded onto a 25$\mu$m thick foil of Pt (99.95%, Goodfellow, UK) or PtIr (10% Ir, Goodfellow, UK) by isothermally heating under a uni-axial compression of 4MPa for 30 minutes. Fig. 1 shows this arrangement. The components were pressed together on top of a full dense high purity alumina plate (Ceramic Oxide Fabricators, Australia) that had been prepared with a spray of graphite release agent (Henkel Technologies, Australia). Post welding, residual graphite was removed in the same way as after metallization.

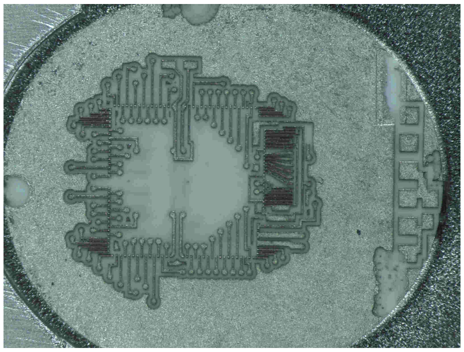

Fig. 1. Forge welded feedthrough/electrode assembly for construction of an implantable bionic device. The metallized surface on top of the ceramic feedthrough was micromachined to match to the electronic components. The metallized layer on the otherside of the feedthrough is welded to a sheet of Pt which can be micromachined to form a neurostimulating electrode array.

### B. *Characterisation*

*1) Metallography:* After Pt metallization, forge welding of foil onto the metal surface and removal of any residual surface carbon, the samples were mounted in epoxy and cut in half using a diamond blade. One half of the samples were prepared for metallography as described by Piotrowski and Accino[6]. The cross section of the samples were polished to a 1$\mu$m finish. Next, the samples were immersed in concentrated HCl with 1g/mL NaCl and electrolytically etched at 6 V AC for 30 seconds. It was important to control the etching time so that the grain boundaries in the metal were sufficiently visible in the microscope without over-etching and removing too much of the polished surface. Fig. 2 shows the apparatus used to achieve an electrolytic etch. Once etched, the samples were examined using an inverted light microscope.

*2) Electron Microscopy:* Samples were cut as for metallography before being polished to a 0.25$\mu$m finish using a diamond suspension and micro-cloth. The samples were then sputter coated with carbon in preparation for examination in a Field-Emission SEM-EPMA (JXA-8500F, JEOL, Japan) running a 20kV, 40nA beam.

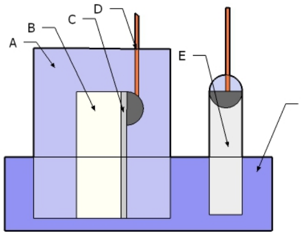

Fig. 2. Schematic of the apparatus used to electrolytically etch metallographical cross-sections of welded Pt components. The sample cross section (B) was mounted in epoxy (A). A wire (D) was soldered onto the Pt metallized surface (C). The cross section was then dipped in etchant (F) and polarised against a Pt counter electrode (E).

*3) Hermeticity:* Metallized layers of Pt on the $Al_2O_3/SiO_2$ substrates were tested to determine whether the interface was hermetic and prevented the passage of helium. This was achieved through the use of a mass spectrometer (Smarttest, Pfeiffer, Germany) to detect penetration of He through the interface using a method similar to that of Guenther et al[7].

## III. RESULTS

### A. *Pt - $Al_2O_3/SiO_2$ Metallization*

Mass spectrometry measurements showed that this method produced samples that were He leak tight through interfaces as narrow as 1mm. A cross section of the metallized layer joined to a conductive feedthrough within the substrate is presented in Fig. 3. The cross section shows that the metal content in the forged-on layer is higher than within the feedthrough. Continuity measurements indicated that the metallized layer was electrically connected to the conductive pathways within the feedthrough.

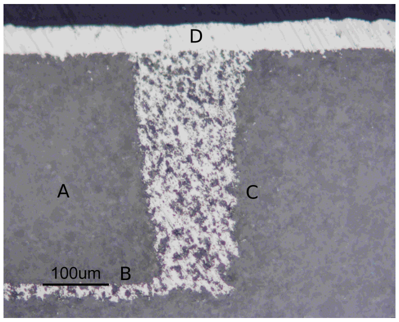

Fig. 3. Cross section of Pt metallized layer (D) joined onto a HTCC glass ceramic substrate (A) containing a co-fired Pt via (C) which feeds through the substrate with PCB type horizontal co-fired Pt tracks (B).

### B. *Forge Welding of Pt*

As expected, the micro-structure of the metal components in the assembly were found to be significantly affected by the thermal profile used to achieve a forge weld.

Fig. 4 shows metallographic sections of the workpieces before and after welding. In the foil sectioned after rolling and before annealing, there existed grain boundaries with an in plane orientation, whereas after the foil has been forged to the substrate the grain boundaries are mostly oriented perpendicular to the surfaces of the sheet. Very thin grains are visible at the interface between the two pieces of metal.

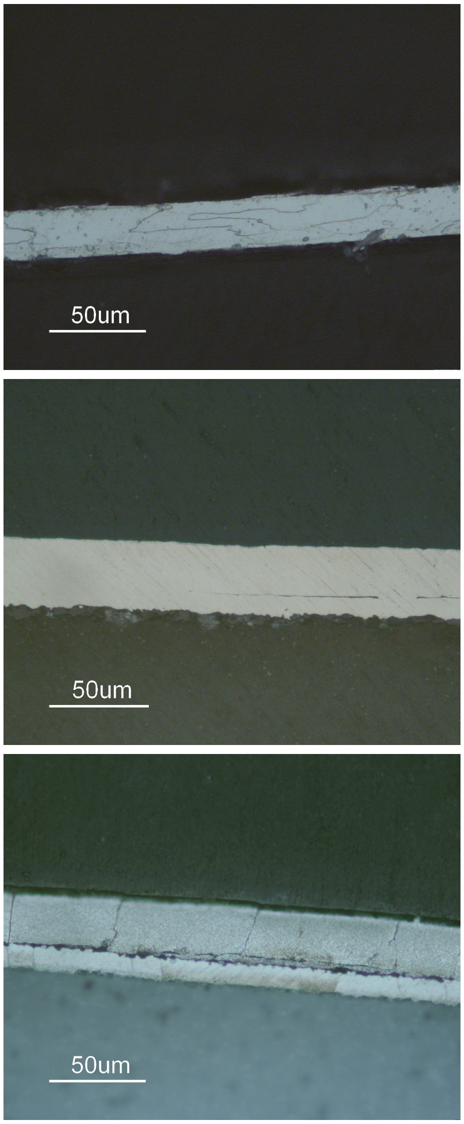

Fig. 4. Metallographic cross sections of Pt foil before (top) and after (bottom) forge welding to metallized substrates. The image in the centre is of the welded assembly before metallographic etching to elucidate the grain boundaries. Clearly, the microstructure of the workpieces is significantly changed by the thermal profile used to weld them.

During sample preparation, it was very difficult to achieve a flat and polished Pt metal surface without locally removing the metal layer from areas of the metallized Keral 96 sample and exposing the ceramic substrate underneath. In all samples sectioned areas were found where a weld did not form, most likely because contact was not achieved between the metallized surface and the larger sheet of Pt.

### *C. Forge Welding of Pt and PtIr*

Welding of Pt to PtIr alloy was found to be very similar to that of Pt to Pt. Fig. 5 shows a cross section of such a weld. The weld line between the two foils that have been forged together was visible due to the persistence of very fine pores after forging. Fig. 6 charts the chemical analysis of Ir across the weld line. This data was obtained through wavelength-dispersive spectroscopy in an electron microprobe (EPMA). The gradient of Ir concentration across the weld line was less when the forge weld was made at higher temperatures.

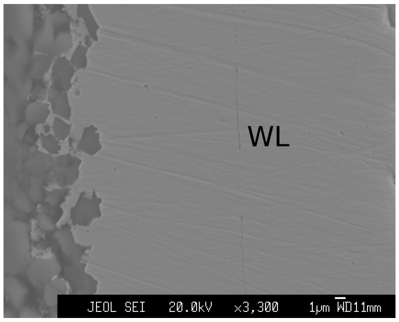

Fig. 5. SEM cross section of a Pt-PtIr joint, forge welded at 900°C for 30 min with 4 MPa of contact pressure. The ceramic substrate is visible at the left of the image. The interface between the welded metals is labelled WL. This image shows that long, high temperature dwells are required to eliminate porosity at the weld interface.

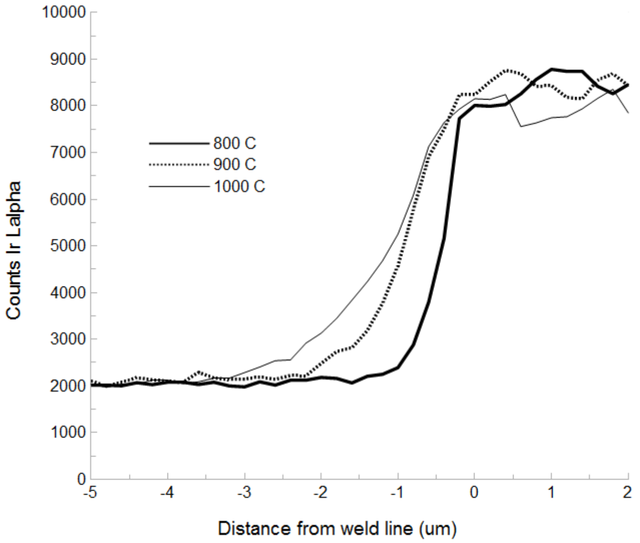

Fig. 6. EPMA results showing the Ir composition across forge weldments made at temperatures ranging from 800 to 1000°C. The x axis indicates distance from the interface between the Pt layer metallized onto a ceramic substrate and the PtIr alloy welded to it. The gradient of the Ir concentration decreased as the temperature of welding increased, this shows that the amount of diffusion was greater at higher temperatures.

## IV. DISCUSSION

The results of characterisation show that the microstructure of the metal components is affected by the thermal profiles used to join components. Therefore it is important to consider these changes in the conceptualisation of manufacturing processes for implantable devices or orthopaedics and develop strategies for minimising undesirable changes.

Pt - $Al_2O_3/SiO_2$ metallization results corresponded with reports in the literature of good bonds being achieved between Pt and high purity $Al_2O_3$ using similar bonding parameters [1]. The bulk metal on the surface of the feedthrough made the interconnection of wires and ICs through resistance and laser welding techniques less difficult than to co-fired Pt paste surfaces. In the case of the Pt-$Al_2O_3$ system; resistance, laser or forge welding and brazing can be carried out in air, eliminating the need for inert gas jets or elaborate vacuum chambers because Pt does not oxidise readily [8], [4].

Another very important advantage yielded by metallizing the surface of the feedthrough is that the structures on the surface can be micromachined after the feedthrough is sintered. This means that any distortions in the geometry of the feedthrough due to uneven shrinkage can be compensated by adjusting the dimensions of the final micromachining step.

Savitskii [4] reported that, depending upon the purity of the Pt, the temperature to elicit recrystallization within it can be as low as 300°C. Therefore, it was not unexpected to observe recrystallization during the thermal profiles required to form forge-welds. The very fine grains visible at the interface in Fig. 4 are likely to have nucleated during the welding profile. It is not likely that these are fine pieces of swarf that were embedded in the foil during the rolling of it to final thickness. If very fine swarf was present, there would be a significant reduction in the free energy of the system if it were consumed by growing grains. This would reduce the total amount of grain boundaries and the energy associated with them, and so it is likely that if any swarf were present it would have been consumed during grain growth.

The presence of small grains; which are likely to have developed by spontaneous nucleation, shows that it is important to consider the JMAK kinetics when optimising a forge weld. If it is possible to stay in the nucleation phase of annealing by forging for short durations at high temperature, it may be possible to complete a weld and maintain a relatively fine grained micro-structure within other components. Similarly, if the components are seeded with nucleating agents such as ceramic powders before the forge weld, it will increase the nucleation rate during welding and help to achieve a fine micro-structure. Nucleating agents are established in the metal casting industry as a method to refine solidified micro-structures [3]. If the components are made from alloys, it is likely that the growth rate of grains will be lower during annealing [4].

The implications of the coarsening in the micro-structure shown in Fig. 4 were that the foil workpiece would have a lower yield stress than before the welding process due to the Hall-Petch effect. Also, less grain boundaries were present post-welding with a favourable orientation to blunt the tips of cracks growing through the sheet [9]. This coarsening can be expected to make the material effectively less tough after exposure to the heat cycle during forge welding.

Results from Pt-PtIr welding show that increasing the temperature of the weld process resulted in greater diffusion of Ir from the alloy across the weld line into the pure metal (see Fig. 6). Because Pt and Ir form a full solid solution binary system [4], achieving diffusion of the alloying element across the weld line indicated that a join has formed which is free from layers of brittle intermetallic compounds and likely to be strong. Therefore, PtIr alloy is feasible for use in implantable bionics as an electrode array joined to components by forge welding. Because the doping of metals with alloying elements reduces the rate of recrystallization [4], and because it has a higher charge injection limit than pure Pt [5], PtIr may be an astute choice of alloy for neurostimulating components joined to electronics within implantable devices through forge welding.

## V. CONCLUSIONS

Through the application of uni-axial hot press bonding, Pt metallized onto $Al_2O_3/SiO_2$ substrates can enable interconnection of feedthroughs, ICs, electrodes and wires. The Pt $Al_2O_3/SiO_2$ joint was found to be He leak tight and so could be exploited to form encapsulations over electronics components in implantable bionics.

Metallographical sections of Pt-Pt forge welds showed that the micro-structure changed significantly during the welding process. Pt and PtIr alloys were found to join by forge welding under very similar conditions to that of Pt and Pt. These results show that materials design needs to be considered when conceptualizing the manufacture of implantable bionics. The convenience of using a forge technique in the manufacturing process must be weighed against the effect of the thermal profile upon the materials design of the device. Similarly, the selection of alloys must be targeted toward finding materials that are effective in application as well as being practical to integrate into manufacturing processes.

## REFERENCES

[1] R. Allen, F. Bailey, and W. Borbidge, "Solid state bonding of ceramics with platinum foil," *Platinum Metals Review*, vol. 25, no. 4, pp. 152 – 155, 1981.

[2] R. Tylecote, *The Solid State Welding of Materials*. New York: St. Martin's Press, 1968.

[3] R. Reed-Hill and R. Abbaschian, *Physical Metallurgy Principles. Third*. Boston: Boston: PWS Publishing Company, 1973.

[4] E. Savitskii, *Physical Metallurgy of Platinum Metals*. Moscow and New York: Mir Publishers, 1978.

[5] A. Petrossians, J. Whalen, J. Weiland, and F. Mansfeld, "Surface modification of neural stimulating/recording electrodes with high surface area platinum-iridium alloy coatings," in *Engineering in Medicine and Biology Society,EMBC, 2011 Annual International Conference of the IEEE*, 2011, pp. 3001–3004.

[6] T. Piotrowski and D. J. Accino, "Metallography of the precious metals," *Materials Characterization*, vol. 39, no. 25, pp. 299 – 344, 1997.

[7] T. Guenther, C. Dodds, N. Lovell, and G. Suaning, "Chip-scale hermetic feedthroughs for implantable bionics," in *Engineering in Medicine and Biology Society, EMBC, 2011 Annual International Conference of the IEEE*. IEEE, 2011, pp. 6717–6720.

[8] J. Chaston, "The solid oxides of platinum and rhodium formation in high pressures of oxygen," *Platinum Metals Review*, vol. 13, pp. 28–29, 1969.

[9] D. Broek, *Elementary Engineering Fracture Mechanics*. The Hague: Martinus Nijhoff Publishers, 1982.