Phase Transformation in the Aluminum/Tungsten System

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PHASE TRANSFORMATION IN THE ALUMINUM/TUNGSTEN SYSTEM

A dissertation submitted in partial fulfillment of the requirements for the degree of

Doctor of Philosophy

By

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ABSTRACT


Formation and growth mechanisms of intermetallic compounds in the aluminum/tungsten system were studied during consolidation and subsequent heat treatment. Three different processes, spark plasma sintering (SPS), laser powder bed fusion (LPBF), and metal inert gas welding (MIG) were used, and each of these processes were followed up with heat treatment at different conditions. Al$_{12}$W, Al$_5$W and Al$_4$W are the three intermetallics that are stable in the system in room temperature, and they were expected to form at the interface between tungsten and aluminum at different temperature ranges. Contrary to expectations, the Al$_5$W phase was not observed during this study and the Al$_4$W phase only formed when molten aluminum reacted with tungsten for a sufficiently long time, for example during welding or during re-melt of LPBF samples. The Al$_{12}$W phase formed on the Al/W interface during solid-state heat treatment starting in the temperature range from 500 °C to the melting point of the matrix. This phase was observed on the interface of Al$_4$W/Al on the side of Al$_4$W during heat treatment of the
welded sample, heat treatment below the aluminum melting point for a long time led to the consumption of $\text{Al}_4\text{W}$ to form more $\text{Al}_{12}\text{W}$.

During solid state heat treatment, the $\text{Al}_{12}\text{W}$ phase formed at the Al/W interface and grew into the Al matrix outside the W particles border. The $\text{Al}_{12}\text{W}$ particles had a hexagonal multi-faceted interface with the Al matrix, with a 120° angle between the facets. No composition variation was observed in the $\text{Al}_{12}\text{W}$ phase over the entire distance from the W/$\text{Al}_{12}\text{W}$ interface to the $\text{Al}_{12}\text{W}$/Al interface. These observations pointed to the possibility that the growth of $\text{Al}_{12}\text{W}$ during solid state transformation was interface-controlled, with rapid transport of Al and W through the ordered $\text{Al}_{12}\text{W}$. The reason for the faceted interface morphology of the $\text{Al}_{12}\text{W}$ is due to the high ratio of the latent heat of fusion to melting point, i.e. the entropy of fusion.

The equilibrium $\text{Al}_{12}\text{W}$ precipitates have hexagonal shape with internal angle of 120°, this is due to expanding the highest planar density {110} plane family during growth and becomes the dominant planes of the crystal due to the high planar density of atom in this family of planes compared with other families of planes.

As in other intermetallics, the tungsten aluminides have complex crystal structures, where cages of aluminum atoms surrounding a single tungsten atom represent the common trait among these structures. The $\text{Al}_{12}\text{W}$ has a BCC-based crystal structure, where it has $\text{Al}_{12}\text{W}$ units at the corners and the body center. The $\text{Al}_{12}\text{W}$ crystal structure has a more open structure and lower density as compared with $\text{Al}_4\text{W}$ and $\text{Al}_5\text{W}$. Despite the open
structure of the Al$_{12}$W, the structure cavities have small size where none of the tungsten atoms nor aluminum can be fitted in, so that there is no interstitial diffusion can occur. The constant composition along the Al$_{12}$W intermetallic phase implies an interface-controlled growth mechanism with rapid diffusion. There are interstitial sites on the cube faces that are the potential fast diffusion paths for both Al and W atoms.
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Due to their high specific properties, composite materials brought attention to many researchers and have been applied in many applications in industries such as automotive and aerospace. Metal-metal composite is an important branch of the material composite field, where in most cases an intermetallic compound(s) forms due to interaction among...
composite components. These intermetallics can have attractive properties as in the aluminum tungsten system, where three stable intermetallic compounds formed at aluminum rich region of the aluminum tungsten phase diagram as shown in the Figure (1.1) $\text{Al}_2\text{W}$, $\text{Al}_5\text{W}$ and $\text{Al}_4\text{W}$ The tungsten aluminides have high specific mechanical properties, where take the advantage of the low density of the aluminum and the high strength of the tungsten [2][3].

The formation and growth of the tungsten aluminides are difficult processes to be handled this is due to the limited solubility, the high reactivity of the aluminum and tungsten to each other and the large difference in their physical properties, this make there is no a specific method for fabrication the aluminum tungsten composites with desired properties [4]. In this study three different processes where applied to create the composite: spark plasma sintering (SPS): which is a powder metallurgy technique includes the application of pressure and pulses of a direct electric current through in graphite mold during consolidation of mixed powders, laser powder bed fusion (LPBF): which is a 3D metal printing process using a laser beam to quickly melt a thin layer of a metal powder, and metal inert gas welding (MIG): which is an electric joining process use heat of an electric arc to melt a filler metal and a base metal to weld parts together. Consolidation was followed by heat treatment at different times and temperature to understand what the conditions that are required to create and grow an individual intermetallic.
The aluminum intermetallics, in general, and the tungsten aluminides in particular
have complex crystal structures.
These compounds are, in most cases, line compounds, that makes
the mechanism of atom transport through the lattice difficult to understand [5] [6]. There are two
types of mechanisms that describe transformation growth processes
depending on the way at which new atoms attach to the surface of the growing phase:
interface-controlled and diffusion controlled. During interface-controlled growth, the
interface is glissile, where transition occurs over about a single atomic layer. This makes
the concentration along an interface almost fixed and the interface has sharp, faceted
surface. In such a type of growth, the activation energy should be large enough to pull new
atoms. In contrast, the interface during diffusion-controlled growth is non-glissile and has
rough and curved edges. The transfer occurs along multi atomic layers that make
concentration along the interface (or at least close to interface edge) have gradient change,
Figure (1.2) shows the two types of the interphase growth There are two types of interfaces
that describe transformation growth processes depending on the way at which new atoms
have been attached to an interphase: interface-controlled and diffusion-controlled. The
interface-controlled growth interphase has a glissile interface where transition occurs over
about a single layer thick; this makes the concentration along an interface almost fixed and the interface has sharp, faceted edges. In such a type of growth, the activation energy should be large enough to pull new atoms. In contrast, the diffusion-controlled growth interphase has a non-glissile interface and has rough and curved edges. The transfer occurs along multi atomic layers that make concentration along the interface (or at least close to interface edge) have gradient change, Figure (1.2) shows the two types of the interphase growth [7][8]. Questions like; at what environment condition each individual intermetallic forms, and which phases form first and at sequence form the tungsten particle. Another important question is what the mechanism of growth and how metal atoms transfer. Also try to explain why the Al₁₂W has a flat interface, and why it has hexagonal-like morphology. All these questions and more will be answered in this dissertation.

This alloy system has been previously studied, where researchers focused on how the intermetallics were created, what are the mechanical properties of each intermetallic and how the composite properties are affected. There is no literature data available that especially deals with growth behavior of the tungsten aluminides, but other literatures discuss similar behavior in other systems.
2. LITERATURE REVIEW

In recent years there has been increased attention to the aluminum-tungsten alloy system because of the high specific properties of the aluminum tungsten intermetallic compounds. However, there is only a limited amount of published literature about aluminum tungsten systems, especially about phase transformations. The following paragraphs review the research papers that report on the aluminum-tungsten and other aluminum-refractory metal alloy systems.

1.1 ALUMINUM-TUNGSTEN COMPOSITE PREPARATION

Most of the papers that deal with aluminum tungsten focus on how to fabricate the composite since getting the desired phases is a complicated process because of the wide disparity in the component properties and the limited solubility of the metals in each other. Rajulapati et al [9] prepared a composite of Al and up to 4 atomic% W powder (44 µm) by mechanical alloying using ball-milling followed by hot compaction. The ball-milling process was carried out at room temperature for 24 hours, then the mixture hot compacted at 300 °C under 1.8 GPa pressure in an argon atmosphere. Examination by XRD showed no tungsten aluminides were formed, probably due to the low processing temperatures.

A roll bonding technique was used to create a tungsten-aluminum composite by Liu et al [10]. Where up to 44.3 g of irregular shape W powder was spread out in between two aluminum sheets using a scraper knife. The assembled sandwich was processed in a roll
bonding machine for 11 cycles at 280 °C for 5 minutes for each cycle, the total reduction in thickness was 50%. As in previous work no intermetallics were observed at the aluminum/tungsten interface as shown in the Figure (2.1), where the interface looked smooth and clear, the EDS line analysis showed a sharp transformation in composition across the Al-W interfaces. The XRD peaks confirmed that there was no interaction between the aluminum sheet and the tungsten powder particles.

Feng et al [11] prepared an aluminum tungsten composite by mixing three tungsten volume fractions (1, 2, and 3 vol%) of 3 μm particle size with 200 mesh pure aluminum powder. The mixtures mechanically stirred in alcohol solution followed by drying. The dried powder mixture was compacted into pellets under higher pressure. The pellets were sintered at 650 °C for 2 hours followed by hot extrusion at 500 °C. The SEM images shown in Figure (2.2) indicate the formation of the Al$_{12}$W intermetallic during heat treatment. The XRD peaks (Figure 2.3) showed that the tungsten particles had been consumed during the heat treatment. These researchers also reported that the interface between aluminum and the intermetallic was smooth and clear as shown in Figure (2.2) and there was good bonding across the interface. Researchers also reported that the interface between aluminum and the
intermetallic was smooth and clear as shown in Figure (2.2) and there was good bonding across the interface.

Peets et al [12] investigated the physical properties of whole crystals of tungsten aluminide and molybdenum aluminides. In the tungsten aluminides portion of the research, a pure aluminum wire and pure tungsten powder in weight ratio of 60:1 were mixed in crucibles under an argon filled box followed by sealing under vacuum inside quartz tubes. The researchers aimed to prepare single crystals of the Al₄W and Al₅W. To produce the Al₄W the crucible heated to 1050 °C at 200 deg C/min to convert the powders into a single crystal.

![Figure 2.2: (a) Al₁₂W/Al interface, (b) is SADP of Al₁₂W particle [11].](image)

![Figure 2.3: XRD peaks patterns of sintered Al/W composite powder [11].](image)

![Figure 2.4: a) Al₄W crystal, b) Al₅W crystal [12].](image)
°C/h and hold 2 hours, followed by cooling and holding at 900 °C for 3-7 days followed by furnace cooling to room temperature. While to form Al5W the second crucible heated to 850 °C and cooled to 720 °C. The result products then dissolved in hydrochloric acid to remove any access aluminum. The observed crystal showing in Figure (2.4) where they have about 300 µm thickness. The crystal structure of the Al5W is formed from aluminum cages of single tungsten atoms surrounded by 12 aluminum atoms, these cages sharing edges in ab-plane, while in c-axis cages sharing corners Figure (2.5). The refined composition is Al4.928W. The Al4W has a more complicated structure, where the cages form from either 10 or 11 aluminum atoms surrounding a single tungsten atom, the refined composition Al3.808W.

Wan et al [4] prepared Al4W/Al12W composite by dipping a tungsten (99.9% purity) porous structure with density of 60%, pre-prepared by pressure tungsten powders (6-8 µm particle size) at 1000 °C for 40 minutes, into molten aluminum (9.99% purity) at 800 °C and holding the mixture in vacuum furnace for four different time periods. After furnace cooling, the Al4W/Al12W composite were observed as shown in the Figure (2.6). According to the XRD analysis of the interface region, where the Al4W formed at the
interface close to the tungsten side while \( \text{Al}_{12}\text{W} \) formed at the interface close to the aluminum side. The researchers mentioned that as holding time increased, the content of the \( \text{Al}_{4}\text{W} \) decreased and \( \text{Al}_{12}\text{W} \) increased. Where for the 10 minutes hold time there is no XRD peaks for the \( \text{Al}_{12}\text{W} \) was observed, as the holding time increased the \( \text{Al}_{12}\text{W} \) peak become stronger, the EBSD maps Figure (2.7)

![Figure 2.6: Morphologies of \( \text{Al}_{4}\text{W}/\text{Al}_{12}\text{W} \) composite [4]](image)

![Figure 2.7: EBSD results of \( \text{Al}_{4}\text{W}/\text{Al}_{12}\text{W} \) composite synthesized at 800 °C for 40, 50, 60 minutes; (a, b, c) [4].](image)

showing how the volume fractions of the intermetallics change with increasing holding time. Also, it has been mentioned that the \( \text{Al}_{4}\text{W} \) grain size get bigger as the holding time increased.
Rajamure et al [13] prepared the tungsten aluminum composite, by applying a
paint-like layer of tungsten about 220 μm in thickness over as-received 1100 aluminum
alloy plate. The paint was created by mixing W powder (1-5 μm particle size) with water based
organic binder as shown in the Figure (2.8). After the layer was dried, a laser beam with
different parameters settings (energies) and varying scan modes over the tungsten layer.
The XRD peaks for the interface area showed that only Al₄W intermetallic was observed
beside the main component (Al and W). The researchers attributed the observation to the only Al₄W among other intermetallics, due to
its lowest Gibbs free energy compared to the other tungsten aluminides as ThermoCalc
software shows.

Golkovski et al [3] apply an electron beam with power of 85 kW and energy of 1.4
MeV was over tungsten aluminum powder that had 85% W and 15% Al in atomic percent
(not mentioned particles size and layer thickness) with adding some flux material to avoid
powder reaction with atmosphere, the powder layer covered an aluminum substrate. The
beam was used as a heat source with frequency of 20-50 Hz with scan speed of 3.5 cm/s,
6 layers were added over the plate and the scan done under vacuum. The researchers
observed only Al₄W intermetallic at W/Al interface as the SEM image and XRD results

![Figure 2.8: SEM backscattering image of W coating on Al 1100 sample processes at 21 J/mm² laser energy [13].](image-url)
showed in the Figure (2.9). The researchers attributed the observation of the only Al₄W among other intermetallics is due to the high concentrated beam energy over small areas leading to rise in temperature to high value followed by fast cooling (high scan speed). The researcher mentioned that the Al₄W forms from melting at 1327 °C. Similarly, Silva et al [14] were implanted with tungsten ions using an ion beam into a thin commercially pure aluminum disc 10 mm in diameter. The prepared samples were subjected to heat treatment for 80 minutes in a vacuum furnace at a temperature range from 280 °C to 620 °C. Al₁₂W was reported as the only intermetallic phase that formed due to aluminum tungsten interaction.
Zhang et al [15] use the PCP (pulsed current processing) a powder metallurgy process to consolidate two different size groups of aluminum tungsten micro and nanoparticles size. PCP process is similar to the SPS (spark plasma sintering) process that has been used in current study. The two powders mixed using a ball mill for 2 hours at room temperature under air and argon shielding atmosphere, the tungsten atomic percent was 20% in both powder particles sizes. A pressure of 20 MPa was applied on the powder that filled into a graphite die and heated to 550 °C for two minutes. The consolidated pellet was then heat treated at 600 and 650 °C for 1 and 2 hours. The results show that the tungsten nanoparticles were completely consumed and converted to Al₄W while both Al₁₂W and Al₄W intermetallics were observed in the interface of aluminum and tungsten of the micro size particles composite. The researchers reported that heating the micro-sized particle

Figure 2.10: Microstructure of Al/W composite compacts heated with different temperature profiles. The high magnification images (b), (d) and (f) show that increased temperature and prolonged heating promote the phase transformation [15].
sample to 600 °C with no holding time resulted in a thin layer of Al₁₂W at the Al/W interphase. While one hour holding time at the same temperature leads to a thick layer of Al₄W at the interface between W/Al₁₂W, where Al₁₂W still has a thin layer at the Al₄W/Al interface. Further heating to 650 °C for one hour holding time lead to form only Al₄W as an interface intermetallic compound, totally consuming the Al₁₂W. Figure (2.10) shows the effect of the heat-treatment. The nano-sized particles powder mixture showed to completely disappear the tungsten particles and form Al₄W and Al₅W. According to researchers the two factors that affect phase formation are heating rate and sintering time, which control the diffusion behavior of the metal particles. The interphase growth was controlled by a diffusion-controlled mechanism during solid state reaction. Tonejc et al. [16] reported that Al₄W forms when rapid solidification of an aluminum-tungsten alloy, and Al₁₂W forms after annealing at temperature above 500 °C. Other high temperature intermetallics that form during the solidification dissolve during annealing. They also mentioned that an equilibrium solid solubility of tungsten in aluminum was formed when heating around 650 °C where at which the lattice parameter of W expands and becomes close value to the aluminum lattice parameter.

Ding et al. [17] successfully synthesized the three tungsten aluminides (Al₁₂W, Al₅W, and Al₄W) using a solid-liquid reaction between aluminum and tungsten using a milling device. The reactions carried out under different temperatures above the aluminum melting point. The tungsten and aluminum powder particles size were 200 mesh (74 μm) and was commercial pure. A 10 g 1:1 mixed powder was loaded into an 80 rpm rotated ball
milling device under a high energy planetary to sinter the powder particle, followed by high speed (400 rpm) milling in an argon atmosphere environment under different temperatures and holding time periods. The XRD peaks show Figure (2.11) that $\text{Al}_{12}\text{W}$, $\text{Al}_5\text{W}$, and $\text{Al}_4\text{W}$ intermetallics formed at 953 K ($\approx 680 $ °C) holding for 24h , 1023 K ($\approx 750 $ °C) holding 12h, and 1163 K ($\approx 890 $ °C) holding 6h respectively.

Harada et al [18] studied the reaction that took place between CVD tungsten film and up to 500 nm aluminum layer that deposited over silicon substrate. The samples were heat treated at 410, 450, and 500 °C, the XRD peaks clearly showed that the $\text{Al}_{12}\text{W}$ interphase was formed as a result of Al/W interaction.

By reviewing the above papers that discussed the formation of the tungsten aluminides, it can be seen that there is no clear understanding that the researchers can agree upon to describe in which environments the aluminides form and grow. In some papers, $\text{Al}_{12}\text{W}$ has been reported to start forming at 500 °C or below [18], while in other papers it formed at higher temperatures ($>550 $ °C) [15][17]. Another disagreement among papers is when the $\text{Al}_4\text{W}$ formed, where most papers reported that the $\text{Al}_4\text{W}$ start forming at a temperature beyond the melting point of aluminum [4][17], only Zhang et al [15] reported that $\text{Al}_4\text{W}$ can be formed at temperatures below the aluminum melting point. The intermetallics formation sequence and growth during heat-treatment is another point of contention, where Wang et al. [4] argue that the $\text{Al}_4\text{W}$ forms first at the Al/W interface followed by the formation of $\text{Al}_{12}\text{W}$ after further heat-treatment, the researchers mentioned
that further heating leads to consumption the $\text{Al}_4\text{W}$ as a credit for the $\text{Al}_{12}\text{W}$. While Zhang et al [15] reported completely an opposite conclusion, where $\text{Al}_{12}\text{W}$ forms at the beginning of the heat-treatment at the Al/W interface followed by the formation of the $\text{Al}_4\text{W}$ as the heat-treatment continued, longer heat treatment causes the $\text{Al}_4\text{W}$ to be consumed for more $\text{Al}_{12}\text{W}$ layers. Most of the papers did not observe $\text{Al}_5\text{W}$ intermetallic, only Ding et al [17] who reported the specific temperatures and holding times under which all of the intermetallic phases including the $\text{Al}_5\text{W}$ are formed. No SEM observation for the $\text{Al}_5\text{W}$ has been presented – only XRD peaks were used as evidence for the formation the $\text{Al}_5\text{W}$. All papers agreed $\text{Al}_4\text{W}$ forms next to the tungsten particles, while the $\text{Al}_{12}\text{W}$ forms next to the aluminum matrix. It should be noted that none of the papers discussed in details the transformation mechanisms that result in the formation of the intermetallics. Fang et al [11] suggested that the interface grows by diffusion-controlled mechanism. No definite evidence or proof of either of the mechanisms has been found.

1.2 CRYSTAL STRUCTURE AND PROPERTIES

Aluminum (fcc, $a=0.405\text{nm}$, $T_{mp}=660^\circ\text{C}$, CTE = $23\times10^{-6}/\text{o}^\circ\text{C}$, $\rho = 2.70 \text{g/cm}^3$ at RT) and tungsten (bcc, $a=0.316\text{nm}$, $T_{mp}=660^\circ\text{C}$, CTE = $4.6\times10^{-6}/\text{o}^\circ\text{C}$, $\rho = 19.35 \text{g/cm}^3$ at RT) have radically different physical properties. The aluminum/tungsten composite properties can benefit from the high strength and hardness of tungsten and the high ductility and
toughness of the aluminum. The intermetallic compounds that form in this system have a complicated crystal structure, which reflected in their properties as well [19]. In the following discussion a number of papers dealing with properties of the aluminum-tungsten composites properties will be reviewed.

A macro size aluminum tungsten composite was prepared by vacuum injection of molten aluminum in between pre-stacked tungsten continuous fibers of 0.008 inch (0.2 mm) diameter. Three groups of composites were prepared, fibers of each group treated differently before injection of the molten aluminum. First group, fibers had been etched and covered by graphite to prevent interaction between the fibers and the matrix. The second group had unetched graphite coated fibers, while the third group used the fibers as they (no etching or coating). The composite had about 12 % volume fraction tungsten and the results show that the etched fibers with graphite had the same strength of the un-etched uncoated fibers, while the composite with un-etched coated fibers exhibited lower strength. This indicated that the key to mechanical strength of the composite is the interface bonding strength [20].
As the volume fraction of the tungsten increases, the tensile strength of the Al/W composite strength increases with a dramatic decrease in the strain to failure in comparison to the aluminum matrix. The 3 volume % of tungsten composite strength reached to about 200 MPa in less than 10 strain % as shown in the Figure (2.12) [11]. The ultimate tensile strength increased by 85 % compared to the unreferenced aluminum, this great improvement is due to formation of the Al$_{12}$W intermetallic after the hot sintering process. The interface of Al$_{12}$W and the aluminum was smooth and clear which indicates it is coherent, the fracture surface had dimples that indicate a ductile fracture.

![Figure 2.12: The strain–stress curve of in situ composites and Al matrix [11].](image-url)
Despite there being no chemical interaction occurring at the interface, the mechanical testing shows there was an improvement in tensile strength (doubled the value) and reduction in engineering strain as the tungsten volume fraction increased compared with aluminum sheets [10]. Vickers microhardness doubled as well with increasing tungsten volume fraction. Figure (2.13), showing a SEM image for the fracture surface of the tensile sample, deep dimples indicate that the fracture of the matrix was ductile. The researchers reported that the composite with 4.2% tungsten volume fraction showed excellent electrical conductivity.

Similarly Rajulapati et al [9] reported that there was an enhanced hardness number of the composite that was prepared at low temperature as the volume fraction of the tungsten increased as shown in the Figure (2.14). Zhang et al [15] showed that the nano hardness number of the sample that had
Al\textsubscript{12}W as an interface is about 50\% greater than the aluminum matrix, while the sample that had Al\textsubscript{4}W as a dominate interphase the nano hardness number reached to 1.55 GPa comparing to Al and W hardness 0.45 and 4.35 GPa respectively. Golkovski [3] showed that Al\textsubscript{4}W has a Nano hardness number between 8-11 GPa.

Figure 2.15: Crystal structures of IMCs: (a) Al\textsubscript{4}W (C1m1), (b) Al\textsubscript{5}W (P63), (c) Al\textsubscript{12}W (Im-3) [12] [19].
Wang et al. [2] in their paper that dealing with tungsten aluminides examined the effect of the high pressure (0-20 GPa) on the mechanical and thermal properties on the intermetallics. The crystal structure of each of the intermetallics is shown in the Figure (2.15), where Al$_4$W has monoclinic structure, Al$_5$W hexagonal and Al$_{12}$W cubic [12] [19].

Table 1 showing the intermetallics crystal parameters and their space groups. It has been seen that the microhardness increased as the pressure increased, where the Al$_4$W and the Al$_{12}$W microhardness numbers 14.26 and 10.39 GPa respectively.

<table>
<thead>
<tr>
<th>Crystal structure properties</th>
<th>Al$_4$W</th>
<th>Al$_5$W</th>
<th>Al$_{12}$W</th>
</tr>
</thead>
<tbody>
<tr>
<td>Space group</td>
<td>Cm</td>
<td>P6322</td>
<td>Im-3</td>
</tr>
<tr>
<td>Number of atoms</td>
<td>15</td>
<td>12</td>
<td>13</td>
</tr>
<tr>
<td>a Å</td>
<td>5.259</td>
<td>4.986</td>
<td>7.599</td>
</tr>
<tr>
<td>b Å</td>
<td>17.77</td>
<td>4.986</td>
<td>7.599</td>
</tr>
<tr>
<td>c Å</td>
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</tr>
<tr>
<td>α°</td>
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<td>90.00</td>
<td>90.00</td>
</tr>
<tr>
<td>β°</td>
<td>100.03</td>
<td>90.00</td>
<td>90.00</td>
</tr>
<tr>
<td>γ°</td>
<td>90.00</td>
<td>120.00</td>
<td>90.00</td>
</tr>
</tbody>
</table>
As shown in the Figure (2.16) the bulk, shear, and elastic (Youngs) moduli and Poisson’s ratio for each individual intermetallic, where all these moduli increased as pressure increased.

![Figure 2.16: Pressure-dependent bulk modulus (B), shear modulus (G), Young's modulus (E): (a) Al₄W, (b) Al₅W, (c) Al₁₂W [2].](image)

Also, the increase in pressure resulted in the Al₄W and Al₁₂W exhibiting brittle to ductile transition as obtained for the ratio of the bulk modulus over shear modulus, Figure (2.17). The researchers suggested that the introduction of extra electrons changes the metallic bond to covalent bond that gives extra strength. The volume also decreased with increased pressure, where Al₄W showed largest anisotropy in modulus especially in c direction of the crystal structure. Finally, the extensive pressure effect on the heat capacity, where the ability to release or store heat of Al₁₂W was much higher than for other intermetallics.

![Figure 2.17: Ductile-to brittle criterion as a function of pressures [2].](image)
Niu et al [21], highlighted a new type of strengthening way when an extra electron in the Al$_{12}$W crystal structure induced covalent bonding instead of metallic bonding. Figure (2.18) showing how the Al$_{12}$X structure forms where Al3 atoms should be removed from the 2x2x2 of aluminum supercell.

The corrosion resistance of the Al$_4$W that was prepared by applying a laser beam over a tungsten covered pure aluminum plate was much higher than the aluminum [13]. The corrosive media was 3.5 wt. % NaCl in 24 h time period. The corrosion pitting was observed on both surfaces of the cladded and non-cladded samples, with higher intensity on the pure aluminum sample. Similarly, Al$_{12}$W showed a good corrosion resistance when immersed into nitrogen de-aerated aqueous solution of 0.05M Na$_2$CO$_3$. The untreated aluminum samples showed higher density of pitting compared with heat treated (80
minutes several temperatures starting from 280 °C to 620 °C) samples. Due to the Al_{12}W not fully covering the sample surface still some pitting was observed [14].

The corrosion behavior and electrical resistance of the Al/W composite where studied by Hukovic et al [22], where a thin layer of pure aluminum and tungsten sputtered over a glass and a sapphire substrate by controlled magnetron source. Under these conditions, the formation of intermetallics was not reported. Hydrochloric acid solution was used as a corrosive environment. The passive file that formed during open circuit potential showed excellent electric and dielectric properties, also the absolute impedance increased with increased tungsten volume fraction. The solid solution of aluminum in tungsten significantly improves the corrosion resistance.

Peets et al [12] studied the physical properties of the tungsten and molybdenum aluminides, where large particles of Al_{4}W and Al_{5}W prepared and tested. The resistivity of all materials shows excellent metallic behavior, but no sign for superconductivity was indicated. Where the residual resistivity ratios (RRR) at 300 K is 47 and 41 for Al_{4}W and Al_{5}W

Figure 2.19: XRD peaks for Al_{4}W and Al_{5}W [12].
respectively. Also, it found that neither the Al₄W nor the Al₅W has phase transition above 100 millikelvin which indicate to the superconductivity, on the other hand Sommerfeld coefficients and Debye temperatures Θ_D for the Al₄W and Al₅W are 4.89 and 2.58 mJ mol⁻¹K⁻² are 444 and 476 K respectively that indicated that the both intermetallics are regular material. The density (g/cm³) of the Al₄W and Al₅W are 5.93 and 5.52 respectively.

Figure (2.15) shows the crystal structure of the Al₄W and Al₅W, Al₄W has more complicated structure composed of layers of decahedral (Al₁₀) cages impeded between hendecahedron (Al₁₁) layers, where inside each cage tungsten atom setting in the middle, Peets report that Al₄W has monoclinic structure in CM space group, with a=5.259, b=17.77, and c=5.228 Å and 100.1088 degree for the beta angle. Al₅W is built from dodecahedral cages (Al₁₂) of which shear edges along ab-plane and corners along c-axis.

The Figure (2.19) also shows the XRD peaks for the both intermetallics.

Adam and Rich [23] studied the crystal structure of both the Al₁₂W and Al₁₂Mo. A sample with 0.25 W and 0.75 Al atomic % composition was prepared and annealed at about 1000 °C for two weeks. The material characterization results showed that the Al₁₂W and Al₄W are in equilibrium. They showed that the Al₁₂W crystal structure is cubic with a lattice parameter of 7.580 Å, the space group T₅h⁻⁵-Im3 where two W atoms are placed in special positions, the calculated density is 3.88 g/cm³.
1.3 INTERFACE GROWTH AND METAL TRANSFORMATION

The metal-metal interface has different forms and aspects depending on the nature of the metals themselves and the environment at which the interface grows. For example, when elements have similar or close atomic size, electronegativity, and valence electrons, in other words obey Hume-Rothery’s rules [24], then a solid solution can form at the interface. Where elements atoms diffuse in each side of the diffusion couple, then a gradient interface will form and grow by diffusion. In other cases, there is a chemical interaction that can occur between metal components which in the case of the Al/W couple, the reaction produces a new phase(s) at the interface. These new phases are called intermetallics, which may have properties that differ from the main component elements. In general, the interfaces could have coherent or incoherent interface with the primary elements, also the growth could have rough or flat (military) transformation [8]. There is no data available for the interface growth mechanism for the tungsten aluminides, but there has been some work that has been done on other aluminides. The following is a survey of literature related to interface formation and growth.

Wang et al [25] studied the thermodynamics of the Al-Co-W ternary system and the sub-binary systems. The enthalpy of formation of the tungsten aluminides were theoretically calculated as shown in the Figure (2.20), where the values were calculated at 0 and 1473 K (-273.15 and 1200 °C). The enthalpy of formation of Al$_{12}$W was about -7.5 KJ/mole-atoms while Al$_4$W and Al$_5$W were much higher about -15.44 KJ/mole-atoms. On the other hand, table (2) shows calculated reaction temperature at which tungsten
aluminides form and become stable. Similarly, studied the thermodynamics of the Al-Co-W ternary system and the sub-binary systems. The enthalpy of formation of the tungsten aluminides were theoretically calculated as shown in the Figure (2.20), where the values were calculated at 0 and 1473 K (-273.15 and 1200 °C). The enthalpy of formation of Al$_{12}$W was about -7.5 KJ/mole-atoms while Al$_4$W and Al$_5$W were much higher about -15.44 KJ/mole-atoms. On the other hand, table (2) shows calculated reaction temperature at which tungsten aluminides form and become stable. Similarly, Dong et al [26] also studied the Al-Co-W system and report the enthalpy of formation of the intermetallics where the it was -5.1 KJ/(g.atom) and -21.15 and -11.83 for Al$_5$W and Al$_4$W respectively.

![Figure 2.20: Calculated standard enthalpies of formation at 298 K of the Al-W system](image)
Abdel-Hamid et al. [27] investigated experimentally and theoretically the growth morphology of the TiB$_2$ intermetallic. Where the compound was prepared by mixing small amounts of the titanium and boron to an aluminum matrix, different liquid solutions were prepared at different temperatures and the intermetallic particles were separated by using electromagnetic phase separation technique. Scanning electron microscopy images (Figure 2.21) showed a large anisotropy of the crystal growth, where large {0001} faces and small {01-10} were developed creating a plate-like morphology, while a hexagonal shape of a large single crystal shape with large {0001} was obtained also. The TiB$_2$ has hexagonal crystal structure as shown in Figure (2.21), where alternatively stacking of the Ti and B atomic plates stacking in the [0001] direction, while a stoichiometry of TiB$_2$ found in the <10-10> direction. The researchers predict the growth morphology and the equilibrium morphology of the TiB$_2$ crystal as shown in the Figure 2.22, where the prediction depends.
on the energy calculation based on Wulff construction. The predicted crystal morphology looked similar to the real one, but there was no evidence that ensured that the calculated faces were the same as the real ones investigated experimentally and theoretically the growth morphology of the TiB₂ intermetallic. Where the compound was prepared by mixing small amounts of the titanium and boron to an aluminum matrix, different liquid solutions were prepared at different temperatures and the intermetallic particles were separated by using electromagnetic phase separation technique. Scanning electron microscopy images (Figure 2.21) showed a large anisotropy of the crystal growth, where large \{0001\} faces and small \{01-10\} were developed creating a plate like morphology, while a hexagonal shape of a large single crystal shape with large \{0001\} was obtained also. The TiB₂ has hexagonal crystal structure as shown in Figure (2.21), where alternatively stacking of the Ti and B atomic plates stacking in the [0001] direction, while a stoichiometry of TiB₂ found in the <10-10> direction. The researchers predict the growth morphology and the equilibrium morphology of the TiB₂ crystal as shown in the Figure 2.22, where the prediction depends on the energy calculation based on Wulff construction. The predicted crystal morphology looked similar to the real one, but there was no evidence that ensured that the calculated faces were the same as the real ones Li et al [28] have continued work on finding an explanation for the growth mechanism of the TiB₂ intermetallic, where samples prepared also into molten aluminum. It had been found that the TiB₂ have hierarchical tower like structure if the reaction rate and atomic diffusion are high which was the observation for the sample that heat-treated at 1200 °C, while the
sample that heated at 800 °C show formation a single crystal of hexagonal plate-like Figure (2.23). The hexagonal shape of the intermetallic was also shown by Han [29], where high-

intensity ultrasound was used to prepare the compound. For the both morphologies, the TiB$_2$ shows a faceted growth which can be explained theoretically due to the high latent heat of fusion to melting point ratio, where and according to Jackson where high-intensity ultrasound was used to prepare the compound. For the both morphologies, the TiB$_2$ shows a faceted growth which can be explained theoretically due to the high latent heat of fusion to melting point ratio, where and according to Jackson [30] if the Jackson alpha factor exceeds 2.0 then an intermetallic should have faceted growth. They also referred that due to the variety of the interplanar distancing among planes $\{0001\} > \{10\overline{1}0\} > \{10\overline{1}1\}$ the

Figure 2.24: Schematic illustration of the growth of TiB$_2$ dendrite in aluminum melt [28].
TiB$_2$ tends to be hexagonal. As sample preparation temperature increases the hierarchical tower like morphology will be preferred to be observed, where the growth controlled by two steps: intermittent two-dimensional nucleation and continuous lateral spreading of layers as shown in Figure (2.24).

Min and Xiangfa [31] studied the morphology and growth mechanism of the aluminum-phosphorus (AlP) intermetallic, where it prepared by heating Al-15Si-2.5P (wt%) alloy at 1600 °C using high frequency induction heating in an argon atmosphere. The zinc-blende like crystal structure of the AlP (Figure 2.25), showing that the [111] direction has the higher atomic density in comparison with other directions. The researchers suggest that due to the high entropy of fusion and large Jackson alpha factor (>2), the AlP has faceted growth as shown in the Figure 2.26, where the AlP hexagonal layer was thin (1μm) and growth layer by layer. They added that the faceted edges of a

Figure 2.26: FESEM images of A in the ternary Al-15Si-2.5P alloy, (a) hexagonal platelet (b) twin planes [31].
single crystal AlP were considered as a necessary requirement for twinning, therefore a faceted AlP hexagonal like plated with twin planes.

Li et al [6] have investigated into three direction morphology and growth of the Al₆Mn intermetallic. An alloy of Al-3at%Mn where melted and heat treated at 925 °C for 30 minutes using directional induction furnace with growth rate of 1 μm/s followed by quenching. Microscope imaging shows that the growth of the intermetallic was faceted with strong anisotropy where it has been found that the morphology depends on the growth conditions. According to the crystallographic theory, the [001] direction of the Al₆Mn structure Figure (2.27) is the preferred growth direction where an octahedron shape formed by bonding eight close-packed planes (011) and (101). Where the growth rate was slow in the [101] and [011] due to the high density in the (011) and (101), that led the other less density planes to disappear as the growth proceeded due to their fast growth rate. The researchers suggest that the spiral growth mechanism is aided by the screw dislocation for the intermetallic compound, as shown in the Figure (2.28). The step size depends on the scale atomic clusters existing in the melt and crystal structure compound.
Figure 2.27: The unit cell of Al6Mn intermetallic compound (a); complicated composed of ten Al atoms and ball-and-stick models, steel balls represent the Al atoms and a Mn atom locates in the center (b); and projections of Al6Mn unit cell along the [100] (c) and [010] (d) directions respectively, the numbers on atoms represents the number of same kind superimposed atoms [6].
The three-dimensional morphologies of the Mg$_2$Si intermetallic were studied by Li et al [32]. Where commercially pure elements of Al, Si, and Mg were mixed in three different weight percent. The mixtures melted together using a frequency induction furnace and then the alloys were remelted at 800 °C and held for 30 minutes. The scanning electron microscopy images (Figure 2.29) showed there were different shapes of the Mg$_2$Si such as hopper, cube, dendrite, and form faceted octahedron. The researchers referred to the faceted growth of the compound as due to high fusion entropy and large Jackson’s factor. The Mg$_2$Si has FCC crystal structure with lattice parameter of 0.639 nm Figure (32), where silicon atoms located at corners and face center while the magnesium atoms occupied the eight tetrahedral interstices. According to the Bravais-Friedel law [33] and as the authors
mentioned, the <100> direction has the highest growth rate among other directions due to high binding energy (lowest planner density) of the [100] compared to [110] and [111] planes, where the equilibrium crystal had that only \{111\} faces appeared. Weatherly [34], had shown that the Mg$_2$Si intermetallic has plate-like morphology, where the growth ledges lying at the top surface of the plate along the close-packed direction, Figure (2.31).

Wang et al [35] have studied the three dimension morphology and growth mechanism of the alpha-Al(FeMnCr)Si intermetallic crystal. Samples were prepared by melting aluminum- 20 wt% silicon master alloy at 780 °C by using frequency induction melting, then other alloying elements (Fe, Mn, and Cr) were added and held the mixture at 810 °C for 30 minutes. SEM images as shown in the Figure (2.31 a) showed that cube shape was the morphology of the intermetallic. No crystal structure was shown for the compound,

![Figure 2.31: (a) Growth ledges at on Mg$_2$Si plate. (b) Schematic diagram showing those ledges lying at the top surface of the plate [35].](image)
but the authors suggest that migration of the {100} was slow so the alpha-Al(FeMnCr)Si intermetallic exhibit perfect (100) planes.

Kinetics of reaction and intermetallics formation between titanium and aluminum were studied by Kar et al. have studied the three-dimension morphology and growth mechanism of the alpha-Al(FeMnCr)Si intermetallic crystal. Samples were prepared by melting aluminum- 20 wt% silicon master alloy at 780 °C by using frequency induction melting, then other alloying elements (Fe, Mn, and Cr) were added and held the mixture at 810 °C for 30 minutes. SEM images as shown in the Figure (2.31 a) showed that cube shape was the morphology of the intermetallic. No crystal structure was shown for the compound, but the authors suggest that migration of the {100} was slow so the alpha-Al(FeMnCr)Si intermetallic exhibit perfect (100) planes.

Kinetics of reaction and intermetallics formation between titanium and aluminum were studied by Kar et al [36]. Samples of dissimilar metals were prepared by using stir friction welding (SFW) for assembly of two aluminum plates and titanium foil has been
inserted in between. The samples then heat treated different temperatures and holding times, a series of intermetallics compounds were formed at Ti/Al interface. At low temperatures and at 24 h holding time Al₅Ti and Al₅Si were formed while at 650 °C Al₅Ti was formed. The interface layer thickness (Figure 2.32) proportional to the holding time, the interface has rough edges where the component concentration changes with distance.

The author relates the formation and growth of the intermetallics to the titanium foil was fragmenting during welding, and the severe deformation during the FSW resulting in a high density of defects (dislocations and twinning) that formed as a result of plastic deformation, which enhanced atoms diffusion by pipe diffusion.

Growth kinetics of interfacial intermetallic compounds between solid steel and molten aluminum were studied by Rong et al [37]. Where 1 mm thickness Q235 steel sheet has been brazed-welded to commercially pure aluminum. The samples preheated to 600 °C for 5 seconds then continued heating to 700-900 °C at a fast rate and the hold time to 9 seconds. Figure (2.33) shows the Al/Steel interface where two compounds η (Fe₂Al₅) and θ (Fe₄Al₁₃) were formed for the steel side of the interface. The interfaces (Steel/ η, η/ θ, and θ/Al) had rough edges, where the η had a tongue-like morphology and the θ had a needle-like morphology. The growth of the η phase growth was into the steel side while the growth of θ growth was into the aluminum.

Figure 2.33: OM micrograph [32].
side. The EDS analysis showed that there is a gradual change in the aluminum concentration along the interface thickness. Cracks had appeared on the steel side of the interface for samples that heat-treated at 750 °C and above. The researchers mentioned that the growth was governed by interfacial reaction and diffusion.
3. MATERIALS AND EXPERIMENTAL PROCEDURES

In order to create and examine the aluminum/tungsten composite, a series of experimental procedures were used. Three different sample preparation techniques were used to give an examination of how the main composite components react with each other. The samples were then heat-treated at different temperatures and times, followed by a series of characterization processes to understand how the different intermetallic compounds form and grow. The following sections describe the materials, sample preparation processes, and testing techniques that were employed.

Figure 3.1: Al-W phase diagram [16]
3.1 MATERIALS

Aluminum was chosen to be the matrix metal and the tungsten was chosen to be the reinforcing phase for the composite. These metals were chosen due to the differences in their properties, where the aluminum has low density and high thermal and electrical conductivity, while tungsten has a much higher strength and density compared to aluminum. The composite metals components properties are shown in Table 3.1. The composite takes advantage of the differences in the properties of the two metals. Figure 3.1 shows the phase diagram of the binary aluminum-tungsten system. At room temperature there are three intermetallic compounds, Al₁₂W, Al₅W, and Al₄W. The phase diagrams show that Al₁₂W is a line compound, whereas Al₅W and Al₄W have some variation in composition, and there is an overlap between the ranges of composition of these two intermetallics. Further, the consolidation and heat treatment processes used result in the formation of intermetallic compounds which are generally stronger than aluminum and less dense than tungsten, leading to the possibility that high strength to weight intermetallic reinforcements may be possible in this alloy system.
3.1.1 Aluminum

The matrix metal of the composite was aluminum. Two different forms aluminum were used to prepare samples: powder and solid metal.

a. Commercial pure aluminum powder of average mesh size of about 325 (44 μm) was provided by Atlantic Equipment Engineers company. This powder was used to prepare the composite by the spark plasma sintering process.

b. AlSi10Mg an additive manufacturing aluminum alloy powder where used for the laser powder bed fusion process was provided by AMPAL, INC company. The powder has been sieved to 30 μm average particles size.

### Table 3.1: Composite metal components properties.

<table>
<thead>
<tr>
<th>Properties</th>
<th>W</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting point °C</td>
<td>3410</td>
<td>660</td>
</tr>
<tr>
<td>Density (g/cm3)</td>
<td>19.25</td>
<td>2.7</td>
</tr>
<tr>
<td>Thermal conductivity (w/m-K)</td>
<td>174</td>
<td>205</td>
</tr>
<tr>
<td>CTE at 25°C (μm/m-K)</td>
<td>4.5</td>
<td>22</td>
</tr>
<tr>
<td>Crystal Structure</td>
<td>BCC</td>
<td>FCC</td>
</tr>
<tr>
<td>Nano-hardness (GPa)</td>
<td>9.7-13.0</td>
<td>0.4</td>
</tr>
<tr>
<td>Specific Hardness (GPa/(g/cm3))</td>
<td>0.5-0.67</td>
<td>0.15</td>
</tr>
</tbody>
</table>
ER4043 aluminum solid welding wire was used to deposit aluminum over a tungsten plate by using a metal inert gas welding process. The wire diameter was 0.8 mm (0.035 inch) and the typical chemical composition in a weight percent is shown in the table (3.1), the wire was provided by McMaster-Carr Company.

Table 3.3. Aluminum ER4043 welding wire composition

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum (Al)</td>
<td>Remainder</td>
</tr>
<tr>
<td>Beryllium (Be)</td>
<td>0.0008 max</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>0.30 max</td>
</tr>
<tr>
<td>Iron (Fe)</td>
<td>0.80 max</td>
</tr>
<tr>
<td>Magnesium (Mg)</td>
<td>0.05 max</td>
</tr>
<tr>
<td>Manganese (Mn)</td>
<td>0.05 max</td>
</tr>
<tr>
<td>Silicon (Si)</td>
<td>4.5-6.0</td>
</tr>
<tr>
<td>Titanium (Ti)</td>
<td>0.20 max</td>
</tr>
<tr>
<td>Zinc (Zn)</td>
<td>0.10 max</td>
</tr>
<tr>
<td>Other elements</td>
<td>0.05 max each &amp; 0.15 max total</td>
</tr>
</tbody>
</table>

d. Commercial pure 1100 aluminum wire 3.2 mm (0.126 inch) in diameter. The wire was provided by McMaster-Carr Company. The wire was used to prepare cast samples.
3.1.2 Tungsten

The tungsten was the reinforcing phase of the composite. It was also used in two forms: powder and sheet.

e. Commercial pure tungsten powder with an average particles size of 30 μm, the powder was provided by US Research Nanomaterials, Inc. The powder was used to prepare samples by both the spark plasma sintering (SPS) and the laser powder bed fusion (LPBF) processes.

f. Commercial pure tungsten sheet with dimensions of 101.6 x 304.8 x 0.254 mm (4 x 12 x 0.01 inch) supplied by the REMBAR Company, LLC used to prepare samples by SPS.

3.2 EXPERIMENTAL PROCEDURES

Experimental procedures include the processes that were used to create the composite, heat treatment, and characterization.

3.2.1 Consolidation

Three consolidation processes were used to prepare the metal-metal composite samples.
3.2.1.1 Welding and Melting:

A gas metal arc welding (GMAW) welder shown in Figure (3.2) was used to deposit aluminum ER4043 welding wire (Table 3.3) over a 4 mm thick tungsten strip. Argon shielding gas at a flow rate of 12 liters per minute (25 cubic feet per hour) was used to protect the weld zone. Direct current of 190 A was used, where the electrode wire was positive, and the tungsten plate was connected to the negative terminal. The arc welding voltage was in the range of 20 to 25 Volts, while the wire feed speed was about 112 mm per second. The aim of choosing this process was essential to confirm if the tungsten is weldable to aluminum by a fusion process. The welding process was carried out in the Wright State University/Mechanical and Materials Engineering (MME) department’s laboratories.

Casting was another way used to prepare the composite, where a 1100 aluminum wire was melted in an air furnace at 800 °C then tungsten particles were added to the molten metal. After holding for ten minutes in the furnace at 800 °C the metals mixture (10 at % tungsten) were poured into graphite mold flowed by water quenching.

3.2.1.2 Spark Plasma sintering (SPS):
As a powder consolidation technology, SPS is a process that uses a pulsed direct electric current to heat up a compacted conductive powder while applying a uniaxial mechanical pressure. High-density graphite die is used and the powder compact is compressed between two graphite rams [38]. The SPS process circle has the main four stages as shown in Figure (3.3). The first stage includes removing gases and creating a vacuum. In the second stage, an axial mechanical pressure is applied and maintained until the end of the process. During the third stage, the specimen is subject resistance heating using a pulsed (on and off) direct current. The fourth stage is cooling down to room temperature. When current is applied, a spark is generated in the gaps or contact points between metal particles, resulting in a high temperature and local melting of the particle surfaces. The melted regions are transferred by the electric current flow to form necks between the powder particles, Figure (3.4) [39].
This process was used to create the aluminum-tungsten composite with the minimum reaction during sample preparation heating. The resultant samples had no reaction between tungsten and aluminum which occurred during the consolidation process due to the relatively short-heating period. This process was carried out at Wright Patterson Air Force Base laboratories. Powder mixture of pure aluminum and 10 atomic % tungsten and aluminum powder/tungsten sheet samples were consolidated at 550 °C in 100 °C/min heating rate for 2 minutes under 20 MPa applied pressure.

Figure 3.4: Mechanism of neck formation by spark plasma [39].
3.2.1.3 Laser powder bed fusion (LPBF)

This is an additive manufacturing process in which a three dimensions part can be created by 3D printing. A printing process starts by drawing the desired part design on CAD-based software, then by slicing software the design is sliced to a number of slices along the part height. As shown in Figure (3.4) a thin layer of metal powder feed from cartridges spread out and compacted over machine build plate by different techniques depends on the machine type, the powder layer thickness typically between 0.075 to 0.1 mm. A laser beam with different energy settings depends on the laser source and machine manufacture scans over the compacted powder layer. The laser scan path is determined by the CAD part design, where powder particles melted and consolidated with layers underneath it. After that, the build plate is lowered preparing for the coming layer, the amount at which the build plate moves is the same as the next powder layer thickness. The process is performed under the influence of heat and shielding environment. Printing settings like laser power, laser scan speed, powder layer thickness, powder particle size and shape, and others can be varied and depend on the metal powder itself and machine manufacturer [40][41].
The LPBF samples (4 cubic mm) have been prepared at Materials Resources, LLC (MRL), where samples with three different tungsten volume fractions (10, 15, and 20 atomic %) were printed out. The AlSi10Mg was the commercial aluminum powder alloy that was used as a matrix material that mixed in advance with tungsten powder, the chemical composition of the aluminum alloy is shown in Table 3.1.

3.2.2 Heat treatment.

The consolidated samples were heat-treated in a calibrated air furnace over a wide range of temperatures and holding time. The heating temperatures were chosen in the vicinity of the melting point of the matrix alloy. It is worth to be mentioned here is that the melting point of the used aluminum alloys are not the same, where AlSi10Mg alloy has a melting point of 570 °C while pure aluminum melts at 660 °C, and the 1100 aluminum alloy has a melting point of 650 °C melting point. Table 3.4 shows the heat treatment conditions for each type of consolidation process.
Table 3.4: Heat-treatment conditions

<table>
<thead>
<tr>
<th>Process</th>
<th>Matrix melting point °C</th>
<th>Temperature °C</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>LPBF</td>
<td>570</td>
<td>500</td>
<td>24 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>550</td>
<td>6 days</td>
</tr>
<tr>
<td></td>
<td></td>
<td>580</td>
<td>3 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>600</td>
<td>2 days</td>
</tr>
<tr>
<td></td>
<td></td>
<td>650</td>
<td>12 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>700</td>
<td>12 hours</td>
</tr>
<tr>
<td>SPS</td>
<td>660</td>
<td>600</td>
<td>1 hour</td>
</tr>
<tr>
<td></td>
<td></td>
<td>600</td>
<td>10 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>600</td>
<td>20 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>630</td>
<td>2 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>650</td>
<td>1 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>700</td>
<td>1 hours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>800</td>
<td>10 minutes</td>
</tr>
<tr>
<td>MIG</td>
<td>600</td>
<td>500</td>
<td>24 hours</td>
</tr>
</tbody>
</table>
3.2.3 Characterization.

For the metallurgical characterization of the composite samples in general and the aluminum/tungsten interface region in more specific an electronic microscope and X-ray diffraction devices were used. These characterization processes helped to catch up any changes that take place at the interface area as a result of the heat-treatment, where formation and growth of the intermetallic(s) can be recognized. The characterization devices and their features are listed below preceded by samples preparation procedure.

3.2.3.1 Sample preparation procedure.

The aluminum tungsten composite samples that have been prepared by the different consolidation processes, have been prepared for the microscopic examination. The sample preparation procedure is as follows.

- Using an electrical discharge machining (EDM) to cut the as received samples to smaller sized samples preparing for the heat-treatment.
- Samples then hot mounted using SimpliMet XPS1 mounting press, KonductoMet conductive filled phenolic mounting compound was used as a mounting martial. Mounting temperature was 150 °C and 4200 Psi as the pressures were applied along 3 minutes followed by water cooling.
- Buehler AutoMet 250 grinder and polisher machine was used to grind and polish the mounted samples. Silicon carbide (SiC) grinding papers grit 320, 400, 600 and 800 were
used in sequence to grind the samples under 3 Newton load and 150 rpm rotation speed. The grinding process was done under running deionized water.

MicroCloth polishing cloth was used to polish the grinded samples. Polishing continued for about an hour under 1 Newton load and 80 rpm disk rotation speed. Different types and sizes of polishing suspensions were used as a polisher, MetaDi monocrystalline diamond suspension with sizes of 9, 6, and 1 μm were used in sequence. MasterMet 2 non-Crystallizing colloidal silica polishing suspension was applied then as a final polisher, it has a pH of 9.8 with particle size of 0.02 μm so it works as a mechanical-chemical polisher. The samples then rinsed with running water and oven-dried.

3.2.3.2 Optical microscope

Nikon Epiphot Microscope optical microscope model number B1 was used with different magnification lances. Polished samples were examined using this microscope to get an initial imaging about the composite cross section. Where optical images give information about if the tungsten particles got melted or affected by consolidation processes. Also, aluminum/tungsten interface can also be observed. Samples defects such as porosities, prose, and cavities also can be seen. This device is found in the Wright State University / Mechanical and Materials Engineering Department’s laboratories.

3.2.3.3 Scanning electron microscopy (SEM).

JEOL high resolution electron microscope model 7900 figure (3.5) was used to investigate the composite components behavior for the as consolidated and for the heat-
treated samples. Two types of signal detectors were used: backscattered electrons (BSE) and secondary electron (SE) to image with different magnifications the aluminum/tungsten interface area. Where the tungsten aluminides formed and growth. A chemical analysis along the aluminum/tungsten interfacial area and for an individual point analysis where obtained using EDAX Energy-dispersive X-ray spectroscopy (EDS) detector. The SEM beam energy was 5 Volts for the SE image and 15 Volts BSE, while beam diameter was in the range of 10-13. This device is found in the Wright State University / Mechanical and Materials Engineering Department’s laboratories.
3.2.3.4 X-ray diffraction (XRD).

Rigaku SmartLab X-ray diffractometer instrument shown in the Figure (3.6) was used for the as-received and for the heat-treated composite solid samples at room temperature to identify any phase changes that include create a new phase(s) or consume others. Copper was the target of the X-ray, the X-ray type was Kα with the wavelength of 1.54059 Å for the Kα₁ and 1.54414 Å for the Kα₂, while tube voltage was 40 kV and tube current were 44 mA. These were the source specification, while the scan conditions were: θ/2θ for the scan-axis, scan method is one direction scan, scan range was 5-100°, and with a step width of 0.001° with a scan speed of 5°/min. SmartLab studio II software was used to analysis and identify the observed peeks for each sample.
This test was carried out at the Wright State University / Mechanical and Materials Engineering Department’s laboratories.
4. EXPERIMENTAL RESULTS

In this chapter, the macroscopic and microscopic observations beside phase and chemical identifications of all the tested samples will be presented. The obtained microscopic results were focused on the aluminum/tungsten interface area at which the tungsten aluminides were formed and grew. The changes in the main composite components' appearances and morphologies were the macroscopic observations focusing goal. Chemical analysis and phase identification tests help in identifying the formed intermetallics. Commercial pure aluminum was the matrix metal for the SPS samples, while aluminum-silicon based alloys were the matrix material that was used for the LPBF and welded samples, Table 1 and 2 showed the composition of the LPBF and welding wire alloy composition respectively.

Table 4.1: LPBF aluminum alloy powder composition.

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum (Al)</td>
<td>Remainder</td>
</tr>
<tr>
<td>Silicon (Si)</td>
<td>10.0</td>
</tr>
<tr>
<td>Magnesium (Mg)</td>
<td>0.34</td>
</tr>
<tr>
<td>Manganese (Mn)</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Fe</td>
<td>0.12</td>
</tr>
<tr>
<td>Titanium (Ti)</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Zinc (Zn)</td>
<td>&lt;0.01</td>
</tr>
</tbody>
</table>
4.1 WELDING AND MELTING

4.1.1 Macroscopic observations

Gas Metal Arc Welding (GMAW) was used to check if the aluminum and tungsten are compatible with each other before using other consolidation processes. A lap welding joint of two tungsten strips was prepared by fusion welded by using aluminum filler wire, where no preheating was applied for the tungsten strips. During the welding process, welding sparks were extremely dense with relatively large size with a yellowish color. The final joint appearance in Figure 4.1 can be shown that the aluminum has low wetting.

Table 4.2: Aluminum ER4043 welding wire composition.

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum (Al)</td>
<td>Remainder</td>
</tr>
<tr>
<td>Beryllium (Be)</td>
<td>0.0008 max</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>0.30 max</td>
</tr>
<tr>
<td>Iron (Fe)</td>
<td>0.80 max</td>
</tr>
<tr>
<td>Magnesium (Mg)</td>
<td>0.05 max</td>
</tr>
<tr>
<td>Manganese (Mn)</td>
<td>0.05 max</td>
</tr>
<tr>
<td>Silicon (Si)</td>
<td>4.5-6.0</td>
</tr>
<tr>
<td>Titanium (Ti)</td>
<td>0.20 max</td>
</tr>
<tr>
<td>Zinc (Zn)</td>
<td>0.10 max</td>
</tr>
</tbody>
</table>

Figure 4.1: GMAW of the aluminum/tungsten welded sample welded sample
characteristics with the tungsten. The melted aluminum preferred to bead up and agglomerate along the joint resulting in unfilled regions along the joint where whole aluminum spheres (contact angle about 180°) can be found. Though good wettability is an essential factor for a good weld, and the aluminum/tungsten lap joint had many unfilled metal areas, the joint showed

Excellent behavior under tensile test where observed, three tensile tests of the joint result in breaking the tungsten strips away from the joining area without affecting the welding zone as shown in Figure 4.1. Another observation is the tungsten strip edges in the joining area did not melt and kept straight. No cracks in this level were obtained.

4.1.2 Optical Microscopic observations.

Figure 4.2 shows the optical microscopic images of the cross section of the aluminum / tungsten welded sample. It can be seen that the tungsten did not melt during welding where the strip edges look sharp and straight. The higher magnification image

![Optical Microscopic Images](image)

Figure 4.2: Optical microscopic images of the aluminum / tungsten welded sample, (a) low magnification, (b) high magnification
details the interface area, where a smooth phase was grown in the aluminum side of the interface and precipitated at grain boundaries. The interface looks non-uniform along the welding joint and it has straight and sharp edges.

4.1.3 Scanning electron microscopic (SEM) observation.

SEM imaging give more details of the interface area. The SEM images shown in the Figure (4.3) for the as-welded sample shows the interface grows into the aluminum alloy side of the joint with curved and rough edges in some places, while it has sharp edges in other regions. The interface is not uniform, with an average thickness of about 5 μm. Color changes through the interface with light-colored regions that appear close to the tungsten side of the interface, indicating that there was more than one intermetallic compound formed at the interface area during welding. Cracks were observed in the tungsten strip edges, they are filled with molten filler metal alloy which converted later to an intermetallic phase.

![SEM images showing interface and intermetallics](image)

**Figure 4.3:** SEM microscope image for the as welded sample, (a) low magnification, (b) high magnification
A microstructure from the 24-hour heat-treated welded sample at 500 °C, shown in Figure (4.4), shows the growth of a new phase with dark gray color at the region of the aluminum/intermetallic interface; the interface now has a more uniform appearance and straight edges in the aluminum side. The light-colored regions have been disappeared.

![Figure 4.4: SEM microscope image for the heat-treated welded sample](image)

4.1.4 Energy Dispersive X-Ray Spectroscopy (EDS).

Both point and line analyses were carried out for both the as-welded and heat-treated samples to understand the chemical composition variation around the aluminum/ tungsten interface. From Figure (4.4) which shows the EDS point chemical analysis of the aluminum/tungsten interface, it can be seen that the tungsten atomic percent along with the interface has a range of 13.51 to 14.38, while it goes to 21.63% in spot number 2 for the light-colored stain close to tungsten side of the interface. It can be noticed that the silicon
atomic percent in the interface area is higher than its percent in the aluminum matrix. Also,
there is a small amount of aluminum dissolved in the tungsten plate side of the interface,
while there was a 26.95 atomic % of silicon in tungsten.

Table 4.3: EDS spots chemical composition of the as welded sample

<table>
<thead>
<tr>
<th>Spot</th>
<th>Element</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Tungsten</td>
<td>70.79</td>
</tr>
<tr>
<td></td>
<td>Aluminum</td>
<td>2.25</td>
</tr>
<tr>
<td></td>
<td>Silicon</td>
<td>26.95</td>
</tr>
<tr>
<td>2</td>
<td>Tungsten</td>
<td>21.63</td>
</tr>
<tr>
<td></td>
<td>Aluminum</td>
<td>30.26</td>
</tr>
<tr>
<td></td>
<td>Silicon</td>
<td>48.11</td>
</tr>
<tr>
<td>3-8</td>
<td>Tungsten</td>
<td>13.51-14.38</td>
</tr>
<tr>
<td></td>
<td>Aluminum</td>
<td>81.11-80.08</td>
</tr>
<tr>
<td></td>
<td>Silicon</td>
<td>5.38-5.55</td>
</tr>
<tr>
<td>4</td>
<td>Tungsten</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>Aluminum</td>
<td>99.5</td>
</tr>
<tr>
<td></td>
<td>Silicon</td>
<td>0.25</td>
</tr>
</tbody>
</table>

Figure 4.5: EDS point analysis for the as welded sample

An EDS line analysis of the heat-treated weld sample is shown in Figure 4.6, where
the analysis line started from the tungsten plate side and ended in the aluminum alloy
matrix side all the way through the interface area. In the EDS graph, the atomic percent is
represented by Y-axis, while X-axis is shows the distance in micrometers. It can be seen
that the tungsten was in the range of 25 % along the major portion of the interface then it
dramatically dropped down to about 10 % in the last few micrometers (≈ 2 μm) of the
interface before it became zero % on the aluminum side. About 15 % of silicon was
detected in the tungsten side of the interface and then it kept at about 10% along with the interface before it dropped down to zero percent on the aluminum side.

Figure 4.6: EDS line analysis of the heat-treated welded sample.
4.1.5 X-Ray diffraction (XRD) observation.

The XRD observed peaks for the as welded and heat treated samples are shown in the Figure (4.7). The X-axis represents the 2-theta value in degree units, while the peaks intensity with arbitrary units are shown in the Y-axis. It can be seen that the Al₄W intermetallic phase has been formed due to reaction between aluminum and tungsten for the as welded sample and for the heat-treated sample as well. Tungsten silicide, Andalusite, and other unknown phases were detected for the as-welded sample. These phase peaks disappeared for the as heat-treated sample. Al₁₂W phase peaks were shown for the heat-treated sample while it is not shown for the as welded sample. The main composite components peaks are clearly observing for the both samples.

![Figure 4.7: XRD peak analysis of the welded sample](image-url)
4.2 SPARK PLASMA SINTERING (SPS)

4.2.1 Macroscopic observations

Two different forms of tungsten were applied into aluminum powder (foil and powder) for the SPS samples. Generally, the cross-section for the as received and for the heat-treated powder samples was homogenous and no macroscopic defects were obtained. For samples that heat treated above the melting point of Al (700 °C for 1 hour), some porosity was observed due to melting. For the as received tungsten foil samples, aluminum was perfectly surrounding the foil and no defects were observed. However, after heat treating at temperature below aluminum melting point (500 °C for 24 h), the aluminum/tungsten joint failed as shown in Figure 4.8, where the aluminum shifts away from the tungsten. Tungsten foils get oxidized and a light green color layer covers the tungsten surface. Moreover, tungsten foil became parts where the foil had been sliced up.

Figure 4.8: Macroscopic observations for the SPS (a) as received sample (b) heat-treated sample
4.2.2 Optical Microscopic observations.

Optical Microscopic observations give more details than macroscopic observations, such as information about tungsten particles’ shape, size and their distribution. In addition to how the aluminum matrix material looks like. Not too much information about the interface area can be obtained. Figure 4.9 shows the as received SPS optical microscopic image, where the tungsten particles appear as small dots spread out in the aluminum matrix. The higher magnification image shows the tungsten particle cross section clearly. It can be seen that the particles have different sizes and shapes where the majority have a circular cross section. The particles were well distributed through the cross section and no particles agglomeration was detected. The aluminum matrix was homogenous, and no defects were found. The higher magnification image shows that the interface area is smooth which indicates that no reaction takes place at the aluminum/tungsten interface area.

Figure 4.9: As received SPS optical microscopic images (a) low magnification (b) high magnification.
Figure (4.10 a) showing the SPS sample that heat treated at 600 °C for 10 hours (below aluminum melting point). There are some observations that can be noticed comparing to the as-received sample image. The area that surrounds the tungsten particle has a darker appearance compared with the matrix material which indicates that intermetallic phases grow at the tungsten/aluminum interface area. The Figure (4.10 b) shows the image of the SPS sample that is heat-treated at temperature (700 °C for 30 minutes) above the aluminum melting point showing an obvious difference in observation compared with the low temperature heat-treated. There are no tungsten particles can be found, where it seems that all tungsten metal has been consumed and transformed to an intermetallic compound which appears in a dark region that spreads out in the aluminum matrix.

The microstructure of the aluminum / tungsten sample that is heat treated at 800 °C for 30 minutes is shown in the Figure 4.11. The aluminum completely melted and become liquid during heat treatment where the sample had a drop shape after heat-treatment is
accomplished. It can be seen that the tungsten particles have completely disappeared and there are dark precipitates (tungsten aluminides as later approved by using XRD and EDS) Figure (4.10 a) showing the SPS sample that heat treated at 600 °C for 10 hours (below aluminum melting point), where there is no obvious difference can be observed when comparing with the as received sample image, except the area that surrounding the tungsten particle has darker appearance comparing with matrix which indicate that intermetallic phases grow at the tungsten/aluminum interface area. The Figure (4.10 b) shows the image of the SPS sample that is heat-treated at temperature (700 °C for 30 minutes) beyond aluminum melting point showing an obvious difference in observation compared with the low temperature heat-treated. Where there are no tungsten particles can be found, where it seems that all tungsten metal has been consumed and transformed to an intermetallic compound which appears in a dark region that spreads out in the aluminum matrix.
The microstructure of the aluminum / tungsten sample that is heat treated at 800 °C for 30 minutes is shown in the Figure 4.11. Where the aluminum completely melted and became liquid during sample preparation. It can be seen that the tungsten particles have completely disappeared and there are dark precipitates (tungsten aluminides) is precipitating in the aluminum grain boundaries, also porosities can be seen in the grain boundaries region.

Figure 4.11: Heat treated SPS sample at 800 °C for 30 minutes
4.2.3 Scanning electron microscopic (SEM) observation.

The SEM observation images gave more details than the optical images due to the high magnification ability. Figure (4.12) shows the SEM image of the as received SPS sample. It shows that the most tungsten particles (white circles) have a circular cross section while other particles have irregular shape. The aluminum matrix (the gray background) looks homogenous and no defects are found. The low magnification image shows that the tungsten/aluminum interface is smooth and clear, while the high magnification image of the interface shows that the intermetallic (light gray color) has grown in a small portion (less than 0.5 μm length) of the interface. No clear features of the interphase can be obtained, but the interface looks rough.

Figure (4.13) showing SEM image of the SPS sample that has been heat-treated at 600 °C (below aluminum melting point) for 10 and 20 hours respectively. It can be seen that there is an interfacial reaction occurring at the interface between tungsten particles.
(white circles) and aluminum matrix (dark gray) where tungsten aluminide (light gray) grows in the aluminum side of the interface. The interface for the both samples has rough appearance. The 10 hours holding time sample interface has an average thickness about 2-3 μm. It reached to bout 8-10 μm thick for the 20 hours holding time.

Figure 4.13: SEM images for SPS sample heat treated at 600 °C (a) 10 hours, (b) 20 hours
Figure 4.14 shows SPS samples that heat-treated at 700 °C (beyond aluminum melting point) for 30 minutes. It can be seen that all tungsten particles have been consumed and intermetallic compounds (the dark gray) grown instead, the intermetallic has sharp and straight edges and the average size is much bigger than tungsten particles. The grown intermetallic particles that were found close to each other in the matrix are fused together. Some defects were formed in the aluminum matrix as shown due to melting during sample preparation.

Figure 4.14: SEM images for SPS sample heat treated at 700 °C for 30 minutes
Figure 4.15 show the microstructure of the aluminum/tungsten sample that heat-treated at 800 °C for 30 minutes. In this image it can be seen that the tungsten has been consumed and no particles can be found. Tungsten aluminide phase has been precipitate in aluminum grain boundaries.

4.2.4 Energy Dispersive X-Ray Spectroscopy (EDS).

Chemical analysis for the composite components and the aluminum/tungsten interface where obtained by using the EDS detector. The results of this test showed what type of the tungsten aluminide(s) was formed at the interface area. Figure (4.16) showing the EDS point analysis of the SPS sample that heat-treated at 600 °C (below matrix melting point) for 10 hours, where the analysis results show that the chemical composition of the spot 1 is 100% tungsten while it was 100% aluminum for the spot 4. The tungsten atomic percent for the spot 2 and 3 were 7.65 and 6.95 respectively with error around 6%.

Figure (4.17) shows the EDS chemical composition analysis for the SPS sample that was heat-treated at 700 °C (beyond matrix melting point) for 30 minutes. The whole tungsten particles got consumed and intermetallic got formed. The atomic percent range of the tungsten was between 5.4 and 7.65 for spots 1 and two respectively.
Other SPS samples that are gradually heat-treated at higher temperatures. Tungsten aluminides precipitated at grain bounders, shows the same chemical analysis of the interface compounded.

Table 4.4: EDS spots chemical composition

<table>
<thead>
<tr>
<th>Spot</th>
<th>Element</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Tungsten</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>Aluminum</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>Tungsten</td>
<td>92.35</td>
</tr>
<tr>
<td></td>
<td>Aluminum</td>
<td>7.65</td>
</tr>
<tr>
<td>3</td>
<td>Tungsten</td>
<td>93.05</td>
</tr>
<tr>
<td></td>
<td>Aluminum</td>
<td>6.95</td>
</tr>
<tr>
<td>4</td>
<td>Tungsten</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>Aluminum</td>
<td>100</td>
</tr>
</tbody>
</table>

Figure 4.16: EDS point analysis for the SPS sample that heat-treated at 600 °C for 10 minutes

Table 4.5: EDS spots chemical composition

<table>
<thead>
<tr>
<th>Spot</th>
<th>Element</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Aluminum</td>
<td>94.59</td>
</tr>
<tr>
<td></td>
<td>Tungsten</td>
<td>5.41</td>
</tr>
<tr>
<td>2</td>
<td>Aluminum</td>
<td>94.59</td>
</tr>
<tr>
<td></td>
<td>Tungsten</td>
<td>5.41</td>
</tr>
<tr>
<td>3</td>
<td>Aluminum</td>
<td>92.35</td>
</tr>
<tr>
<td></td>
<td>Tungsten</td>
<td>7.65</td>
</tr>
<tr>
<td>4</td>
<td>Aluminum</td>
<td>94.53</td>
</tr>
<tr>
<td></td>
<td>Tungsten</td>
<td>5.47</td>
</tr>
</tbody>
</table>

Figure 4.17: EDS chemical point analysis for the SPS sample that heat-treated at 700 °C for 30 minutes.
4.2.5 X-Ray diffraction (XRD)

XRD testing was applied to identify the main composite components and the intermetallic phase(s) that formed at the aluminum/tungsten interface. The test was applied for the as prepared and for heat treated SPS samples. Figure 4.18 showing the XRD peaks results for the as prepared, below melting point (600 °C, 20 h) and above the melting (800 °C 30 minutes) heat-treated samples. The X-axis represents the 2-theta value in degree units, while the peaks intensity with arbitrary units are shown in the Y-axis. It can be seen that aluminum has the highest peak intensity compared with other phases, 38.47, 44.72, 65.07, 78.259 were the two-theta values of the aluminum peaks. The tungsten peaks were less intense, where 40.256 and 58.22 the two-theta values of tungsten. Also, it can be noticed that the tungsten peaks disappeared for the melted sample which indicates that it got consumed due to reaction with aluminum to form an intermetallic compound. Al_{12}W intermetallic phase was the only tungsten aluminide that can be detected and appeared at 16.57 and 28.92 two-theta values. Al_{12}W peaks were missing for the as prepared sample which indicated that there was no reaction occurring between composite components.
Figure 4.18: XRD peak analysis for the SPS samples
4.3 LASER POWDER BED FUSION (LPBF):

4.3.1 Macroscopic observations:

The as received LPBF cubes samples’ cross section was homogenous and had no visual defects, where low porosities density were observed. The heat-treated samples over the matrix melting point (600 °C for 2 days) show formation some cavities and porousness through the sample. Re-solidification was observed for these samples and during furnace heat-treatment at a temperature around 600 °C. At the begging the samples melted and tend to flow towered the lower edge of the crucible and become a drop-like shape and then re-solidified where the samples surfaces become solid surfaces where the indentation tool cannot penetrate the surface.

4.3.2 Optical Microscopic observations

Information such as tungsten particle sizes, distribution in the matrix alloy and the microstructure of the samples, cross section obtained for the as received, below melting and above melting heat treatment. Limited information was obtained for the aluminum/tungsten interface. Figure 4.19 shows the optical microscopic image of the as received LPBF sample. It can be seen that the tungsten particles are well distributed in the matrix alloy, the average particle size was about 30 μm, and most of the particles had irregular shape with close to a circular shape.
The microstructure of 580 °C for 3 hours heat-treated sample is shown in the Figure 4.20. There are no big differences that can be shown at this level of the magnification, the tungsten particles were surrounded by dark-colored rings which indicate an interaction occurred between aluminum alloy and tungsten during heat-treatment.
Microstructure of a sample that is heat-treated at higher temperature and holding time (600 °C for 2 days) is shown in the Figure (4.21). It can be seen that the aluminum / tungsten interface appears in a light smooth gray color which indicates that an interaction occurred between aluminum and tungsten during heat treatment resulting in forming an intermetallic. More details are showing in the higher magnification image, where the interface layer dimension is not uniform around the tungsten particles. Generally the smaller the particle diameter the wider the intermetallic layer. Tungsten particles were not flash with matrix, where they appear as a dimple-like shape that explain why the image looks unclear. More growth for the interface is shown in Figure (4.22) where the sample had heat-traded at 700 °C for 12 hour. It can be noticed that the majority of the intermetallic layer had grown.
of the tungsten particles got consumed, some cracks were observed in the intermetallic region.

4.3.3 Scanning electron microscopic (SEM) observation.

SEM images giving more details than the optical microscope images due to the high magnification ability. Figure (4.23) showing the microstructure of the as received LPBF sample. It can be seen that the tungsten particles had been well distributed into an aluminum alloy matrix. Particles had different sizes and shapes, most of them had circular-like shapes. The aluminum / tungsten interface can tell that a weak interaction occurred where no clear intermetallic layer around tungsten particles was observed as can be seen in the high magnification image. The tungsten particles had a smooth surface for the most part of the particle's circumference as it can be seen, while the rest was rough. The rough part of the
particles was caused by the effect of the laser beam when it hit the particles during sample preparation. The matrix alloy showed some unfused powder particles.

The below matrix alloy melting point heat-treated (550 °C for 6 days) sample microstructure is showing in the Figure (4.24). It can be shown that the matrix/tungsten interface has grown within the 1 μm thickness range, where it appears in gray-colored region. The interface growth into the aluminum side of the interface with rough edges. That indicate that the intermetallic form at interface region. The interface growth into the aluminum side of the interface with rough edges. That indicate that the intermetallic form at interface region. Matrix alloy having stains that had similar color to the interface.

Figure 4.24: Microstructure of sample that heat-treated at 550 °C for 6 days, a) low magnification, b) high magnification

78
The above matrix alloy melting point heat-treated (650 °C for 12 hours) sample microstructure is shown in the Figure (4.25). It is clearly shown that the interface is widely grown with sharp, straight, and smooth edges. The grown intermetallic was has a hexagonal-like shape with a corner angle of about 120 degrees, with thickness in the order of 10 μm. The interface has two regions with two gray-tones color, indicating that there are two phases formed at the aluminum/tungsten interface region. The light gray color phase appears within and around the tungsten particles, while the majority of the interface was occupied by the darker phase. A third phase was precipitated at some places outside of the interface as shown.

![Microstructure of the LPBF sample that heat-treated at 650 °C for 12 hours, a) low magnification, b) high magnification.](image_url)

Figure 4.25: Microstructure of the LPBF sample that heat-treated at 650 °C for 12 hours, a) low magnification, b) high magnification

4.3.4 Energy Dispersive X-Ray Spectroscopy (EDS).

Figure (4.26) shows the EDS point analysis for the aluminum/tungsten interface area (less 1 μm) of the as received LPBF sample. It can be seen that the tungsten atomic % is about 7.2 and 83.1 for aluminum and the rest percent is for the silicon.
Table 4.6: EDS point analysis

<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic %</th>
</tr>
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<tbody>
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<td>Al</td>
<td>83.14</td>
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<tr>
<td>Si</td>
<td>9.62</td>
</tr>
<tr>
<td>W</td>
<td>7.24</td>
</tr>
</tbody>
</table>

Figure 4.26: EDS point analysis of the as received LPBF sample

Table 4.7: EDS point analysis

<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>82.87</td>
</tr>
<tr>
<td>Si</td>
<td>9.36</td>
</tr>
<tr>
<td>W</td>
<td>7.77</td>
</tr>
</tbody>
</table>

Figure 4.27: EDS point analysis of LPBF sample that heat-treated at 550 °C for 6 days

Aluminum / tungsten interface region EDS analysis of a sample that is heat-treated at temperature below aluminum alloy matrix (550 °C for 6 days) is shown in the Figure
Tungsten atomic % is about 7.7 and aluminum was 82.8 and silicon was about 9.36%.

Figure (4.28) shows the EDS line analysis of the over melting (650 °C for 12 hours) heat-treated LPBF sample. The line scan as shown starts at the aluminum matrix side before the interface and crosses over the interface and tungsten particle and ends at aluminum side beyond the interface of the opposite side of the scan starting point. The first part of line analysis shows a fluctuation between aluminum and silicon atomic percent over a few micrometers while no tungsten was detected. This fluctuation is due to the silicon alloying element appearing in the dark gray color prior to the interface. Fluctuation is over when the scan enters the interface region, where aluminum becomes stable at around 85 % while tungsten to about 10% and the rest is for silicon. Around the tungsten particle border, tungsten and silicon percent increased to about 35% and aluminum reduced to about 25%. Along the tungsten area silicon average percent was about 15, while aluminum was just about 5 %. Another change in atomic percent is shown in the next few micrometers beyond the tungsten area, where tungsten becomes in the range of 35%. While aluminum and silicon fluctuate repeatedly between 20 and 35 %. Then aluminum percent stable at 85 % and tungsten at 10 % while 5 % was for the silicon.
4.3.5 Electron backscatter diffraction (EBSD).

The EBSD analysis was carried out for the samples that show a wide growth of the interface area since the other samples failed to give an acceptable confidence index for the desired tested region (aluminum/tungsten interface region), the angular view of the EBSD camera image Figure (4.29) shows that tungsten particles and the small portion of the surrounding interface are not flat with matrix surface even after a long time period of
sample preparation (grinding and polishing) prior to the EBSD test, this explains why these field give an acceptable confidence index.

![Figure 4.29: Tilted SEM image of the LPBF that heated at 650 °C for 12 h.](image)

Figure (4.30) showing an EBSD point analysis map for the tungsten and interface area of the LPBF sample that was heat-treated at 650 °C for 12 hours. The kikuchi patterns for spot 1 are confirmed if the examined area was tungsten, while spot 2 shows it is Al₄W region, spot 3 was Al₁₂W.
Figure 4.30: EBSD point analysis of the LPBF that heated at 650 °C for 12 h.

Figure (4.31 a) showing the image quality and inverse pole Figure for the Al$_{12}$W intermetallic interface area of the sample, where the compound was shown as a single crystal and the crystal orientation was \{011\}, while Figure (4.31 b) showing the phases distribution over the examine area.
Figure 4.31: EBSD point analysis of the LPBF that heated at 650 °C for 12 h, a) showing the image quality and inverse pole figure, b) the phases distribution
4.3.6 X-Ray diffraction (XRD)

Figure (4.32), shows the XRD peaks results for the as-received LPBF, below melting point (550 °C 6 days) heat treated and beyond melting (650 °C 12 hours) heat-treated samples. It can be seen the Al₄W intermetallic phase was detected for the as-received sample and for the low-temperature heat-treating sample, it disappeared for the sample that was heat-treated beyond the melting point of the aluminum alloy. Unlike, no peaks for the Al₁₂W were obtained for the as-received sample while it clearly showed for the heat-treated samples. Due to the reaction of the matrix alloying elements in between and with tungsten particles, new compound peaks were detected. Two tungsten silicide compounds peaks were received, W₅Si₃ compound peaks were shown only for the as-received sample. While it disappeared for the heat-treated samples and replaced by WSi₂. Andalusite, aluminum-silicon-oxygen compound peaks were detected beside the peaks of the main composite components (tungsten, aluminum, and silicon) for all samples. While no peaks were detected for the Al₅W for all samples.
Figure 4.32: XRD peaks results for the LPBF samples.
5. DISCUSSION

In this chapter, the obtained results shown in the previous chapter will be discussed with the goal of identifying the reasons for the tungsten aluminides formation, growth behavior, and morphology. Computer-based modeling of the crystal structure and known thermodynamic properties of the crystal structures of the intermetallic compounds is applied to help gain a clear understanding of the phase transformation in the aluminum/tungsten system.

5.1 WELDING AND MELTING.

Despite the non-uniformity of the deposited aluminum alloy over the tungsten strip during welding, when the filler metal shows a high wetting angle over the base metal as shown in Figure 4.1, the tensile test shows that the welded joint was stronger than the base metal. Fracture occurred in an area away from the welding zone. That indicates that the reaction between aluminum and tungsten resulted in a strong joint. Optical microscopy of the joint, shown in Figure 4.2, indicates that there is an interaction occurs between aluminum and tungsten where tungsten aluminides formed at the interface. SEM image of the interface (Figure 4.3) shows that the interface has rough and curved edges and has different colored areas. EDS chemical analysis and the XRD peaks results (Figures 4.5 and 4.7) for the as-welded sample show formation of intermetallic compounds, including Al₅W, tungsten silicide, and other compounds were detected. Due to the fast deposition and solidification process, these compounds were unstable and transformed to more stable compounds upon heat-treatment.
Upon heat-treatment of the welded sample at 500 °C for 24 hours, the phases at the interface became well defined. The EDS chemical analysis and the XRD peaks, shown in Figures 4.6 and 4.7, show the formation of more stable phases. Specifically, two Al-W-Si phases were observed – Al_{12-x}Si_xW and Al_{4-y}Si_yW. A more stable tungsten aluminide intermetallic compound (Al_{12}W) form at the Al/Al_{4}W interface as shown in the EDS, and XRD Figures 4.6 and 4.7 [45].

5.2 SPARK PLASMA SINTERING (SPS).

In contrast to the welded samples, SPS as-received samples were prepared at relatively low temperature (550 °C) when no melting occurs. The short hold time of 2 minutes at the reaction temperature, there is not enough time for any observable reaction between the aluminum and tungsten to take place (Figures 4.9 and 4.12). The slow and gradual heating at the furnace for different heat-treatment conditions (550 to 800 °C) leads to form only Al_{12}W intermetallic compound at the interface area where no other tungsten aluminide was detected as shown in the XRD peak results in Figure 4.18. This is due to the fact that the Al_{12}W is the more stable intermetallic among other tungsten aluminides [45], Even if other intermetallics are formed then they directly consumed as a credit for Al_{12}W.

The interface for the below aluminum melting point heat-treated sample shown in Figure 56 has rough and curved edges which indicate that the growth is a diffusion-controlled process. The solid-sate growth of the Al_{12}W of the samples that heat-treated at 600 °C for different time periods where represented in the Figure 5.1, the volume fraction
of the Al\textsubscript{12}W intermetallic increased parallel to the holding time. The growth was not uniform and have curved edges as shown in the Figure 4.13 which is indicate that the growth at the solid state is a diffusion-controlled process.

![Graph showing the increase in volume fraction of Al\textsubscript{12}W with time during the heat treatment at 600 °C.]

Figure 5.1: Increase in volume fraction of Al\textsubscript{12}W with time during the heat treatment at 600 °C.

The interface-controlled was the growth mechanism for the samples that heat-treated at a temperature above the aluminum melting point. Samples that heat-treated at 800 °C show precipitation at solidified aluminum at grain boundaries as shown in Figure 4.15 where all tungsten particles have been consumed. The hexagonal shape of the Al\textsubscript{12}W precipitation and growth mechanism will be discussed later in this chapter. Where
aluminum reacts with Al₄W compound to form Al₁₂W which is considered to be stable at the reheating temperature [4]. It can be noticed also that the growth of the interface occurs out of the tungsten side border where the interface grows at the aluminum side of the interface. The low enthalpy of formation of the tungsten aluminides acts as a driving force that pulls the tungsten atoms to the interface area which is rich with aluminum atoms where the reaction occurs [2]. The morphology and growth of the interface will be discuss in other part of this chapter.

5.3 LASER POWDER BED FUSION (LPBF)

Similar to the welding samples, the as-received LPBF samples show a formation number of intermetallics including Al₄W and tungsten silicide compounds. These intermetallics are formed due to the super fast heating and solidification process of the combination of the metals during the LPBF sample preparation as a result a smaller amount of the compound was formed compared to the welding sample that explains why these compounds cannot be shown in the SEM images.

A partial part of the tungsten particle surface has some bumps and deformities as shown in Figure 4.23. The rest of the particle is relatively smooth. This is due to the fact that the rough part of the particle surface was facing the incident laser beam at the moment when the beam crossed over the particle during the printing process.

Similar to the SPS sample that is heat-treated below the aluminum matrix melting point, the LPBF sample (Figure 4.24) has a curved and rough interface, this is due to the diffusion-controlled growth mechanism of the interface. Excessive growth of the interface
was observed for the samples that heat-treated beyond the aluminum alloy matrix metal melting point (Figure 4.25). The XRD peaks results (Figure 4.32) detect the formation of both Al₄W and Al₁₂W intermetallics. On the other hand, the EDS chemical analysis (Figure 4.28) show three alloying elements across the interface which are: aluminum, tungsten, and silicon. Tungsten atomic percent maintained at 10% (which is the same percent for the Al₁₂W) along the interface area (gray region of the interface). While the tungsten atomic percent of the interface next to the residual tungsten particle is maintained at the range of the Al₄W intermetallic. Silicon atomic percent was maintained at 5% along the Al₁₂W area and about 40% of the Al₄W region while it was about 20% along with the residual tungsten particle. Silicon precipitates were detected at Al₁₂W/Al interface due to the insolubility of silicon in aluminum. It is believed that some aluminum atoms in the Al₁₂W and Al₄W have been replaced by silicon atoms (the next element of the aluminum in the periodic table with smaller atomic radii size) that make the compounds more stable and explained the excessive growth of the interface where the crystal structure unit cell with replaced aluminum atoms will have larger interstitial sites size as what will be discussed later. The effect of the alloying element of the aluminum matrix alloy of the welding and LPBF processes was not a focus area of the dissertation. However, none of the elements (table 3.2 and 3.3) or their compounds except the silicon was detected in the EDS or XRD results due to their limited amounts. It is worth to be mentioned is that the magnesium alloying element of the LPBF aluminum alloy has an important effect in enhancing the hardness of the matrix by age-hardening by forming the Mg₂Si compound [42].
5.4 TUNGSTEN ALUMINIDES PHASES FORMATION SEQUENCE

As has been seen for the welding and LPBF samples that the Al₄W intermetallic compound was the main phase formed with other side compounds (tungsten silicide and Andalusite) at the interface area between aluminum alloy matrix and tungsten with a clear absence of the Al₁₂W compound. While gradual furnace heating of the SPS samples and heat-treatment for the welding and LPBF samples promoting of formation the Al₁₂W compound as the main compound and some time the only phase at the interface this due to the Al₁₂W is the most stable compound among the all tungsten aluminides [45]. So the tungsten aluminides formation sequence depends on the reaction conditions of the aluminum and tungsten. Deposition of a molten high temperature aluminum alloy over tungsten followed by fast cooling is the condition of formation of the Al₄W, while slow heating of the composite produce formation the Al₁₂W. This formation sequence agrees with Wang [4] and Chen [17]. No Al₅W or other tungsten aluminides compounds was detected during examination testing.

5.5 INTERFACE MORPHOLOGY

Since the major part of the interface of the whole examined samples of the preparation processes is Al₁₂W intermetallic where other compounds did not show a proper growth, then the morphology of the Al₁₂W will be discussed.

The below melting point heat treated samples the interface has curved and rough edges as shown in Figures 4.13, 4.24, which indicate the growth mechanism is a diffusion-
controlled process. While beyond aluminum alloy melting point heat-treating samples, the interface has sharp, straight, and smooth edges as shown in the Figures (4.17 and 4.25). The interface has hexagonal morphology with internal angle of 120°. According to Jackson alfa theory [30], the interface of a compound should have straight and sharp edges if and only if the α value as shown in the equation (5.1) is equal or larger than 2.

\[
\alpha = \frac{\Delta S}{R} \left(\frac{\eta}{\nu}\right) \quad (5.1)
\]

Where \(\Delta S\) is the entropy of fusion at melting temperature (J/mol.K), \(R\) is the gas constant (8.314 J/mol.K). While the \(\eta\) is the number of nearest neighbor atoms and \(\nu\) is the coordination number. ThermoCalc software using the TCHEA4 database [46] show that the entropy of formation is about 58.179 (J/mol.K), and the EBSD IP results show that the (110) is the prefer orientation plane of the \(\text{Al}_{12}\text{W}\) crystal as shown in the Figure 4.31 and the interphase is growth as a single crystal where no grain bounders were found. The number of the nearest neighbor atoms is 6 and ordination number is 12 this give the following expression of the equation 1 [31][32] [28] [29].

\[
\alpha_{(110)} = 58.17 \times \frac{6}{12} \cong 3.5 > 2 \quad (5.2)
\]

As a result the shape of the \(\text{Al}_{12}\text{W}\) interface is sharp and smooth and the growth is an interface-controlled process.
As discussed above, the EBSD IP of the Al$_{12}$W results show that the plan family \{110\} is the preferred orientation of the equilibrium crystal. With help of the CrystalMaker computer modeling program [47], Figure 5.1 show the (110), (100), and (111) planes of the Al$_{12}$W crystal structure ball and stick model. Aluminum atoms and bonds have been removed to get a clear view for the rest of the structure.

![Figure 5.1: Al$_{12}$W crystal structure: (a) (100), (b) (110), and (c) (111) planes](image1)

According to the Barvais-Frediel law [33] the importance (the frequency of observation) of a crystal plane is dependent on the planar density and the interplanar spacing, where the higher density and higher interplanar spacing plane should have the higher frequent appearance among other planes. For the equilibrium Al$_{12}$W crystal
structure the {110} plane family have the highest planar density (0.0244 atom/Å²) comparing to {100} and {111} plane families (0.0173 and 0.0199 atom/Å²) respectively, while the {110} have the second bigger interplanar spacing after the {100} of the structure. Therefore the {110} planes have the minimum total surface energy among the other planes, which persuade that the [110] direction has the lowest growth direction compared to [111] and [100]. As a result the {111} and {100} planes will disappear and the {110} planes will expand for the equilibrium crystals. Figure 5.2 shows how the hexagonal Al₁₂W precipitate can form by

![Hexagonal Al₁₂W precipitate](image)

Figure 5.3: the Al₁₂W hexagonal shape, (a) CrystalMaker model, (b) SPS sample 700 °C for 30 minutes Al₁₂W precipitate
Intersecting six \{110\} planes, where the computer modeling looks identical to the real \(\text{Al}_1\text{W}\) precipitates, that explain why the equilibrium precipitates has a hexagonal shape.

5.6 ATOM TRANSFORMATION.

As shown in the result chapter for all the applied processes (welding, SPS, and LPBF) \(\text{Al}_{12}\text{W}\) is the main intermetallic compound grown at aluminum/tungsten interface for the heat-treated samples. A question of what is the mechanism by which metals atoms

![Crystal structure of \(\text{Al}_{12}\text{W}\) including interstitial sites.](image)

Figure 5.4: The crystal structure of the \(\text{Al}_{12}\text{W}\) including interstitial sites.
transfer across the interface region during heat-treatment is brought for discussion. Grain
boundaries, pipe dislocations, interstitial sites, and atom substations represent the path by
which atoms transfer for a regular metal alloy [43].

Since the EBSD results (Figure 4.31) show that the Al$_{12}$W interphase area is grown
as a single crystal where no grain boundaries were observed, then the grain boundaries
diffusion path is not the path by which atoms transfer through the Al$_{12}$W intermetallic. The
Al$_{12}$W is a line compound as shown in the aluminum/tungsten phase diagram (Figure 1.1)
where the composition is fixed and a strong covalent bonding is the bond between
aluminum and tungsten atoms [2]. This cancels the option of the substation diffusion
mechanism. The crystal structure unit cell of the Al$_{12}$W is shown in the Figure 5.3 where
tungsten atoms (R=1.62 Å) occupied the corners and center of the unit cell, while
aluminum atoms (R=1.21 Å) are surrounding the tungsten atoms [48]. The open structure
of the Al$_{12}$W has six face interstitial sites (R=0.93 Å) where the two face-middle sites are
sitting next to each other as shown in Figure 78. It is believed that these sites can coalesce
together during heat-treatment and create a bigger site which is able to accommodate
aluminum or tungsten atoms; this could be the path by which metal atoms transform
through the compound. Replacing some aluminum atoms with silicon atoms (R=1.11 Å)
of the Al$_{12}$W structure as discussed above enlarges the intestinal sites and besides the fact
the effect of silicon in increasing the molten aluminum fluidity [44], that helps to speed up
and facilitate the metals atoms transformation process. That explains why there is excessive
growth of the interface of the LPBF heat-treated samples compared to the SPS samples (Figures 4.13 and 4.25). The pipe dislocations diffusion mechanism could be another fast path by which atoms can be transferred through the interface.
6. CONCLUSIONS AND RECOMMENDATIONS

In conclusion, tungsten aluminides are intermetallic compounds with complex crystallographic structures. They are difficult to prepare by common processes and different researchers have reported different conditions for the formation of the three tungsten aluminides – Al$_{12}$W, Al$_5$W, and Al$_4$W that are stable at room temperature. In spark plasma sintered (SPS) compacts of elemental Al and W, tungsten aluminides form and grow at the interface between Al and W regions, but not all three of the compounds are observed after consolidation and heat treatment. Al$_4$W forms at high temperature when molten aluminum interacts with metallic tungsten and is retained upon fast cooling to room temperature. While slow heating leads to the formation of the more stable Al$_{12}$W at the interface. The growth of Al$_{12}$W is diffusion-controlled when the heat-treatment is carried out below the melting point of aluminum while heat-treating above the melting point of aluminum leads to the formation of a smooth faceted interface between Al$_{12}$W and aluminum, indicating an interface-controlled growth process. Due to the high planer density of atoms on the {110} plane family and large interplanar distancing compared with other planes of the Al$_{12}$W crystal structure, an Al$_{12}$W equilibrium precipitates has a hexagonal shape bounded by {110} planes.

No composition variation is observed within the Al$_{12}$W phase, even when the transformation is only partially completed. This, along with the observed facetted interface means the growth of Al$_{12}$W is interface-controlled, with diffusion occurring fast enough to even out any composition variation. Al$_{12}$W has an open crystal structure with an atomic
packing factor of only about 50%, Interstitial vacancy sites which occur in pairs at the edges and face-centers of the crystal structure are represent a fast path for the movement of Al and W atoms through the crystal.

The laser powder bed fusion (LPBF) samples were made using, not elemental aluminum, but an Al-Si alloy with a nominal composition of Al-12 wt %Si. Silicon, which has been added to improve the fluidity of aluminum casting alloys has a great impact on the intermetallic compounds that are formed. The intermetallic phases that form when silicon is present exhibit features which are similar to that observed in the elemental Al-W alloys, i.e. faceted interface between the intermetallic compound and the aluminum matrix, and a constant composition. The composition of the intermetallic phase seems to indicate some aluminum atoms are being replaced by silicon in the crystal structure of Al₄W and Al₁₂W. In this study, it was not possible to determine the crystal structures of the two Al-W-Si intermetallic compounds that were formed.

As a future work of this field of study, it recommended that heat treatment be conducted in vacuum or in an inert atmosphere and over a larger range of temperatures and times to obtain information the transformation kinetics. Transmission electron microscopy is suggested to identify whether there are other fact diffusion paths, such as pip-diffusion along dislocations, and the details of the mechanisms involved in the interface-controlled growth, such as the ledge or screw dislocation mechanisms.
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