Densification and Bonding of Copper and Aluminum Powders in Ultrasonic Powder Consolidation

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ABSTRACT

Ultrasonic powder consolidation (UPC) is a novel rapid, full-density powder consolidation process in which metal powders confined in a die under uniaxial loading is subjected to ultrasonic vibration at low temperature for a few seconds or less. In this research, copper powders with dendritic and spherical morphologies and an aluminum powder with spherical morphology were subjected to UPC under various conditions to investigate the effects of the process variables on the densification and metallurgical bonding of the compact. An ultrasonic washing test, developed in this research, was used to determine the extent of densification and bonding achieved in specimens consolidated under systematically varied UPC conditions. Hardness testing was also employed as a supplementary means for the assessment of compact density in both as-consolidated and ultrasonically washed states. The degree of metallurgical bonding was also qualitatively assessed from the fracture surfaces of manually broken specimens.

With the dendritic and spherical copper powders, the minimum consolidation temperature, time and uniaxial pressure required for nearly full densification were determined to be 450 °C, 4 s and 84 MPa, respectively. The best conditions were 500 °C, 4 s and 100 MPa for both copper powders. Dendritic-powder specimens exhibited better densification and bonding than spherical-powder specimens above 450 °C. However, below 450 °C, the spherical powder produced better results, due probably to its higher packing geometry and repacking under the ultrasonic vibrations. With the spherical
aluminum powder, specimens with best densification and bonding were obtained under the conditions of 400 °C, 2.5 s and 80 MPa.

Compact densification generally requires a good amount of powder deformation. In UPC, this occurs very quickly in a fraction of a second if consolidation temperature is sufficiently high. However, a high degree of compact densification does not necessarily assure good metallurgical bonding. Bonding must be preceded by the formation of nascent metal-to-metal contact of powder particles, which requires good compact densification. Thus, densification is only a necessary condition for bonding. For the copper compacts, metallurgical bonding, at a given temperature, increased with increasing consolidation time, indicating that bonding is a thermally activated (diffusional) process. Densification of dendritic powder produced more fresh metal surface and hence higher degrees of bonding. The bonding in the aluminum compacts, however, followed densification more closely, reflecting the higher homologuos temperatures (up to 0.72 of the melting point in K).
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1. INTRODUCTION

1.1 Overview

Ultrasonic welding (USW) is a solid-state joining process that facilitates rapid-joining of similar or dissimilar materials at room temperature. USW has been used extensively in industry for various applications like electronics, semiconductor, and automotive industries for rapid joining of metal wires, cables, and sheets [1]. Ultrasonic powder consolidation (UPC) [2] is a USW technique that applies to the consolidation of metallic powders. Conventional techniques for the consolidation of powders generally require a two-step process, cold compaction and sintering. The first step forms a green compact which usually has a high amount of porosity, and the second step facilitates compact densification and metallurgical bonding which requires heating the compact to a high enough temperature, generally well above half of the melting point of the material on homologous scale. Previous research on vibratory compaction resulted in green compacts that require subsequent sintering [3-5]. However, it has recently been shown that UPC can create fully dense Al consolidates without subsequent sintering within a fraction of a second [6]. Another recent research on applying UPC to the consolidation of Cu and Cr powder mixtures has shown that fully dense Cu-Cr composites could be produced at low pressures and temperatures in a short vibration time [7]. As a result, this new technique, UPC, has shown its capability on powder consolidation and further work
is required to find a processing window for the full consolidation of metal powders and also to test the quality of consolidated materials.

1.2 Objectives

This study focused on the consolidation of Cu and Al powders with the objectives listed below:

- Consolidation of Cu powders with dendritic and spherical morphologies by UPC under various conditions.
- Consolidation of Al powders by UPC under various conditions.
- Determination of the processing window for full metallurgical consolidation of all Cu and Al powders.
- Analysis of the microstructure of produced specimens by optical microscopy and scanning electron microscopy.
- Analysis of produced specimens in terms of porosity, hardness and ductility by ultrasonic washing tests, Vickers micro-hardness tests and bending tests respectively.
2. BACKGROUND

2.1 Powder Consolidation Techniques

Powder metallurgy (PM) is a widely used technique to produce metal products and metal composites. Generally, it consists of four major steps: (1) production of powders, (2) blending or mixing, (3) pressing powders into green compacts which still contain porosity, (4) formation of a bulk material by a consolidation process. In a conventional method, green compaction is normally achieved by cold pressing, while consolidation is obtained by sintering at an elevated temperature. Another widely used method is hot isostatic pressing (HIP) in which powders vacuum-encapsulated in a metal container is held under isostatic pressure at an elevated temperature for a sufficient amount of time which depend on the material and selected temperature. Generally, a complete cycle of HIP takes 12 to 24 hours for full consolidation at 80% of the absolute melting temperature [8].

Since the compaction and subsequent sintering processes are time consuming, there is a vast amount of research in the field of high strain-rate processes [9-21] conducted to minimize the processing time. However, these methods still require the powder to be compacted prior to deformation processing. The most commonly employed high strain-rate powder consolidation techniques are powder forging, rolling and extrusion, equal channel angular pressing (ECAP), and shock wave consolidation. Although, materials produced by these techniques can generally achieve more than 99% density, they may still need to be further processed in order to improve mechanical properties.
Extrusion is generally a two-step process where the powders are first cold pressed into a can and subsequently extruded at room temperature or an elevated temperature after evacuation and sealing. Many studies have been done on the extrusion of Al and Al alloy powders at temperatures from room temperature to 773 K [17-19]. By conducting cold isostatic pressing at 350 MPa and following hot extrusion at 773 K together, Lee et al. [17] obtained full density Al-Le alloy composites.

The shock wave consolidation process produce a bulk material by passing a shock wave through the powder and the necessary shock wave is generated by detonating an explosive. In 1991, a review published by Rosato, Vreeland, Jr, and Prinz [14] summarized various studies covering this technique. Jin Yuan et al. [21] consolidated Al-Li alloy powders by this method and they concluded that the shock wave could break up the surface oxide layer and resulted in compacts with densities above 98% while preserving the initial microstructure of the powders. However, they also stated an abnormal softening in comparison with the original powders.

2.2 Ultrasonic Welding of Metals

As a solid-state joining process, USW produces metallurgical bonding by the application of high-intensity vibration in the ultrasonic frequencies when used for metals, combined with normal compression of the materials [22]. The friction forces between the parts or powders, sonotrode, the anvil, the die and the punch prevent the slippage, such
that the vibratory energy is efficiently transmitted and dissipated at the weld interface by high frequency scrubbing action. This highly localized interfacial slip initially causes disruption and dispersion of surface films like oxides, permitting fresh metallic contact and formation of micro-welds shown in Figure 2.1 [23].

Further processing results in the coalescence of micro-welds until a fully developed weld zone is realized. Continued application of the vibration causes high strain-rate elastic-plastic deformation, generating modest heating with a transient maximum at 35-50% of the melting point [21]. Furthermore, these highly localized high strain rate shear strains have been shown to create large concentration of excess vacancies in the parent metals [24-27] and high dislocation densities [24, 28]. This creates a fine-grained cold-worked structure, recrystallization, phase transformations and atomic diffusion across the bond [29-31].

An extraordinary feature of USW is its capability for both monometallic and bimetallic welding [32] like metal bonding to polymers and ceramics [33-36]. In particular most metals, including aluminum, copper, nickel, titanium, iron and steel and precious metals and most of their alloys can be readily welded to themselves and to others. Another advantage of USW is its short welding time and limited pressure and temperature, preventing damage to plastics and semiconductors, as well as joint deformation and residual stresses. As a result, ultrasonic bonds which are made properly exhibit shear strength, hardness, high temperature behavior and corrosion resistance comparable to those of normal materials [22]. In addition, weld quality is not sensitive to
original materials and surface conditions like oxide films and coatings, and usually requires no protective atmosphere. Finally, there is no need for special health, safety precautions and environmental hazards [37].

The first studies and publications on ultrasonic welding appeared in 1955 and 1956 [38], where the design of ultrasonic welding equipment for the welding of aluminum, copper, steel and some alloys and along with initial metallurgical studies were presented. In the early 1960’s after the establishment of the USW equipment, researchers were able to focus on the fundamentals of the joining mechanism. Jones et al. published a phenomenological treatise in which the weldability of aluminum, copper, nickel and stainless steel was investigated [39]. In 1965, another treatise on USW was published [40] where the formulation of contacting and oscillating spheres [41], which was verified by the experimental results in Ref. [42], was used to estimate the energy dissipation during ultrasonic cycles. According to Refs. [43, 44], it was proposed that adhesion first occurred at micro-slip regions at the interface, followed by rapid temperature rise produced by energy dissipated through hysteresis, which resulted in softening, plastic flow, enhanced interfacial diffusion and increase in total micro-weld areas.

The effect of high intensity ultrasound on the deformation of metals was reported in 1966 and 1970 [45, 46]. Standard tensile test samples of aluminum, zinc and steel were tested while ultrasonically vibrating the samples at varying degrees of intensity. The results showed immediate reductions in the elastic modulus and yield strength of the materials during the application of ultrasonic vibration when a threshold intensity was
reached. Another threshold intensity was found above which permanent strain hardening occurred. However, the calculations indicated that the acoustic stresses were not sufficient to cause dislocation glide and subsequent hardening.

The first comprehensive analysis of wire bonding [26] investigated the effects of clamping load on bond quality and noted that the weld strength increased up to a certain degree by bonding, but started to decrease after the wire degraded on the perimeter of the weld area, therefore giving the first explanation to the widely observed trend. The motion of the welded parts was observed by a laser interferometer and high-speed recording techniques. The results indicated that in good quality welds, little relative motion was observed between the parts.

Simultaneous measurement of interface temperature and inter-diffusion was first performed by Kulemin et al. [47] for aluminum copper pairs. The concentration profiles obtained by electro micro-probe analysis were related to diffusivities. The results indicated diffusion enhancements by seven orders of magnitude and it was considered that this was due to alternating stresses and strains.

Enjo et al. [48] investigated the effect of ultrasonic vibration on the diffusion welding of aluminum and they observed that bond strength increased significantly due to oxide breakup at the interface.

In recent studies, Yadav [49] and Doumanidis and Gao [50] focused on the modeling of USW, where finite element methods were employed to predict strain fields around the weld interface, which were verified with strain measurements. In 2005, the study result
from Gunduz et al. [51] reported enhanced inter-diffusion and melting point depression during elevated temperature USW of aluminum zinc pairs. They provided the first thermodynamic model that included the effects of excess vacancies induced by high strain rate deformation during USW on solid phase stabilities.

2.3 High Rate Plastic Strain Effects

Since the majority of ultrasonic metal studies are concerned with enhancing weld formation and optimizing processing parameters for industrial welding applications, most welding is performed at room temperature. As a result, most of the literature describes ultrasonic metal welding as a low temperature, solid-state joining process that creates metallurgical bonding without melting. However, there are varying opinions about the fundamental mechanisms of ultrasonic welding of metals. For example, some of them indicate that enhanced interdiffusion and local melting may occur during ultrasonic welding which can be attributed to high strain rate plastic deformation and increase in vacancy concentration \((X_v)\). Non-conservative motion of jogs on screw locations can produce increased \(X_v\) that has a drastic effect on the thermodynamic stability of the solid solutions [51]. In 1966, Langenecker performed tensile tests to study the effects of ultrasound on deformation characteristics of metals [45]. In this literature, Langenecker discussed two pronounced effects of ultrasound on metals: acoustic softening which occurs during application of ultrasound is an apparent reduction in shear stress necessary
for plastic deformation and subsequent acoustic hardening which is observed after ultrasound application. Langenecker first described the mechanism of ultrasonic softening as acoustic energy being absorbed at dislocation sites, increasing dislocation amplitudes until freeing them from pinned equilibrium positions, forcing dislocations to move in a preferred direction. Then Langenecker attributed the acoustic hardening to dislocation multiplication during intense ultrasonic irradiation. A TEM image (Figure 2.1) of aluminum after ultrasonic irradiation showing increased dislocation density with sub-boundaries consisting of dislocation networks. Before ultrasonic irradiation, the sample was free of such sub-boundaries and had a relatively low dislocation density (< $10^9$ m$^{-2}$).

![Figure 2.1: TEM image of aluminum specimen under ultrasonic irradiation [45].](image)
Awatani et al. [52] performed ultrasonic fatigue testing experiments in which copper exhibited strain hardening and increased microhardness after both the low and ultrasonic frequency compared to the annealed hardness. Patches of dislocations and subgrains were observed as shown in Figure 2.2 that were similar to those observed by Langenecker.

![Figure 2.2: Dislocation networks in copper specimen under ultrasonic fatigue test [52].](image)

Awatani and Katagiri also performed ultrasonic fatigue tests with aluminum and analyzed the microstructure by TEM [53]. They observed jogs and mentioned that vacancies can be generated due to the non-conservative motion of jogs on screw dislocations. The vacancies may sink to grain boundaries or edge dislocations leading to climb, but Awatani and Katagiri observed that some vacancies may condense to form loops and voids (Figure 2.3). The appearance of loops and voids indicates that a large number of vacancies were created by the ultrasonic irradiation.
Figure 2.3: (a) Dislocation loops and (b) voids appear in aluminum specimens under ultrasonic irradiation [53].

The process of ultrasonic softening is most likely due to the production a large concentration of vacancies above the thermal equilibrium value. Hull discusses the process of creating strain-induced vacancies [54], in which a critical applied stress is required for dislocation movement. During high rate plastic deformation, many screw dislocations acquire vacancy producing jogs as shown in the schematic in Figure 2.4 to create a large concentration of vacancies. If the glide plane of the dislocation is different from that of the jog, the screw location moves forward and takes jogs with it by a non-conservative process of climb.
Figure 2.4: Schematic showing generation of vacancies by climb of jogs on screw dislocations [54].
3. EXPERIMENTAL PROCEDURE

3.1 Materials

The powders used in this research are (1) electrolytic Cu powders (>99%) and spherical Cu powders (>99%), 8-12 μm and 6-10 μm in size, respectively, (Figure 3.1) both of which were supplied by Fukuda Metal Foil and Powder Co. Ltd., Japan, and (2) Al powders, 7-15 μm, (Figure 3.2) supplied by Alfa Aesar. In this research, the electrolytic Cu powder is called the “dendritic Cu powder” because of its morphology.
Figure 3.1: SEM pictures of as-received (a) dendritic Cu powder (b) spherical Cu powder.
Figure 3.2: SEM pictures of as-received Al powder.
3.2 Ultrasonic Powder Consolidation

A CONDOR ultrasonic welding unit, shown in Figure 3.3 manufactured by STAPLA Ultrasonic Corporation, Wilmington, MA was used for the UPC experiments. This unit operates at a maximum power of 3.5 kW and a fixed frequency of 20 kHz. The controller unit transmits high frequency signals to the converter and the resulting vibrations are transmitted to the ultrasonic sonotrode through the booster unit and their amplitude can be a value between 4-9 μm at a 2:1 ratio during the transmission as shown in Figure 3.4. The amplitude can be measured with the micro-gauge shown in Figure 3.3(a). The micro-gauge is connected to the machine by using a fixture. A uniaxial force is applied by using the Z-axis positioning knob and measured through a calibrated Rubbermaid Pelouze 4040 digital scale whose load cell stage was mounted on the anvil. The welding parameters are adjusted by the controllers shown in Figure 3.3(c) and (d). There were two controllers that were used during the research. All copper specimens were consolidated by an old-type analog controller (D-65451) and all aluminum specimens were consolidated by a new digital controller (ST30). Both controllers provided the same welding conditions and parameters.
Figure 3.3: STAPLA Condor ultrasonic welding unit, (a) Front view, (b) Side view, (c) Old control unit, (d) New control unit.
Figure 3.4: Amplitude enhancement of sonostrode due to booster configuration [7].
The sonotrode is made of high speed tool steel, so the maximum consolidation temperature is limited by the tempering temperature of the steel, and as such was kept below 500 °C in order to avoid softening of the sonotrode tip by over-tempering. The sonotrode tip has the size of 3.68 mm × 3.68 mm with its surface machined into a 14 × 14 grid of knurls as shown in Figure 3.5.

![Schematic diagrams of the sonotrode tip](image)

Figure 3.5: Schematic diagrams of the sonotrode tip [55].

To conduct experiments at elevated temperature, a heater plate was designed to be used with the ultrasonic welding unit. Two cartridge heaters from TUTCO which are 9.5 mm in diameter, 51 mm in length and 400 W in power and a K-type thermocouple probe from OMEGA are inserted in a stainless steel plate, as seen in Figure 3.6. The heaters and the thermocouple are connected to a control box (Figure 3.7) which is controlled by a computer equipped with a National Instruments (NI) PCI-6035E multifunction data acquisition (DAQ) card through a NI BNC-2110 connector block. A wiring diagram is
given in Figure 3.8. The computer program, which is coded on LabView 8.6, acquires the temperature data from the thermocouple and sends signals to the heaters to heat the plate to the set temperature.

Figure 3.6: Schematic views of the heater plate [7].

Figure 3.7: The control box for the heater plate.
Both copper and aluminum powders were consolidated under a uniaxial pressure and ultrasonic vibrations through a punch and die arrangement as shown in Figure 3.9. For copper consolidation experiments, the die was made of a 1.2 mm thick nickel plate with a 4.3 mm die hole and the punch has the dimensions of 4.1 mm in diameter and 3.4 mm in thickness. For aluminum consolidation experiments, the thickness and hole diameter of the die were 1.16 mm and 3.66 mm respectively and the thickness and diameter of the punch were 1.83 mm and 3.56 mm. The dies were tightly fitted into the die fixture with the powders being placed in it. For copper powder consolidation, a copper substrate which was 0.089 mm in thickness was placed under the die. For aluminum consolidation, a 0.025 mm nickel foil was placed under the die. For both metals, to prevent the powder from sticking of the powders to the punch, 0.025 mm thick nickel foil was placed between the punch and the powders.
After the materials and the punch and die arrangements were ready, the heaters were turned on to heat the whole assembly to the specified temperature which ranged from 473 K to 773 K. The time that reaches the specified temperature was about a-b s. Then the welding sonotrode was lowered to apply a specified normal load on the punch ranging between 80 and 100 MPa. When the load settled to the set value, ultrasonic vibration was applied parallel to the die surface at a frequency of 20 KHz with an amplitude of 9 μm and duration of 1 to 4 s. For aluminum, the whole process was done in air, and after consolidation, the die was cooled to room temperature, and ejected from the stage. Then the specimen was finally removed from the die. However, for copper, the same process was done in argon atmosphere. A large glove box, custom-designed and fabricated for the whole ultrasonic welding unit, was used to maintain the argon atmosphere as shown in Figure 3.10. The glove box was evacuated by a rotary pump and back-filled with argon gas a few times.
Figure 3.10: Custom-designed glove box.
3.3 Metallographic Characterization and Microscopy

After being removed from the die, the specimens were mounted in epoxy and cured for 30-40 minutes at room temperature. Then the specimens were ground on abrasive papers of different grit sizes (400, 600, 1000, 1200, 1500, 2000). Subsequent to grinding, the specimens were polished on buffing wheels with fine alumina powder with sizes of 1, 0.3, 0.05 μm. A Buehler ECOMET 5 two speed grinding-polishing table was used for the preparation of metallographic specimens. To see the microstructure and grain boundaries clearly, all copper specimens were etched by one part nitric acid and one part water for 35 seconds.

The microstructural characterization of samples was done with an Olympus VANOX-T optical microscope and a JEOL JSM-6360 scanning electron microscope (SEM) (3 nm resolution at 30 kV accelerating voltage).

3.4 Porosity Measurements and Ultrasonic Washing Tests

The porosity of the cross section is a good indicator of consolidation quality of the specimen. Since the specimen size is too small to apply the Archimedes’ principle, porosity was determined by image analysis wiring, the software ImageJ [1]. By counting the number of pixels that is associated with the porosities and dividing it by the total number of pixels in the image, ImageJ can calculate the area fraction of the porosities. Before the porosity calculation, to remove the contaminant on the cross section caused by
grinding and polishing, the specimens were washed in water by Fisher Scientific FS20D ultrasonic cleaner.

3.5 Hardness Tests

Vickers micro-hardness measurements were performed by using a Shimadzu HMV micro harness tester at loads of 245.2 mN for copper and 98.07 mN for aluminum, which corresponded to the Vickers micro hardness designations of HV 0.01 and HV 0.025. A loading time of 5 s was applied to all specimens.

3.6 Bending Tests

Just as well as the porosity measurements, manual bending tests can also measure the consolidation quality of specimens. The specimens were small, so half of every specimen was mounted in epoxy for metallographic characterization and the other half was manually bent with tweezers until it failed. Then the fracture surfaces of the specimens were examined by SEM to investigate the mode of fracture.
4. ULTRASONIC POWDER CONSOLIDATION OF COPPER AND ALUMINUM PARTICLES

Applying high strain rate deformation which allows for full-density consolidation at low to moderately elevated temperatures, UPC is a novel new technique for the consolidation of metal powders. Intense vibrations break and displace surface oxide layers and contaminants, thus facilitate full metal contact between powder particles and metallurgical bonding thereof. The softening effect observed in the ultrasonic welding of sheets and wires [1] should also be promoting the deformation of metal powders, leading to high degrees of compact densification.

This chapter addresses the production of copper and aluminum specimens by UPC. The UPC experiments were performed under different temperatures, pressures and ultrasonic consolidation time to investigate how these parameters affected the consolidation quality and under what conditions full-density consolidates could be achieved. Additionally, for the copper powder consolidation experiments, two different kinds of powders with different morphologies, dendritic and spherical, were used to investigate how the shapes of powders affected the consolidation results.
4.1 Ultrasonic Consolidation of Copper Powder

4.1.1 Microstructural Analysis

Figures 4.1 and 4.2 show the microstructures of copper specimens consolidated from dendritic and spherical powders, respectively, at 425 and 500 °C for 4 s under a uniaxial pressure of 84 MPa. The micrographs exhibit the microstructure changes of copper specimens caused by different consolidation temperatures. At 425 °C, both the dendritic and spherical specimens show the particle shape and boundaries clearly, which indicates that densification was obtained but metallurgical bonding was not fully attained. At 500 °C, the most particles were deformed and many of the prior particle boundaries disappeared, indicating better densification and a possible increase of metallurgical bonding.

An increase in temperature reduces the material’s yield point with the help of thermal activation to overcome the energy barrier for dislocation motion [57]. This makes the powders easier to deform. Therefore, high consolidation temperature leads to higher degrees of densification and metallurgical bonding. However, there is also a difference between dendritic and spherical powders. The effects of temperature were less significant for the spherical powders than for the dendritic ones. Compared with dendritic ones, spherical powders in a compact can more easily slide against each other, which suggests that they cannot be deformed as efficiently as dendritic ones. This explains why spherical specimens retain more original particle shape and boundaries at higher temperatures.
Figure 4.1: Optical micrographs of dendritic Cu powders consolidated under 84 MPa for 4 s at temperature (a) 500 °C, (b) 425 °C.

Figure 4.2: Optical micrographs of spherical Cu powders consolidated under 84 MPa for 4 s at temperature (a) 500 °C, (b) 425 °C.
Figures 4.3 and 4.4 show how pressure affected the dendritic and spherical copper powder consolidation at 500 °C and 4 s. At the higher pressure of 100 MPa, both powder consolidates showed good densification and metallurgical bonding, especially for spherical powders. It is considered that the higher pressure of 100 MPa restricted the slide of the spherical powders so that they can be deformed and bonded better than under 84 MPa.

Figure 4.5 shows the effects of consolidation time on the consolidation quality. The longer consolidation time produced a much better performance than the shorter one indicating that the densification of the powder compact is a time-dependent process.
Figure 4.3: Optical micrographs of dendritic Cu powders consolidated at 500 °C for 4 s under pressure (a) 100 MPa, (b) 84 MPa.

Figure 4.4: Optical micrographs of spherical Cu powders consolidated at 500 °C for 4 s under pressure (a) 100 MPa, (b) 84 MPa.

Figure 4.5: Optical micrographs of dendritic Cu powders consolidated at 500 °C under 84 MPa for (a) 4 s, (b) 2 s.
4.1.2 Ultrasonic Washing Test and Porosity Measurements

4.1.2.1 The phenomenon of porosity increasing during ultrasonic washing

Microstructural analysis can show how consolidation conditions affect the densification and bonding of powders, but it is only a qualitative observation. Porosity measurements of the cross section of consolidated specimens can show the consolidation quality in a quantitative manner. At first, ultrasonic washing in water was used to clean the cross section of the samples to avoid contaminants from grinding and polishing as shown in section 3.4. However, during this procedure, a new phenomenon was found that after different times of ultrasonic washing, different amounts of new pores appeared on the cross section as shown in Figures 4.6 and 4.7. With increasing washing time, the porosity increased initially and reached a constant value. This phenomenon indicates that under ultrasonic washing, some powder particles that were not compacted or bonded well came off the polished surface and those that were well bonded remained. As a result, this ultrasonic washing test was thought to provide a good means to study the extent of metallurgical bonding achieved in UPC and to see how the process parameters affected the consolidation. These measurements and tests were carried out for specimens consolidated under different processing conditions, as summarized in Tables 4.1-4.5. All as-consolidated specimens were ultrasonically washed in water for 2, 5, 10, 15 and 20 minutes. Optical micrographs were taken after each washing time and then were used to calculate the porosity by image analysis using the software ImageJ.
Figure 4.6: Optical micrographs of dendritic Cu powders consolidated under 500 °C and 4 s at pressure 84 MPa (a) before ultrasonic washing, (b) after 20 min ultrasonic washing.

Figure 4.7: Optical micrographs of spherical Cu powders consolidated under 500 °C and 4 s at pressure 84 MPa (a) before ultrasonic washing, (b) after 20 min ultrasonic washing.
Table 4.1: Processing window of copper powders consolidated under 84 MPa, 4 s and different temperatures.

<table>
<thead>
<tr>
<th>Copper Specimens</th>
<th>Temperature (°C)</th>
<th>Pressure (MPa)</th>
<th>Time (s)</th>
<th>Punch (mm)</th>
<th>Die (mm)</th>
<th>Atmosphere</th>
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</thead>
<tbody>
<tr>
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<td>4.3</td>
<td>Argon</td>
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<td>4.3</td>
<td>Argon</td>
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</table>

Table 4.2: Processing window of copper powders consolidated under 100 MPa, 4 s and different temperatures.

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</tr>
<tr>
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<td>100</td>
<td>4</td>
<td>4.1</td>
<td>4.3</td>
<td>Argon</td>
</tr>
</tbody>
</table>

Table 4.3: Processing window of copper powders consolidated under 84 MPa, 500 °C and different consolidation time.

<table>
<thead>
<tr>
<th>Copper Specimens</th>
<th>Temperature (°C)</th>
<th>Pressure (MPa)</th>
<th>Time (s)</th>
<th>Punch (mm)</th>
<th>Die (mm)</th>
<th>Atmosphere</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dendritic</td>
<td>500</td>
<td>84</td>
<td>4</td>
<td>4.1</td>
<td>4.3</td>
<td>Argon</td>
</tr>
<tr>
<td>Dendritic</td>
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<td>2</td>
<td>4.1</td>
<td>4.3</td>
<td>Argon</td>
</tr>
<tr>
<td>Dendritic</td>
<td>500</td>
<td>84</td>
<td>1</td>
<td>4.1</td>
<td>4.3</td>
<td>Argon</td>
</tr>
</tbody>
</table>
4.1.2.2 Porosity measurements and analysis for copper consolidated for 4 s under 84 MPa at different temperatures

Figure 4.8 shows how the density of the dendritic copper specimens changed with ultrasonic washing time. The degree of densification, or density defined as “100%-porosity”, is another way to express the quality of consolidation. With increasing ultrasonic washing time, the densities of all specimens decreased, which indicates that some powder particles which were not bonded well were removed from the specimens leaving pores. For dendritic powders consolidated for 4 s under 84 MPa, when the temperature was at and above 450 °C, both the densification and bonding of the powder were well-achieved as indicated by the high value of density above 98% even after a 20 minute wash. But for the specimen consolidated 425 °C, the density decreased very quickly with increasing washing time. This means that the densification and bonding between dendritic powder particles were poor at 425 °C, and there should be a critical temperature between 425 and 450 °C above which good ultrasonic consolidation is achieved.

The relationship between the density and consolidation temperature is shown in Figure 4.9 for specimens consolidated from the dendritic powder for 4 s under 84 MPa. The upper curve shows the density changes of as-consolidated specimens with respect to consolidation temperature. The lower curve shows the density changes of specimens that were after 20 minutes of ultrasonic washing. The upper one indicates that dendritic copper powder could achieve nearly full density (~100%) at and above 450 °C, while at
425 °C, the density stayed below 98%. After 20 minutes of ultrasonic washing, the specimens consolidated at above 450 °C, still kept their density above 98%, while the specimen consolidated at 425 °C suffered a large decrease in density to less than 90%. Thus, particle bonding was good in specimens consolidated at/above 450 °C, but was poor in specimens consolidated at 425 °C.
Figure 4.8: Density change with respect to ultrasonic washing time for dendritic copper powders consolidated under 84 MPa for 4 s at different temperatures.

Figure 4.9: Density change with respect to consolidation temperature for dendritic copper powders consolidated for 4 s under 84 MPa before and after ultrasonic washing.
Figures 4.10 & 4.11 show the porosity measurement results for the spherical copper powders obtained under the same conditions as the dendritic powders above. In Figure 4.10, all curves have declining trends with increasing washing time. The specimen consolidated at 500 °C has the best densification and bonding with the density remaining above 98% even after a 20 minute wash. The specimens consolidated below 500 °C have faster rates of density declining. Nonetheless, even at 425 °C, the final density still stays at 96% which is much higher than the density of the dendritic powder specimen consolidated at this temperature. Figure 4.11 shows the density values of the specimens consolidated from the spherical powder against the consolidation temperature, in both the as-consolidated and ultrasonically washed conditions. Unlike the results for the dendritic powders shown in Figure 4.9, the density values for the spherical powder specimens are not strongly affected by the consolidation temperature within the range investigated. This implies that the spherical powder particles repack more readily during UPC than dendritic powder particles, thereby promoting densification. Furthermore, the high density value of 96% of the specimen consolidated at 425 °C and washed for 20 minutes suggests a high degree of metallurgical bonding achieved in this specimen. Conversely, the dendritic powder specimen consolidated at 425 °C had only 88% after washing, indicating poorer bonding. The large drop of density from the as-consolidated value of 97% for the latter specimen suggests that mechanical interlocking may have been a significant contributor for the consolidation of dendritic powder in this specimen.

Figures 4.12 and 4.13 compare the density of the dendritic powder and spherical
powder specimens from the consolidation temperature of 425 °C to 500 °C in the as-consolidated condition and after 20 minutes of ultrasonically washing, respectively. Except at 425 °C, the density values of the dendritic powder specimens exceeded those of the spherical powder specimens both in the as-consolidated and ultrasonically washed conditions. The higher as-consolidated density values of the dendritic powder specimen above 450 °C suggest that the dendritic powder particles deformed to fill space more readily than the spherical powder particles above 450 °C. This may be attributed in part to the decrease in flow stress at elevated temperature. It also implies that the dendritic powder morphology helped the ultrasonic energy to be more effectively transferred for the deformation of the powder particles, and hence for compact densification. The latter energy transfer may not be efficient for spherical powder densification in which particle rotation, repacking and sliding would consume a significant portion of the applied ultrasonic energy.

The higher degrees of particle deformation in the dendritic powder densification would translate into higher degrees of fresh metal to metal contact at the particle boundaries, hence better metallurgical bonding in the dendritic powder specimens as seen in Figure 4.13.
Figure 4.10: Density change with respect to ultrasonic washing time for spherical copper powders consolidated under 84 MPa for 4 s at different temperatures.

Figure 4.11: Density change with respect to consolidation temperature for spherical copper powders consolidated for 4 s under 84 MPa before and after ultrasonic washing.
Figure 4.12: Density change with respect to consolidation temperature for dendritic and spherical copper powders consolidated for 4 s under 84 MPa before ultrasonic washing.

Figure 4.13: Density change with respect to consolidation temperature for dendritic and spherical copper powders consolidated for 4 s under 84 MPa after ultrasonic washing.
4.1.2.3 Porosity measurements and analysis for copper consolidated under 100 MPa for 4 s at different temperatures

This section discusses ultrasonic copper powder consolidation under a higher uniaxial pressure of 100 MPa for 4 s at temperatures of 300 °C, 400 °C, 450 °C and 500 °C. As shown in Figure 4.14 and 4.15, at 100 MPa, the dendritic copper powder consolidated very well at and above 450 °C, with the as-consolidated densities ranging from 97-100%. The specimen consolidated at 500 °C maintained very high density (~100%) even after 20 minutes of ultrasonic washing. The specimen consolidated at 450 °C, too, kept a high density (~98%) after washing. The specimen consolidated at 400 °C had a somewhat lower as-consolidated density (~97%) but suffered only a small loss of density during the ultrasonic washing, with the final density value above 96%. The specimen consolidated at 300 °C also had a comparably high as-consolidated density of 97%, but its density dropped rapidly below 91% during ultrasonic washing. This strongly suggests that attaining high degrees of densification does not necessarily translate into good metallurgical bonding.

The above results indicates that producing high density consolidates with good inter-particle bonding requires a sufficient uniaxial pressure that permits the powder particles to deform and fill space which exposes fresh metal surface needed for metallurgical bonding. Powder particle repacking may be easier at lower uniaxial pressures, but its effect is less significant than that of powder deformation, particularly for dendritic powder particles.
Figure 4.14: Density change with respect to ultrasonic washing time for dendritic copper powders consolidated under 100 MPa for 4 s at different temperatures.

Figure 4.15: Density change with respect to consolidation temperature for dendritic copper powders consolidated for 4 s under 100 MPa before and after ultrasonic washing.
Figures 4.16 and 4.17 show the density of the specimens consolidated from the spherical powder. The specimens consolidated at 450 °C and 500 °C had very high as-consolidated density above 99%. The specimens consolidated at 400 °C and 300 °C have reasonably high values of as-consolidated density. Thus, under higher uniaxial pressure of 100 MPa, near full-density states were achieved in these specimens as well. The fact that densification improved under 100 MPa over that attained under 84 MPa suggests that particle deformation has greater effects than initial particle repacking and densification.

Despite the high values of as-consolidated density for all four consolidation temperatures, the specimens consolidated at 400 °C and 300 °C quickly lost some powder particles during ultrasonic washing. This suggests insufficient bonding of powder particles in these materials. Lack of metallurgical bonding generally reflects two things: lack of fresh metal-to-metal contact and lack of thermal activation. The fact that the spherical powder specimen consolidated at 400 °C had a lower density after ultrasonic washing than the dendritic powder specimen consolidated at the same temperature suggests that the densification of spherical powder particles produced less metal-to-metal contact because of their simpler initial spherical geometry that requires less deformation to fill space. Also, spherical powder would produce less mechanical interlocking, a possible mechanism of powder integration.

The low density of the specimens consolidated at 300 °C, whether from the dendritic or spherical powder, is likely caused mainly by a lack of thermal activation for atoms to
reposition at the mating particle surface to form a metallurgically bonding interface.

Figure 4.16: Density change with respect to ultrasonic washing time for spherical copper powders consolidated under 100 MPa for 4 s at different temperatures.

Figure 4.17: Density change with respect to consolidation temperature for spherical copper powders consolidated for 4 s under 100 MPa before and after ultrasonic washing.
Figures 4.18 and 4.19 compare, respectively, the as-consolidated and after-wash densities of the dendritic and spherical powder specimens consolidated under 100 MPa. Except at 500 °C, the spherical powder specimens have higher as-consolidated density, reflecting the simpler particle shape of the spherical powder, which enhance particle initial repacking, lowering the initial density. The difference, however, deceases with increasing consolidation temperature above 400 °C, and at 500 °C, dendritic powder specimens attain a slightly higher density than spherical powder specimens.

After 20 minutes of ultrasonic washing, the density values of the spherical powder specimens fall behind those of the dendritic powder specimens, particularly at consolidation temperature below 450 °C, Figure 4.19, due to a lack of metallurgical bonding of powder particles despite the higher degrees of densification. The difference may have arisen from the difference in the amount of fresh metal-to-metal contact, and possibly from the degree to which mechanical interlocking is achieved between the spherical and dendritic powder morphologies.
Figure 4.18: Density change with respect to consolidation temperature for dendritic and spherical copper powders consolidated for 4 s under 100 MPa before ultrasonic washing.

Figure 4.19: Density change with respect to consolidation temperature for dendritic and spherical copper powders consolidated for 4 s under 100 MPa after ultrasonic washing.
4.1.2.4 Porosity measurements and analysis for dendritic copper consolidated at 500 °C under 100 MPa for different consolidation time

Figures 4.20 and 4.21 show the density changes of dendritic copper powders consolidated at 500 °C under 84 MPa for different consolidation times. The specimen consolidated for 4 s retained a high density value (~99%) even after 15 minutes of ultrasonic washing. In comparison, the density of the specimens consolidated for 2 s and 1 s decreased very quickly during the first 5 minutes of ultrasonic washing and then continued to decrease further to low values of 93% to 94%. Figure 4.21 shows that although the densification of the compacts was nearly complete at 500 °C in 1 second, the bonding of particles required 4 seconds, indicating that forming metallurgical bonding at the mating surfaces of powder particles requires thermal activation. This also suggests that the integrity of the material consolidated at 500 °C is provided largely by metallurgical bonding, and not so much by mechanical interlocking as the degree of mechanical interlocking is considered to be directly proportional to the degree of densification.
Figure 4.20: Density change with respect to ultrasonic washing time for dendritic copper powders consolidated at 500 °C under 84 MPa for different consolidation time.

Figure 4.21: Density change with respect to consolidation temperature for dendritic copper powders consolidated at 500 °C under 84 MPa before and after ultrasonic washing.
4.1.3 Hardness Tests

Figures 4.22-4.24 show the micro-Vickers hardness of the dendritic and spherical powder consolidates which were ultrasonically washed for 20 minutes as a function of consolidation temperature and time. The hardness of a powder compact is expected to correlate with the density of the compact. That this is true for the copper consolidates produced in this research is confirmed by comparing Figures 4.22-4.24 with Figures 4.13, 4.19 and 4.21, which shows striking similarities.

Figure 4.22: Hardness change with respect to consolidation temperature for copper powders consolidated for 4 s under 84 MPa.
Figure 4.23: Hardness change with respect to consolidation temperature for copper powders consolidated for 4 s under 100 MPa.

Figure 4.24: Hardness change with respect to consolidation time for copper powders consolidated at 500 °C under 84 MPa.
4.2 Ultrasonic Powder Consolidation of Aluminum Powders

4.2.1 Ultrasonic Washing Tests and Porosity Measurements

The densifications and metallurgical bonding of Al specimens consolidated by UPC were also investigated through the same porosity measurement of ultrasonic washing procedure. Al specimens were consolidated under various conditions to study the effects of consolidation temperature and time as shown in Tables 4.4 and 4.5. The uniaxial pressure was fixed at 80 MPa and an air atmosphere was used for all of the experiments for aluminum.
Table 4.4: Processing window of aluminum powders consolidated under 80 MPa, 1 s and different temperatures.

<table>
<thead>
<tr>
<th>Aluminum Specimens</th>
<th>Temperature (°C)</th>
<th>Pressure (MPa)</th>
<th>Time (s)</th>
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<td>350</td>
<td>80</td>
<td>1</td>
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Table 4.5: Processing window of aluminum powders consolidated under 80 MPa, 300/400 °C and different consolidation time.

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<th>Aluminum Specimens</th>
<th>Temperature (°C)</th>
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4.2.1.1 The Phenomenon of Porosity Increasing during Ultrasonic Washing

Figure 4.25 shows optical micrographs of a specimen from Exp. 2 in Table 4.4 consolidated at 350 °C for 1 s under 80 MPa pressure. The as-consolidated specimen show virtually no pores, but, just as in the copper specimen, the porosity on the polished surface of the specimens increased when the specimen was washed ultrasonically, indicating that some of the Al particles were not well bonded to their neighbors.

Figure 4.25: Optical micrographs of aluminum powders consolidated at 350 °C for 1 s under pressure 80 MPa (a) before ultrasonic washing, (b) after 20 min ultrasonic washing.
4.2.1.2 Porosity Measurements and Analysis of Aluminum Consolidated for 1 s under 80 MPa at Different Consolidation Temperatures

Figure 4.26 plots the density of the Al specimens consolidated at 200 °C-400 °C as a function of ultrasonic washing time. It is noticed that the specimens consolidated at 350 °C and 400 °C had a nearly full density above 99%, whereas the specimens consolidated below 350 °C had a lower density that decreased with decreasing consolidation temperature. It is also noticed that the consolidate density decreased with increasing ultrasonic washing time for all of the 5 consolidation temperatures. After 20 minutes of washing, the density decreased about 0.5 to 0.6 points regardless of consolidation temperatures. This indicates that the extent of metallurgical bond formation was largely independent of temperature within the range tested.

Figure 4.27 replots the data in Figure 4.26 to show the effects of consolidation temperature on the density of the Al specimens consolidated under 80 MPa for 1 s in the as-consolidated state and after the 20 minute ultrasonic wash. Both the as-consolidated and after-wash values increase almost linearly with consolidation temperature up to 350 °C, with the after-wash values falling consistently below the as-consolidated values by about 0.6-0.7 points. Above 350 °C, the density levels off for both states, but with the as-washed values still falling behind the as-consolidated values by about 0.8 points. This suggests that the extent of compact densification is proportional to the degree to which the metallurgical bonding is achieved in the Al specimens consolidated for 1 s. In other words, metallurgical bonding occurs almost as quickly as densification proceeds, i.e.,
metallurgical bonding in aluminum compacts requires much less time than in copper compacts (see Figure 4.10). This suggests that the rate of metallurgical bond formation is limited by the rate of creating fresh metal-to-metal contact in the compact, which increases with increasing densification.
Figure 4.26: Density change with respect to ultrasonic washing time for aluminum powders consolidated under 80 MPa for 1 s at different consolidation temperature.

Figure 4.27: Density change with respect to consolidation temperature for aluminum powders consolidated for 1 s under 80 MPa before and after ultrasonic washing.
4.2.1.3 Porosity Measurements and Analysis for Aluminum Consolidated at 300/400 °C under 80 MPa for Different Consolidation Time

The above observation that metallurgical bonding proceeds as quickly as densification increase, is further verified in Figure 4.28 which plots the change of density due to ultrasonic washing for aluminum compacts consolidated at 300 °C for different times ranging from 1 to 2.5 s. It is clear that the compact densification, represented by the density of as-consolidated materials, increases with increasing consolidation time. Thus, the powder particles in the compact kept deforming more when consolidated for longer times, i.e., the deformation of powder particles is time-dependent.

However, when subjected to ultrasonic washing, all of the consolidated materials suffered nearly the same amounts of mass loss, as reflected by the nearly identical shapes of the density-washing time curves for all consolidation times. Thus, the increases in densification caused by the application of longer consolidation times translate directly into proportional increases in metallurgical bonding in aluminum specimens consolidated at 300 °C. Figure 4.29, which plots the density against consolidation time for both the as-consolidated and 20 minute washed conditions, clearly shows that any increase in densification gained at an increased consolidation time is followed by almost the same amount of increase in metallurgical bonding.

The above observations further confirm that any fresh metal-to-metal contact caused by powder particle deformation contributes almost directly to metallurgical bonding. In addition, the fact that the density values after washing lag behind those of as-consolidated
materials by a nearly constant amount of about 0.6-0.8 points implies that some of the oxide films that existed in the original powder particles may remain as oxides fragments at the prior particle boundaries, preventing full metal-to-metal contact, hence metallurgical bonding.
Figure 4.28: Density change with respect to ultrasonic washing time for aluminum powders consolidated at 300 °C under 80 MPa for different consolidation time.

Figure 4.29: Density change with respect to consolidation temperature for aluminum powders consolidated at 300 °C under 80 MPa before and after ultrasonic washing.
Figures 4.30 and 4.31 plot the density changes of the Al specimens consolidated at a higher temperature, 400 °C, under 80 MPa for the same time range of 1 to 2.5 s. It is clear that the densities of as-consolidated materials at 400 °C all remained above 99.1% and decrease only slightly with decreasing consolidation time. Thus the deformation of Al powder particles was not very strongly time-dependent at 400 °C, i.e., the powder particles deformed very quickly within 1 s under the ultrasonic consolidation conditions.

When subjected to ultrasonic washing, the consolidated materials suffered different amounts of mass loss. The density declining rate decreased with increasing consolidation time, but even for the shortest time of 1 s, the after-wash density still remained above 98.4%. This indicates that at 400 °C, the aluminum powder particles achieved high levels of metallurgical bonding very quickly. When the time increases to 2.5 s, the as-consolidated density increased to 99.4%, the highest value achieved for Al consolidation in this research. The drop in density during 20 minutes of washing was decreased from 0.8 points for 1 s, the value comparable to those of the Al specimens consolidated at 300 °C, to 0.2 points at 2.5 s. This indicated that some of the oxide fragments at the particle boundaries were sucked into the interior of the particles as the powder compact was subjected to ultrasonic deformation.

Figure 4.32 and 4.33 compare the results obtained for the Al consolidated at 300 °C and 400 °C under the same pressure for the same range of consolidation time. It is are clear that, besides the contributions of flesh metal-to-metal contact caused by powder particle deformation, thermal activation, which increases with increasing temperature,
plays a significant role in powder particle deformation and metallurgical bonding formation, especially for a short time. Specifically, increased thermal activation would allow dynamic recovery or recrystallization to take place more readily, thereby allowing for higher degrees of particle deformation and oxide removal from the particle boundaries.
Figure 4.30: Density change with respect to ultrasonic washing time for aluminum powders consolidated at 400 °C under 80 MPa for different consolidation time.

Figure 4.31: Density change with respect to consolidation temperature for aluminum powders consolidated at 400 °C under 80 MPa before and after ultrasonic washing.
Figure 4.32: Density change with respect to consolidation temperature for aluminum powders consolidated at 300/400 °C under 80 MPa before ultrasonic washing.

Figure 4.33: Density change with respect to consolidation temperature for aluminum powders consolidated at 300/400 °C under 80 MPa after ultrasonic washing.
4.2.2 Hardness Tests

Figures 4.34 and 4.35 show the micro-Vickers hardness results of aluminum powder consolidation under the conditions shown in Tables 4.4 and 4.5. Comparison with the density results showed in Figures 4.27 and 4.33, respectively, shows that although the hardness of Al did decrease with decreasing consolidation temperature and time, the hardness and density curves do not show as striking similarities as those of the copper powder consolidations show before. This indicates that the hardness results of aluminum powder specimens are not entirely for related to the density. There might be other factors that influence the hardness of the aluminum specimens. Nonetheless, the hardness results still suggest that both temperature and consolidation time influence the attainment of densification and metallurgical bonding.
Figure 4.34: Hardness change with respect to consolidation temperature for aluminum powders consolidated under 1 s and 80 MPa.

Figure 4.35: Hardness change with respect to consolidation time for aluminum powders consolidated under 300/400 °C and 80 MPa.
4.2.3 Bending Tests

The consolidated aluminum specimens were subjected to manual bending tests to evaluate the material ductility and examine the fracture surfaces. All specimens withstood 90° bending tests to varying degrees at first. To break them to observe the fracture surface, these specimens need to be cut slightly on the edge and bent several times. This indicates that different degrees of metallurgical bonding were achieved for specimens consolidated at different conditions. Figure 4.36 shows the fracture surfaces of aluminum specimens consolidated at 400/300 °C for 1 s under 80 MPa. At 400 °C, the fracture surface was more ductile than the surface at 300 °C as verified by the observation that the particle shape and boundaries were not discernible, while at 300 °C, features indicative of particle boundaries and pores are seen on the surface. Figure 4.37 shows the fracture surfaces of aluminum powders consolidated at 300 °C for 2 s and 1 s under 80 MPa. At the longer consolidation time of 2 s, a higher degree of the fracture surface exhibits much fewer features indicative of prior particles or pores.
Figure 4.36: SEM micrographs of the fracture surfaces of aluminum powders consolidated for 1 s under 80 MPa at (a) 400 °C, (b) 300 °C.

Figure 4.37: SEM micrographs of the fracture surface of aluminum powders consolidated at 300 °C under 80 MPa for (a) 2 s, (b) 1 s.
5. CONCLUSIONS

UPC provides a viable route for the rapid consolidation of metal powders at low temperature under low pressure. This research focused on studying the densification and bonding of copper and aluminum powders in ultrasonic powder consolidation.

For copper powder with dendritic and spherical morphologies, the minimum conditions for nearly full densification were determined to be 450 °C, 4 s and 84 MPa, with the best ones obtained at 500 °C for 4 s under 100 MPa. Dendritic copper powder specimens exhibited better densification and metallurgical bonding than spherical ones above 450 °C because they had to deform to a higher degree to fill the space between particles, hence creating more metal-to-metal contacts for bonding formation. Spherical copper powder specimens produced better results below 450 °C due to their better packing geometry and better repacking under ultrasonic vibration.

Application of higher uniaxial pressure makes the copper powder particles deform more readily to achieve higher densification while exposing more fresh metal-to-metal contact for metallurgical bonding. However, specimens consolidated under higher pressure but at lower temperature performed worse in terms of bonding, indicating that both fresh metal-to-metal contact surface and thermal activation are necessary for good metallurgical bonding. The degree of bonding increased with increasing consolidation time, indicating that bonding is a thermally activated process.

The results of hardness tests show that the hardness of copper powder
For aluminum, the best consolidation conditions were found to be 400 °C, 2.5 s and 80 MPa. Within the testing range from 200 °C to 400 °C for 1 s under 80 MPa, aluminum specimens could achieve nearly full density above 350 °C and the density decreased with decreasing consolidation temperature below 350 °C. After 20 minutes of ultrasonic washing, the density decreased almost the same amount (0.6 points) below the as-consolidated values, suggesting that the extent of compact densification correlates proportionally to the degree to which the metallurgical bonding is achieved in the Al specimens which is largely independent of temperature. The phenomenon that metallurgical bonding occurs almost as quickly as densification proceeds indicates that the rate of metallurgical bond formation is limited by the rate of creating fresh metal-to-metal contact in the compact.

At 300 °C under 80 MPa for different consolidation times ranging from 1 to 2.5 s, the deformation of aluminum powder particles is time-dependent. However, when subjected to ultrasonic washing, nearly the same amounts of mass loss happened for all of consolidated specimens regardless of consolidation time. This confirms again that any fresh metal-to-metal contact caused by powder particle deformation translates almost directly to metallurgical bonding. In addition, the observed constant differences in density values between as-consolidated and after-washing specimens implies that oxide fragments persist at the aluminum powder particle boundaries, preventing full metal-to-metal contact, hence metallurgical bonding.
At 400 °C under otherwise identical conditions, densification was readily achieved within 1 s, indicating the dependence of densification in time. The mass loss of 400 °C-consolidated Al specimens during ultrasonic washing decreased with increasing consolidation time, and was only 0.2 points for specimens consolidated for 2.5 s. Thus, long consolidation times cause oxide fragments to move into and more uniformly distributed in the powder particle interior, thereby increasing metallurgical bonding. Further comparison of the data obtained at 300 °C and 400 °C suggests that both densification and bonding are thermally activated processes.

The hardness of aluminum specimens does not entirely correlate with the density. Nonetheless, the hardness results also suggest that both temperature and consolidation time affect the attainment of densification and metallurgical bonding.
REFERENCES


