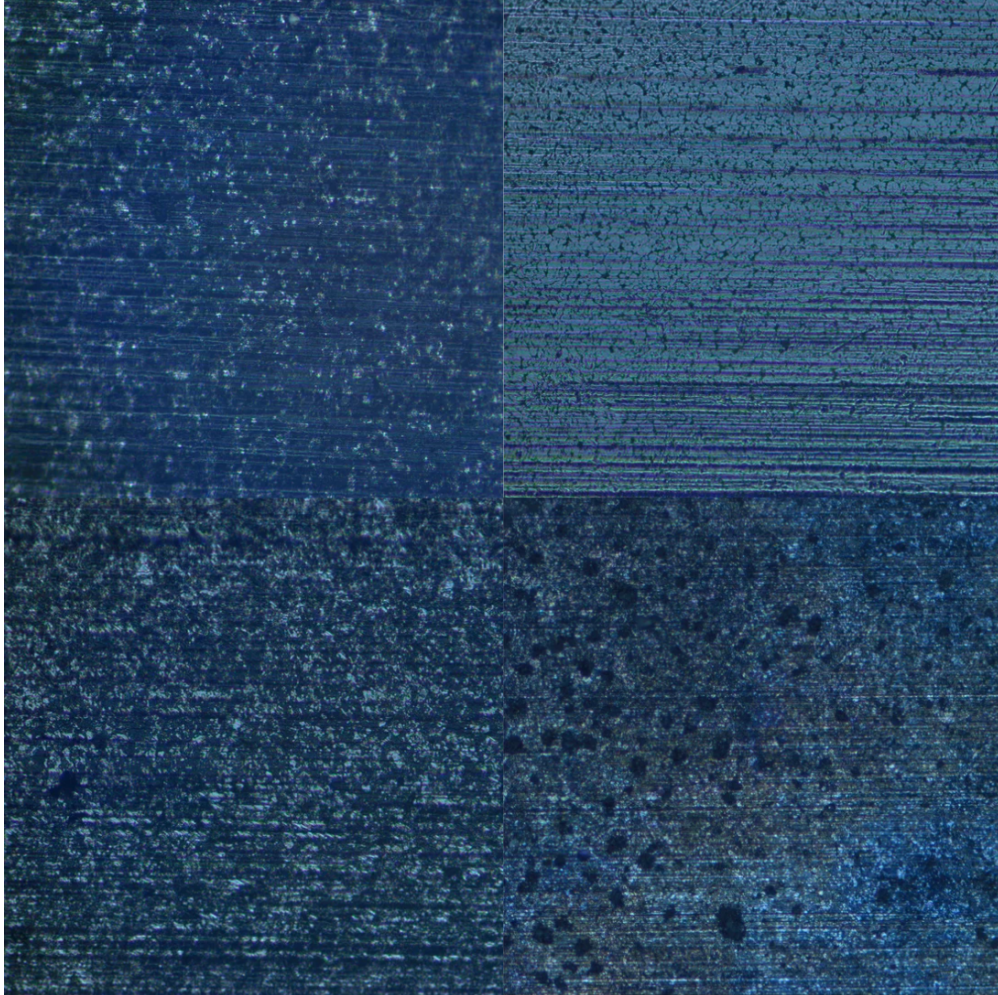




CHALMERS
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Integrating Highly Conductive 2D Materials in Metal Matrix

Master's Thesis in Physics

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CHALMERS UNIVERSITY OF TECHNOLOGY
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Abstract

MXenes have attracted significant attention in recent due to their high electrical conductivity and potential for usage in composite material. This thesis investigated $\text{Ti}_3\text{C}_2\text{T}_x$ MXene/Al composites with varying aluminum content to see whether the conductivity of the composites can overcome the one for pure aluminum. The MXene was synthesized through an in-situ HF etching process, mixed with aluminium powder, pressed into coin-like pellets, and sintered in argon atmosphere at 550°C . Contact conductivity and Rockwell hardness measurements were performed, as well as microstructure analysis through SEM and EDX.

The results showed that high-Al composites showed measurable conductivity and improved mechanical integrity, while lower-Al composites exhibited fragile behaviour and measurement challenges. Sintering influenced both conductivity and hardness, indicating strong dependence on densification and microstructure. This was supported by the microstructure analysis. The study demonstrates the importance of composition and thermal processing in determining the electrical and mechanical properties of MXene/Al composites. None of the composites overcame the electrical performance for pure aluminium, however there are many aspects of this project that could be investigated further before declaring MXene/Al composites not useful for electrical transport applications. These aspects include examining the residual aluminium in the pure MXene samples and comparing the results of other sintering methods better suited for powder composites, such as spark plasma sintering (SPS).

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List of Acronyms

Below is the list of acronyms that have been used throughout this thesis listed in alphabetical order:

EDX - Energy-Dispersive X-ray Spectroscopy

MMC - Metal Matrix Composite

SEM - Scanning Electron Microscopy

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1

Introduction

1.1 Background

The global demand for electricity continues to increase as society becomes more dependent on electrical systems, renewable energy technologies and electrified transportation. Today, one of the biggest challenges we face is how to use this energy more effectively. A large part of that problem is not just how we generate it but also how we transport it. The electrical resistance of a material determines how efficiently it can transport the energy and when energy passes through a conductor with finite resistance, part of the energy is converted into heat through Joule heating[1]. This not only reduces the efficiency of power transmission but also results in greater energy losses when the transmission distance increases. Consequently, improving the electrical conductivity of conductive materials has become an important topic in the development of more energy efficient electrical systems.

Specifically for energy transport, the most widely used material in transmission lines is aluminium. Aluminium is a conductive metal but not as conductive as copper and silver. However, aluminium has a lot of benefits. This thanks to it being lightweight and a cheap metal[2]. Still, this raises a question, if there was a way to make aluminium more conductive, how could that work? Could a composite of aluminium and another highly conductive material together become more conductive than pure aluminium?

One highly interesting material that matches that description is a relatively new material called MXene. MXene is a material that, just like graphene, exhibits a 2D sheet structure and is built up by a sheet of a transition metal, and a layer of either nitrogen or carbon[3]. These layers are then repeated and stacked. One thing that makes the MXenes so remarkable is their customizability. The transition metal can be chosen from more than 10 elements and the surface of the MXenes can be modified by addition of many functional groups. This makes the MXenes very versatile and can be constructed such that they fit the wanted applications. For example, many of the MXenes have shown highly electrical conductive properties and some have even shown properties of superconductivity at critically low temperatures [4].

In recent years, MXenes have been investigated as reinforcement phases in a variety of metal matrix composites [5][6][7]. Their combination of high intrinsic electrical conductivity, two dimensional morphology and large surface area makes them at-

tractive candidates for improving both the electrical and mechanical properties of metallic materials. Aluminium is especially interesting as a matrix material since it has low density, good electrical conductivity and is already widely used in power transmission. However, incorporating MXenes into aluminium is challenging. Good homogeneous dispersion of the MXene sheets, little to no agglomerations and good interfacial contact during processing is crucial for high electrical performance and these are large challenges for the project. Furthermore, formation of oxidation layers on aluminium particles and thermal stability of the MXene may influence the final properties of the composite.

Even though many studies have demonstrated promising electrical properties of individual MXenes [4][8], fewer studies have investigated their incorporation into bulk aluminium composites aiming for conductive. More importantly, the influence of MXene content on electrical performance after powder pressing and sintering remains insufficiently understood. In addition, the relationship between the composite microstructure, elemental distribution and electrical performance have not been fully established.

1.2 Purpose

The findings from this project is aiming to understand more about the MXenes role in aluminium composites with specific focus on the electrical conductivity and structure of the composites. Could the contribution of MXene in the composite generate a combined conductivity that may surpass the one for pure aluminium and maybe get close to the one for the slightly more conductive metal, copper?

By measuring the electrical conductivity of composites with different Al-to-MXene ratios the aim of this research is to investigate the contribution of material synthesis, composition, surface terminations and processing.

The objectives for the first part of this project is to synthesize the MXene, fabricate and sinter composites. For the second part of the project, the objectives are to measure the conductivity as well as the hardness and then to perform SEM and EDX characterization.

1.3 Limitations

In every stage in this process, there will be multiple roads to advance. For this project to be concise and not too widespread, there is a need for limitations.

One limitation made is that the project is focused on and limited to $\text{Ti}_3\text{C}_2\text{T}_x/\text{Al}$ composites. The composites will range from 0 % to 100 % aluminium content and no other metal or MXene will take part in this study.

Another limitation made is that the heat treatment process will be the same for all

composite samples. They all will be sintered in argon atmosphere, at 550 °C for 2 h. Optimizing the sintering parameters was outside the scope of this work.

Another limitation is connected to the main measurements of this project, and since the sample size is small, only contact resistance can be measured, not resistivity or bulk resistance. Measurements of real resistivity would be very difficult to perform.

The last limitation made was what measurements and characteristic methods were used. For this project contact resistance and Rockwell hardness tests were made, as well as SEM and EDX.

2

Theory

2.1 Electrical conductivity in materials

2.1.1 Conductivity and resistivity

Conductivity is the measurement that describes how well a conductor can carry electric current under an applied electrical field [1]. The conductivity σ of a material or system depends on two main factors. The number of charges n in a unit volume that are free to move in the material, and how efficiently they move. Their relationship can be described by Equation 2.1

$$\sigma = ne\mu \quad (2.1)$$

where e is the charge of the particle, n is the number of charged particles, and μ is the mobility of the free particles. This equation shows that a material can become highly conductive from either a large amount of free electrons, high mobility or a combination of both.

The reciprocal of conductivity is called resistivity and is the measure of the opposition to the flow of electricity. It is described by Equation 2.2

$$\rho = \frac{1}{\sigma} \quad (2.2)$$

where ρ is the electrical resistivity and σ is the electrical conductivity. This can then be directly translated to resistance like in Equation 2.3

$$R = \rho \frac{L}{A} \quad (2.3)$$

where L is the conductor length and A its cross-sectional area. Thus, highly conductive materials exhibit low resistivity and even lower electrical resistance.

When a potential difference is applied across a conductor, the current is related to the electrical resistance through Ohm's law,

$$V = IR \quad (2.4)$$

where V is the applied voltage, I the electrical current and R is the electrical resistance. In practical systems the measured resistance often consists of a few contributions. The total measured resistance may therefore be expressed as in Equation 2.5

$$R_{total} = R_{bulk} + R_{contact} + R_{lead} \quad (2.5)$$

where R_{bulk} is the intrinsic resistance of the samples, $R_{contact}$ originates from the electrical interfaces and R_{lead} is the resistance of the measurement leads.

2.1.2 Charge transport in metals

In metallic materials, the electrons are delocalized. This means that they are not bound to any atoms, and can move around freely[9]. The high concentration of mobile electrons gives the metals highly conductive properties. In contrast, insulating materials have their electrons heavily bound to their atoms, and are therefore less effective to transport charge. Metallic conductivity arises because electronic energy bands overlap at the Fermi level, allowing electrons to move under an applied electric field.

2.1.3 Factors affecting conductivity

The mobility of the charge carriers is highly dependent on the microstructure and composition of the material. Defects, grain boundaries, impurities and interfaces can act as scattering centres that decrease the mobility of the electrons and therefore reduce their conductivity[10]. Scattering can also occur due to lattice vibrations, phonons, and this directly links conductivity to temperature, where lower temperatures decrease the lattice vibrations and therefore reduce scattering.

The surface terminations are particularly important for materials such as MXenes. Electronegative termination such as fluorine can shift the electron density from the conductive metal to the termination and that will in turn decrease the mobility of the electrons. These surface terminations can, in some situations, pull in the electrons so much that the electrons scatter completely from the metal.

As a result, the electrical conductivity of a material is controlled both by the number of charge carriers and to how effectively they can move through the structure without scattering.

2.1.4 Contact resistance and four-point measurements

When two conductive surfaces are coming into contact, the visible contact area is much larger than the real contact area. Due to the microscopic surface roughness of the sample, electrical contact only occurs at small areas where the sample is more pointy and therefore only where the surfaces physically touch. The limited number of contact points introduce additional electrical resistance called contact resistance. When the contact load between the samples increase, so does the number of contact points, due to deformation of the surface, and therefore the contact resistance decrease. Consequently, more conductive pathways are established and the contact

resistance decreases until a stable value is reached.

Since contact resistance and lead resistance can become significant compared to the bulk resistance for highly conductive materials, a four-point measurement configuration is commonly used. In this arrangement, one pair of electrical contacts supplies the measurement current, while a second pair measures the voltage drop across the sample. Because the voltage leads carry negligible current, the influence of lead resistance and much of the contact resistance is minimized, resulting in a more accurate measurement of the sample resistance.

2.2 Conductive metals

Silver exhibits the highest electrical conductivity of all metals and also possesses exceptionally high thermal conductivity. At room temperature, its conductivity can reach 7×10^7 S/m [1]. and because of this reason it is sometimes used in special types of wiring but more often in electrical and electronic contact points [11]. Even though it is the highest conductive metal, silver has a few problems. The main problem is that it is expensive. This makes it difficult for big scale usage.

The second most conductive metal is copper with a room-temperature conductivity of 5.9×10^7 S/m. Copper is cheaper than silver and is therefore used more in wiring, often for metal layers in integrated electrical circuits [12]. Both copper and silver have relatively high density, copper is slightly less dense than silver. This becomes a practical problem since transporting highly dense materials comes with increased transportation costs but also with structural requirements in applications such as long-distance power lines.

Aluminium is another material with high conductivity. In room temperature it has the electrical conductivity of 3.7×10^7 S/m which is considerably lower than both copper and silver but still quite high in comparison to other metals [2]. Aluminium is significantly lighter and generally less expensive than the two and is therefore the natural choice in power lines and other wiring and cable applications.

Although silver and copper exhibits higher electrical conductivity than aluminium, aluminium offers an attractive combination of conductivity, low density, low cost and processability. For these reasons, aluminium is frequently used in metal matrices in electrically metal matrix composites[13][14]. By incorporating conductive reinforcements such as MXenes, it is possible to tailor both electrical properties and mechanical properties of the composite. The resulting conductivity depends not only on the innate conductivity of the components but also on how well the reinforcement is distributed and the qualities of the interfaces between the phases.

2.3 MXenes

MXenes are a family of two-dimensional transition metal carbides, nitrides and carbonitrides first reported in 2011[15]. This material is generated from an etching process of their precursor MAX phase, selectively removing the A-layer, and creates a 2D sheet like structure with atomically thin sheets terminated by functional groups such as $-O$, $-OH$ and $-F$. Since their discovery, MXenes have attracted considerable attention because of their high electrical conductivity, hydrophilicity, mechanical strength and tunable surface chemistry.

MAX phases are layered carbides or nitrides with the general form $M_{n+1}AX_n$ where M is the transition metal, A the main group element and X the nitrogen and/or carbon[3]. The atomically thin sheets of M, A and X are in a layered crystal structure held together by strong metallic covalent bonds. In an etching process the main group element is removed, this due to weak M-A bonds and strong M-X bonds [16], and this results in the MXene. These sequences of events can be viewed in Figure 2.1. MXenes also forms layered structures, held together by weak van der Waals bonds, and is therefore relatively easy to separate from stacks into few-layer or mono-layer sheets.

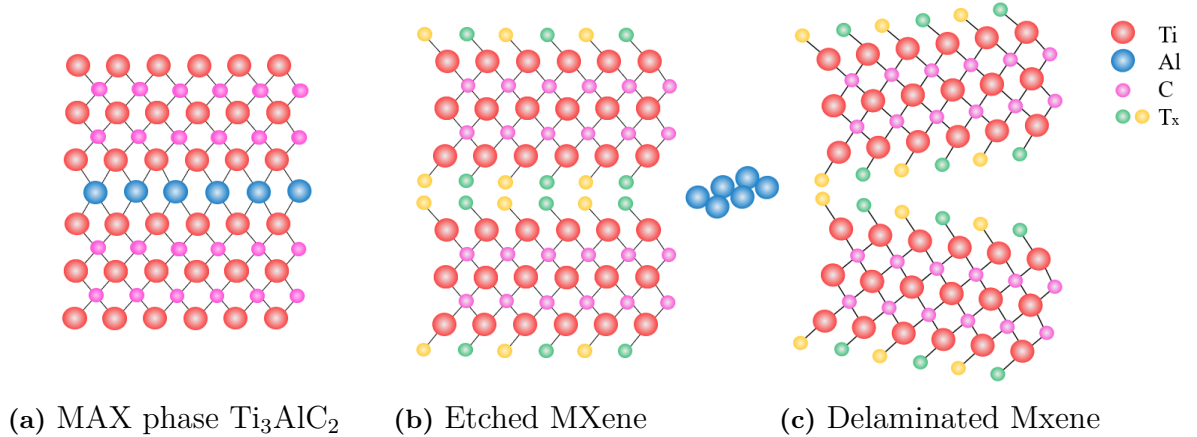


Figure 2.1: Schematic illustration of the formation of $Ti_3C_2T_x$ MXene. a) shows the MAX phase where the Al are still attached. b) shows the molecules after etching, removing the Al and gaining surface terminations T_x and c) shows the MXene sheets after delamination through mild ultrasonication.

2.3.1 $Ti_3C_2T_x$

In recent years, many MXenes have been fabricated and now we have more than 100 different variations. The first ever constructed MXene was the titanium-based carbide $Ti_3C_2T_x$, where the T_x denotes the surface termination groups attached to the MXene surface, generated from the MAX phase Ti_3AlC_2 and among the MXene family, $Ti_3C_2T_x$ is the most studied one. From its relatively simple synthesizing process it has allowed this MXene to be greatly investigated and has shown high

significance for applications for its high electrical conductivity, such as in energy storage[17], EMI shielding[18], sensors[19] and conductive composites.

The high electrical conductivity of $\text{Ti}_3\text{C}_2\text{T}_x$ originates from its titanium-carbon framework. Strong overlap between the Ti 3d orbitals and C 2p orbitals forms metallic electronic bands that cross the Fermi level, allowing electrons to move freely through the material. Therefore, charge primarily occurs within the Ti–C layers, while surface terminations modify the electronic structure and influence the overall conductivity. An individual $\text{Ti}_3\text{C}_2\text{T}_x$ sheet is intrinsically conductive due to its metallic framework, however in stacked MXene structures the conductivity can increase further since this allows the charge transport between neighbouring sheets.

2.3.2 MXene synthesis principles

2.3.2.1 Selective etching

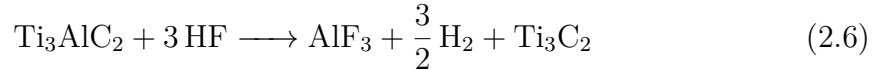
During the selective etching, aluminium atoms are dissolved while the Ti–C framework remains intact. This is possible because the metallic Ti–Al bonds are considerably weaker than the Ti–C bonds. As a result, the A-layer can be removed without disturbing the carbide layers, producing stacked MXene sheets Ti_3C_2 with surface terminations where the A-layer used to be.

2.3.2.2 Etching methods

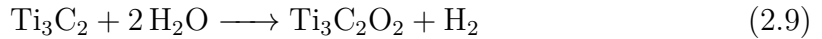
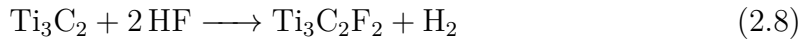
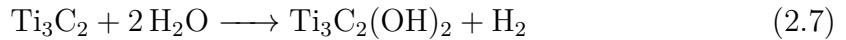
As MXene research increases, so does the ways in which to create the material. Through different methods of synthesizing the MXene, or through different etchants, we can achieve various properties based on the expected applications. To especially synthesize $\text{Ti}_3\text{C}_2\text{T}_x$ there are two types of etching methods.

Direct HF

The research team to synthesize the first MXene used the method of direct HF etching [15]. This method relies on the reaction



followed by either reactions 2.7, 2.8 or 2.9.

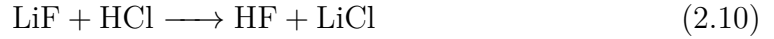


This method is widely used for its ease and simplicity of use [20]. However HF is highly corrosive and toxic. This method also commonly generates high contents

of F-termination groups. Since the F–Ti bond is strong this causes the electron density to shift from the titanium layers, and thus decrease the conductivity of the material. That specific disadvantage makes the direct HF etching method unfit for any enhancements in conductivity.

In-situ HF

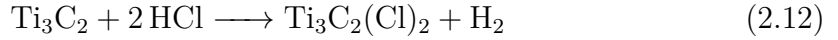
Another similar and more common etching method is called in-situ HF etching [21]. This method relies on lithium-fluoride LiF and hydrochloric acid HCl to create hydrofluoric acid HF directly in the bottle. The HF is generated continuously inside the reaction mixture, reducing the need to handle concentrated HF directly. The reactions therefore becomes



then when adding the MAX phase



followed by a combination of 2.7, 2.8, 2.9 and the additional 2.12 showing the different surface terminations for the MXene.



In situ HF etching, compared to direct HF etching, have shown to fabricate larger flakes with fewer defects [20]. This is mostly connected with in situ HF etching being a milder etching method. As a direct consequence from larger flakes with few defects, better electrical conductivity is generally observed from this method.

Therefore, this method is the more interesting one for a project whose main priority is to increase the conductivity of the material.

2.3.2.3 Surface terminations

As mentioned above, there are a few surface terminations that can attach to the MXene sheets. The ones prominent are the ones displayed in equations 2.7, 2.8 and 2.9, thus –OH, –F and –O respectively. The relative amounts of the different surface terminations depends on the synthesis conditions and therefore vary between MXene samples. The –O is the most chemically stable termination and will generally increase the conductivity of the MXene the most, as the Ti–O bonding preserves a more favourable electronic structure for charge transport. The –OH termination makes the MXene hydrophilic and useful for dispersion. The last main surface termination –F will decrease the conductivity of the MXene and this is due to the very electronegative nature of the fluorine. The Ti–F will form strong bonds, reducing the electron density of the metallic conduction between the Ti–C sheets. This will lead to increased electron scattering and therefore reduced electrical conductivity. Hence, if the goal is to increase conductivity for the MXene, the number of fluorine terminations should be minimized.

2.3.2.4 Washing and purification

After an etching process it is important to wash away the acid residuals and the dissolved reaction products. This is made through many centrifuging cycles until the pH of the mixture is 5 or more. At this stage the solution is stable and prevents further etching since the salts, residual acids and AlF_3 is removed from the mixture.

2.3.2.5 Delamination

The MXene in the solution is now in the form of stacked monolayers of MXene sheets. These are tightly packed and held together by weak van der Waals and hydrogen bonding interactions. Therefore, to increase the surface area and enabling the electrical and mechanical properties of MXene to be utilized, exfoliation through mild ultrasonication is used. This breaks the weak interlayer interactions and separate the layers into single- or few-layered sheets. With these individual MXene sheets the accessible surface area is increased and will improve the dispersion in liquids. This is a great benefit to composite fabrication since it allows the intrinsic electrical properties of the MXene sheets to be more effectively utilized.

These characteristics make $\text{Ti}_3\text{C}_2\text{T}_x$ an attractive reinforcement material for metal matrix composites, where its high intrinsic conductivity, large aspect ratio and surface chemistry may influence both the mechanical and electrical properties of the composite.

2.4 Metal Matrix Composites

Metal matrix composites is materials consisting of a continuous metallic matrix reinforced by a secondary phase such as ceramic particles, fibres or two-dimensional materials [22]. The objective is to create a material combining the desired properties of both materials separately into one super-material. Compared with conventional metals, MMCs can exhibit improved mechanical strength, wear resistance, thermal stability or electrical performance, depending on what reinforcement were used. An example of how a metal matrix composite can be constructed can be seen in Figure 2.2. Here the metal matrix material represent the lighter part and the reinforcement represents the darker parts. For this project, aluminium will act as the continuous metallic matrix and $\text{Ti}_3\text{C}_2\text{T}_x$ will act as the reinforcement.

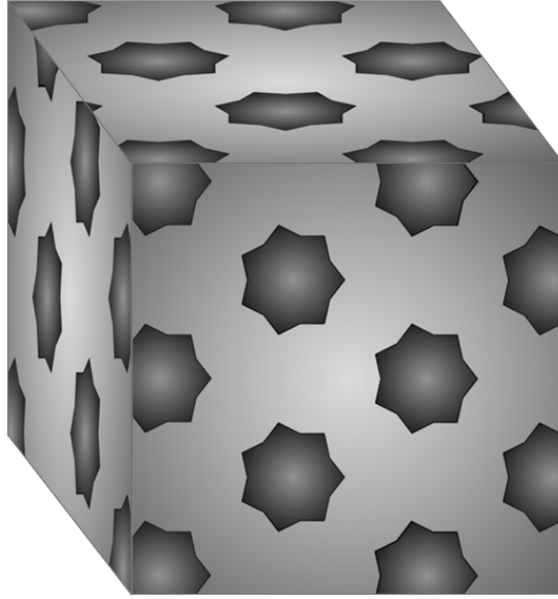


Figure 2.2: Schematic of a metal matrix composite where the light part represent the metal matrix and the darker part represent the reinforcement.

The electrical performance of conductive MMCs depends not only on the conductivity of the individual components but also on the continuity of the conductive network. A homogeneous distribution of the reinforcement phase, low porosity, good particle bonding and strong interfacial contacts are therefore essential for achieving high electrical conductivity.

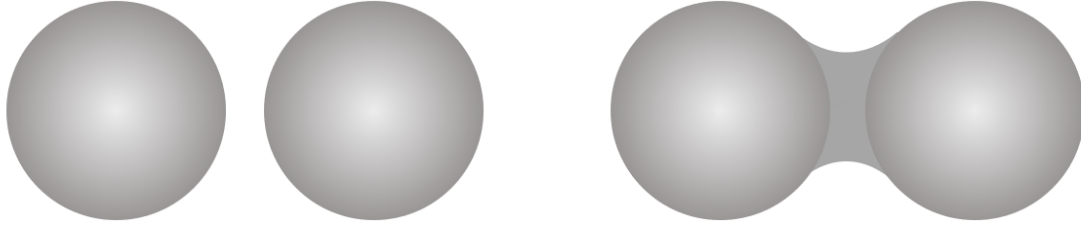
2.4.1 Freeze drying

One way to preserve a structure or dispersion created in a metal matrix composite mixture is to freeze it quickly before the materials separate. From the freezing a freeze dryer can be used so that, through sublimation, the water goes directly from ice crystals to water vapour. This method of freeze drying was selected since the sublimation process minimizes the capillary forces that could otherwise restack the MXene sheets or agglomerate the aluminium particles. Conventional evaporative drying would enable movement in the mixture and therefore would not preserve dispersion. Furthermore, freezing limits the reactivity between aluminium and water which could cause oxidation.

2.4.2 Sintering

The final step in the sample preparation is to apply a heat treatment to the coin-shaped samples. One of the ways to do so is by sintering. Sintering is a type of heat treatment that heats the sample to a temperature under the melting point and through atomic diffusion, makes material spread to neighbouring particles. In this way, the particles do not melt fully together but instead forms something called sintering necks. These sintering necks form between neighbouring particles creating a conductive pathway for electrons to travel throughout the composite. This can be

seen in Figure 2.3. These necks grow with higher sintering temperature and time and does not only make the composite more conductive but also strengthens the structure altogether. The sintering necks also minimize the porosity in the composite by taking up space that would otherwise contain voids. This also contributes to the improved mechanical strength.



(a) Two particles before sintering.

(b) Two particles after sintering.

Figure 2.3: Schematic of before and after sintering. In the process two neighbouring particles form a sintering neck connecting them and decreasing resistance for electrons travelling between them.

So, what temperature is the most optimal in this case? For aluminium the melting point is around 660°C . However excessively high temperatures may degrade or alter the surface chemistry of $\text{Ti}_3\text{C}_2\text{T}_x$. Therefore, the sintering temperature must be high enough to promote aluminium to diffuse while remain low enough to limit degradation of the MXene.

2.5 Characterization

2.5.1 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy is a technique that uses a focused electron beam to scan a surface of a sample[23]. By detecting the emitted secondary electrons, a high resolution image of the composition and surface topology can be constructed. SEM can therefore be used to evaluate particle morphology, porosity, cracking, neck formation after sintering and the over all microstructure of the composite.

2.5.2 Energy-Dispersive X-ray Spectroscopy (EDX)

Another technique used for microstructure characterization is the Energy-Dispersive X-ray Spectroscopy[24]. This technique is usually an addition to a SEM and detects the characteristic X-rays from the incident electron beam hitting the sample. The X-rays have different energies based on which element they have interacted with in the sample, and by detecting the X-rays we can obtain elemental mappings for the sample. With this kind of technique we can conclude elemental compositions and distributions for chosen areas in the sample, especially interesting for observing composite homogeneity and aluminium distribution. In this work, EDX was primarily used to investigate the distribution of the element Ti, Al, O and F within

the composites and thereby evaluate the homogeneity of the reinforcement and differences between sintered and non-sintered composites.

This technique, together with SEM, will enable us to investigate where the difference between composites with poor and strong electrical performance differ based on the microstructure of the samples.

2.5.3 Rockwell hardness

In addition to electrical characterization, the mechanical properties of the fabricated composites were evaluated using the Rockwell hardness test. Hardness is a measure of a materials resistance to permanent plastic deformation when being affected by an applied load. While hardness does not directly measure strength or toughness for example, it is often used as a simple indicator of a materials mechanical performance.

The Rockwell hardness is determined from the permanent penetration depth after applying a specified major load into the surface of the sample [25]. Materials with a shallower indentation are assigned a higher hardness value and deeper indentations suggest softer materials with lower hardness values. Different Rockwell scales exist depending on the indenter geometry and applied loads, allowing the method to be used for materials ranging from soft metals to hardened steels.

For this project, the Rockwell hardness measurements were used as a complementary characterization technique. Since the electrical performance of a powder based metal matrix composite is strongly influenced by particle bonding, porosities and densification, hardness measurements can provide additional information about the structural integrity of the samples. A higher hardness value can indicate improved densification and stronger bonding between neighbouring aluminium particles, which may also contribute to lower electrical contact resistance.

According to Holm's contact theory, the constriction component of the contact resistance increases with material hardness and decreases with applied load, this due to the contact area being smaller for harder materials[26]. The relation between hardness, applied load and contact resistance is described in Equation 2.13

$$R_c = \frac{\rho}{2} \sqrt{\frac{\pi H}{F}} \quad (2.13)$$

where R_c is the contact resistance, ρ is the resistivity, H is the hardness and F is the applied load, calculated for a circular contact area. Although Holm's model assumes smooth areas, homogeneous materials and isotropic conductivity, the composites fabricated in this project most likely will be porous, have rough surfaces and oxide layers. Therefore, Holm's model is not expected to quantitatively predict the measured contact resistance in this work. Instead it provides a relationship with a useful qualitative framework for interpreting how changes in hardness may influence the measured contact resistance.

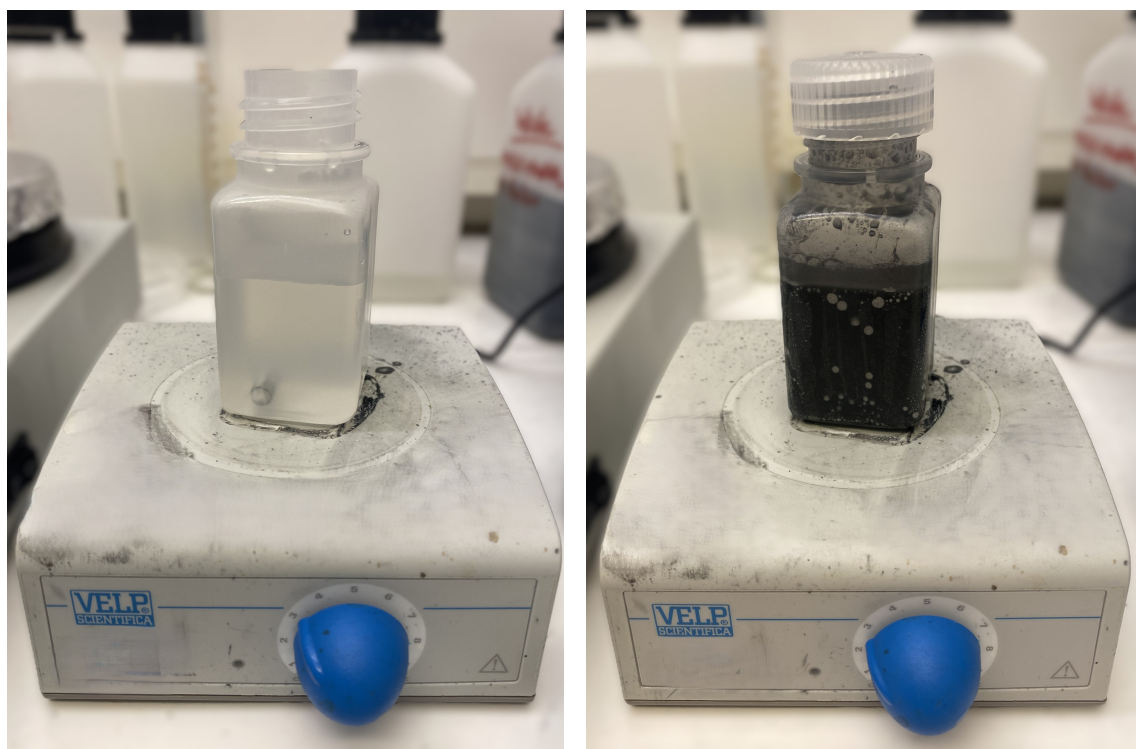
3

Method

3.1 MXene synthesis

3.1.1 Combining HCl, LiF and Ti_3AlC_2

The in-situ HF process starts with adding the necessary ingredients to remove, etch, the aluminium from the MAX-phase. In a plastic bottle 10 mL distilled water was added. The bottle was placed on a magnetic stirrer, with a magnet mixing the solution, and 30 mL HCl (fuming 37% from Supelco) was then added.



(a) Before adding MAX phase TiAlC_2 . (b) After adding MAX phase TiAlC_2 .

Figure 3.1: Figures of the etching process. Before adding MAX phase the bottle contains an in-situ HF solution from mixing LiF and HCl. After adding the MAX phase an exothermic reaction between the HF and Ti_3AlC_2 begins and therefore also the etching process.

On a scale, 3.2 g LiF (97% from thermo scientific) was measured and added to the

bottle while stirring. After about 5 minutes the mixed ingredients were combined and it was time to add the MAX phase (from Carbon Ukraine, particle size $< 40 \mu\text{m}$). In Figure 3.1 we can see the before and after of adding the MAX phase Ti_3AlC_2 visually. On a scale, 2 g of Ti_3AlC_2 was measured and added little by little to the etchant. When all MAX phase was added the lid was placed on top of the bottle, however, not screwed tight. This reaction was stirred for 24 h at room to complete the etching reaction.

3.1.2 Washing

After 24 h of mixing it was time to clean out the acidic mixture by washing and centrifuging. The etchant was poured into centrifuge tubes and underwent its first centrifuging cycle. This cycle lasted for 5 min and at 3500 rpm. The heavier particles, like MXene, unetched MAX phase and aluminium, will during the process be pushed to the side of the tube while the acidic supernatant will be poured out. The tube was then filled with distilled water and underwent another cycle. This process was then repeated. The first supernatant will have a greenish colour and a pH of 1, the rest will be different shades of see-through and by the last cycle the supernatant is black and no longer see-through. These supernatants are displayed in Figure 3.2. At this point the mixture have reached a pH around 5 and is now ready for delamination. A pH of approximately 5 was chosen to ensure the removal of acids while maintaining colloidal stability of the MXene suspension.

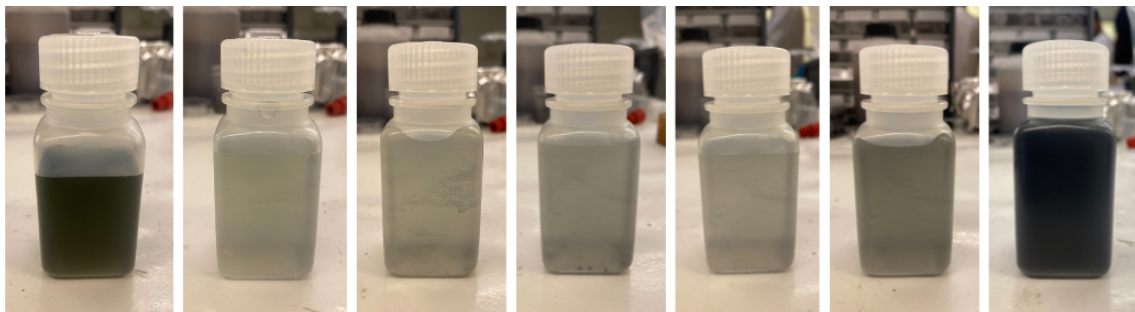


Figure 3.2: Figure showing the collection of the supernatant in the washing phase of the synthesizing process. The first supernatant have a dark green colour an pH 1, the next ones are see-through with increasing pH and the last one is coloured black with a stabilized pH of 5.

Now, to separate the stacks of MXene into single- or few-layered sheets the tubes are exfoliated. This was done through 5 cycles of mild ultrasonication for 10 s and light handshaking.

After the exfoliation it was time for a slower speed centrifuging once more. Just as before around five cycles was done for 5 min at 2000 rpm. This time the supernatant was collected instead of discharged since by this cycling round the supernatant contains the MXene $\text{Ti}_3\text{C}_2\text{T}_x$. In this process, as a cause of the lower centrifuging speed, the heavier, larger MXene particles are separated from any remaining

unetched Ti_3AlC_2 . The sediment stuck to the centrifuge tube side, the pellet, contains these unetched MAX phase as well as smaller MXene particles, and for every centrifuging cycle this pellet seemed to visually become less black and more gray. Hence, more aluminium and less MXene in the pellet.

3.1.3 Concentration of MXene

From collecting the MXene, the water content in the solution is high, so to increase the concentration of the MXene one more round of centrifuging is needed. Filling centrifuge tubes with part of the MXene solution, it was centrifuged for 20 min at 10 000 rpm. This higher centrifuging speed ensures that the water separates from the MXene flakes. After this centrifuging cycle, the supernatant, containing mostly water, was removed and replaced with additional MXene suspension. The process was repeated until all of the MXene solution has been processed, resulting in a concentrated MXene slurry.

When the centrifuging part of this step was completed, and the MXene was a thick, black liquid, it was time to calculate the concentration of MXene in the water-based slurry. For this a TOB-DZF-6050 vacuum lab furnace was used. Some of the slurry was put in a glass jar and weighted. This glass jar with MXene was covered with aluminium foil, with small holes poked, and put into the vacuum oven at 40°C for 20 h. During this time, the water content in the slurry was removed leaving dry MXene material. When weighting this glass jar once more calculations of the MXene mass fraction could be made using Equation 3.1.

$$w_{MXene} = \frac{m_{dry}}{m_{slurry}} \quad (3.1)$$

3.2 Composite preparation

When the MXene mass fraction of the MXene slurry was known, the mass of the aluminium was calculated through equations 3.2.

$$m_{MXene, dry} = m_{MXene, wet} \cdot w_{MXene}$$

$$wt\%Al = \frac{m_{Al}}{m_{Al} + m_{MXene, dry}} \implies m_{Al} = \frac{wt\%}{1 - wt\%} \cdot m_{MXene, dry} \quad (3.2)$$

where $wt\%Al$ describes the weight percentage aluminium powder in the samples, m_{MXene} describes the weight of MXene in the sample, and where w_{MXene} describes the calculated mass fraction of MXene.

When preparing the composites, the required amount of MXene slurry was weighted out in a small container. The aluminium powder was then gradually added while continuously mixing with a needle in a fume hood to prevent metallic powder to spread in the lab. Small amounts of distilled water were added when necessary to maintain a workable consistency and to prevent the mixture from drying due to

the increased airflow in the fume hood. This manual mixing was continued until all the powder was added. The walls of the container was then used in a "lift-and-smear" method with a plastic spatula. This technique was mainly used to break the agglomerates, redistribute the powder and to spread the MXene throughout the slurry. This mixing continued until a visually homogenous slurry was obtained.

3.3 Freezing and freeze drying

When the composite was well combined it was covered with a thin paper, secured with a rubber band and placed in a bath of liquid nitrogen. The container was covered to reduce the amount of contaminations though still allowing water vapour to escape during the freeze drying. The samples were rapidly frozen by immersion in the liquid nitrogen and when the samples were completely frozen they were moved to the freeze dryer. This process took somewhere from 24 to 48 h, depending on the water content where more water equals longer time in the freeze dryer.

3.4 Pressing

When collecting the samples from the freeze dryer the samples were either in clumps or powders, this was based on the water content of the composites. With a mortar and pestle the lumps were ground into a fine powder. This powder was then poured into a mould and pressed with an hydraulic press. In Figure 3.3 a representative sketch shows the setup. As the hydraulic press was lowered, a thinner bar was pressed into the hole in the thicker cylinder where the powder was located. With this pressure the powders were formed into coins with a diameter close to 1 cm. These resulting compacted pellets are referred to as green bodies and weight around 0.4 to 0.6 g each. The added force onto the samples were approximately 8 ton, corresponding to a pressure of approximately 1 GPa, and the weight was applied for 30-60 s.

Pellets containing 0, 10, 20, 40, 60, 80, 90 and 100 wt% Al were created. For most compositions two samples were fabricated, this to ensure one green body and one sintered sample per composition. The exceptions were the 10 and 20 wt% Al samples where only one sintered sample were fabricated each, and the 40 wt% Al sample where two were fabricated, however both sintered.



Figure 3.3: Pressing setup. As the hydraulic press is lowered, the powder of the composite is pressed into a coin-shaped pellet. The added pressure was equal to around 8 ton and were applied for 30-60 s.

3.5 Sintering

For the sintering process a quartz tube furnace was used, seen in Figure 3.4. The sintering temperature of 550 °C was selected as it is below melting temperature for aluminium and within the range where $\text{Ti}_3\text{C}_2\text{T}_x$ MXene has shown to have structural stability in inert atmospheres.



Figure 3.4: The quartz tube furnace used for the sintering process of the Al/MXene composites in argon atmosphere.

The furnace program started at room temperature with an temperature increase of $5^{\circ}\text{C}/\text{min}$. At 550°C the temperature was maintained for 2 h, and after this dwell period, the furnace cooled down naturally while maintaining the protective argon atmosphere. This scheme is displayed in Figure 3.5.

The samples were positioned in an alumina boat crucible and was placed in the centre of the furnace. To prevent the neighbouring samples from interacting, the samples were spread 1-2 cm apart. The argon gas was connected to the tube with a continuous flow and the heating process was then initiated. The argon flow was first set to high to remove all air from the tube, but after approximately 30 min decreased to a steady lower argon flow.

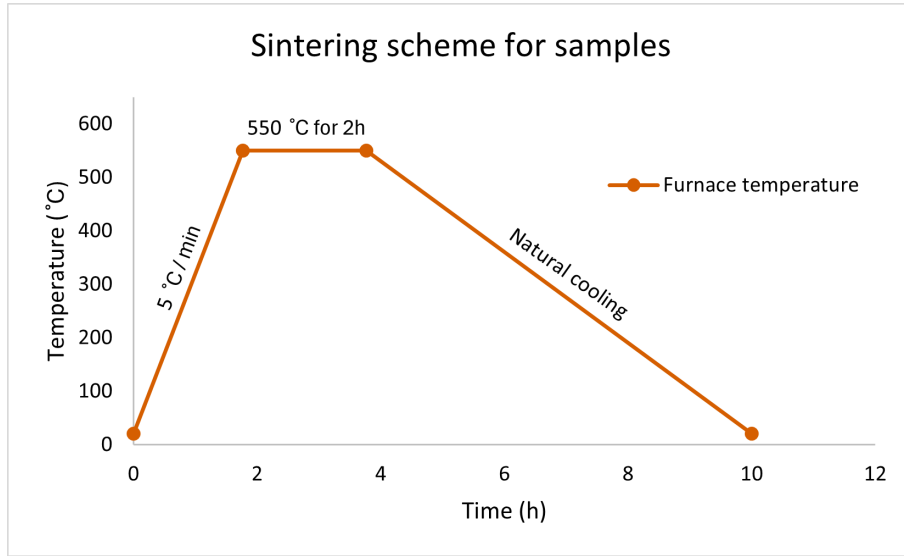


Figure 3.5: Sintering scheme used for all samples. The scheme starts from room temperature, with an increase of $5^{\circ}\text{C}/\text{min}$ up to 550°C , a dwell time of 2 h and ends with natural cooling until room temperature.

3.6 Electrical characterization

Electrical resistance measurements were performed using a four-probe setup, a micro-ohm meter, a newton meter and a contact resistance measurement setup connecting the meters and samples to each other. The experimental setup can be seen in Figure 3.6.

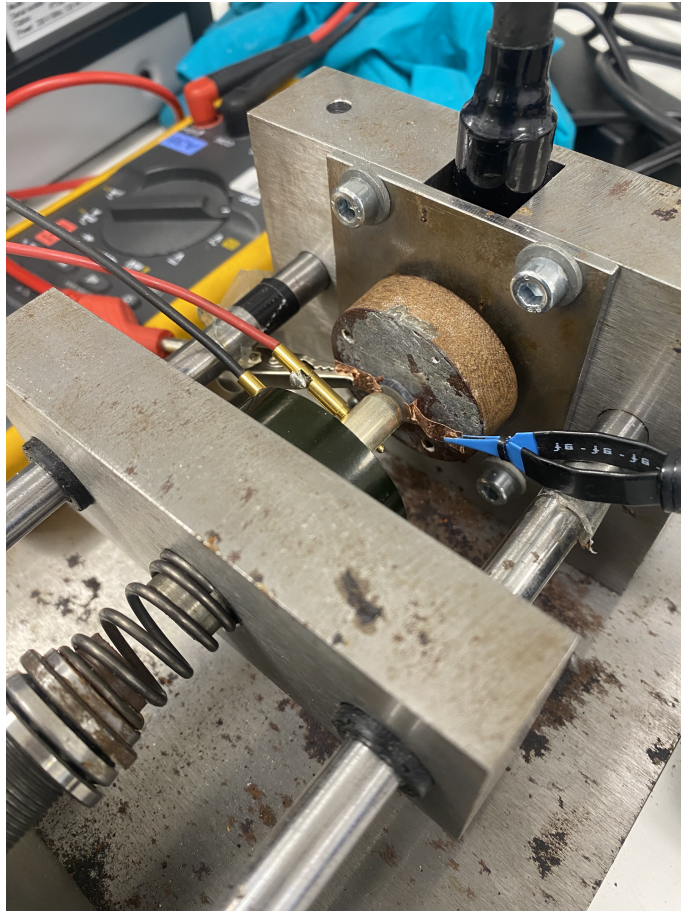


Figure 3.6: Experimental setup used for four-probe electrical resistance measurements as a function over applied contact load.

Since the pressed coin-sized pellets were quite small, two wing-like copper tape connections were added to the sides of the coin, this is visible in Figure 3.7. In these, two crocodile clips were attached and connected to a micro-ohm meter. From that same micro-ohm meter two other wires were connected to a silver contact that was then in turn connected to the sample. This silver contact was connected to a spring that can be wired up to apply force to the sample. When the measurement was initiated, a constant current was supplied through the outer probes while the voltage drop was measured using the inner probes. The silver contact, and connected spring, was attached to a newton meter and by increasing the load a relation between the contact resistance and the contact load can be observed.



Figure 3.7: Close up over the copper wings connected to the sample. This is a way to facilitate electrical contact during electrical resistance measurements.

For these measurements the contact load was ranging between 0 N to 110 N and would ideally be measured at least 2 times, with different placements of the silver contact, this to get a mean value of the measurements.

3.7 SEM/EDX characterization

Apart from measuring the conductivity we also want to understand how the composite is constructed. This will be a complementing study since the conductivity is heavily dependent on the structure, particle connectivity and composite homogeneity. For this, Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray spectroscopy (EDX) were performed.

SEM was used to investigate the morphology of the samples. This includes particle shape, porosity, cracking and possibly sintering neck formation. Before being analysed the samples were mounted on SEM stubs using conductive carbon tape.

In SEM imaging the accelerating voltage (EHT) was set to 5 kV, this relatively low voltage was selected to obtain high-quality images while minimizing the charging effects on the samples. For EDX, the EHT was increased to 15 kV, allowing sufficient X-ray generations for compositional analysis and elemental mapping. From EDX samples could be investigated by elemental distribution and the homogeneity of the sample. Most interesting were the distributions of the main components Al and Ti and the distributions of the surface terminations O and F.

3.8 Hardness test

When measuring the Rockwell hardness of the samples, a Ernst Bench hardness tester NR3D was used, see Figure 3.8. The indentation was made with a 1.59 mm ball, using the HRB scale, and the applied load was 5 kg, manually held in place for 3 s. The instrument reported the hardness as equivalent Vickers hardness values (HV5), and these values are therefore used throughout this work. For each sample 5 or more circular indents were made to calculate a mean value.



Figure 3.8: The hardness tester measured the hardness of the samples using a ball indenter in the HRB Rockwell scale.

The samples were mounted on the holder and elevated until tightly secured directly under the penetrator. After tested, the sample was moved slightly to test another area.

4

Results and Discussion

4.1 MXene synthesis and characterization

4.1.1 Appearance of the synthesized MXene

Following washing and concentration, the synthesized material formed a black suspension characteristic of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. After freeze drying, the suspension transformed into a lightweight porous monolith. The porous structure originates from growth of ice crystals during freezing, which templates the material. During freeze drying the ice sublimates, preserving the macroporous structure. Figure 4.1 shows a representative SEM image of the freeze dried material.

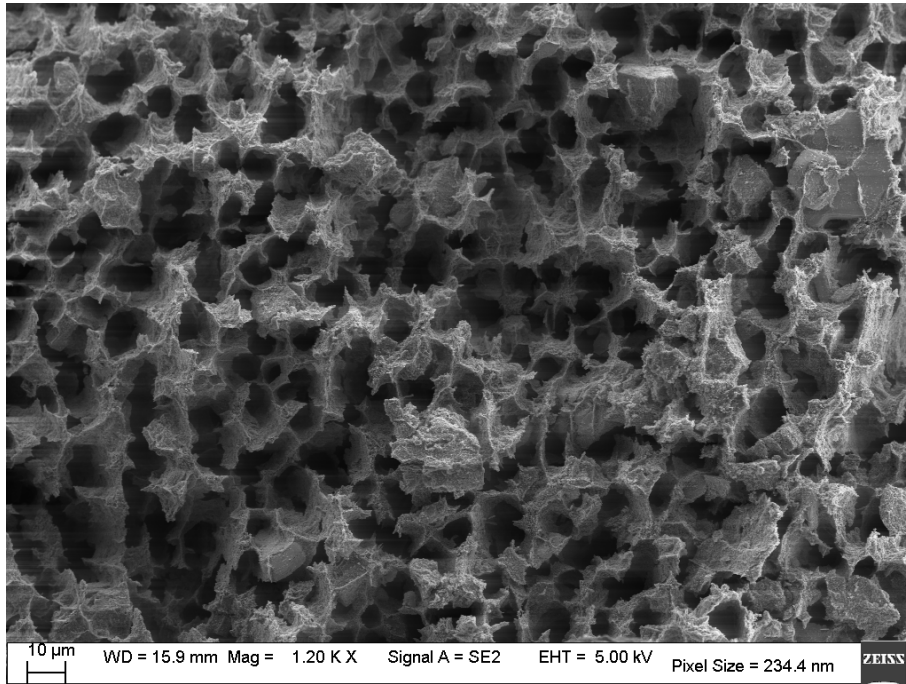


Figure 4.1: SEM figure of the MXene $\text{Ti}_3\text{C}_2\text{T}_x$.

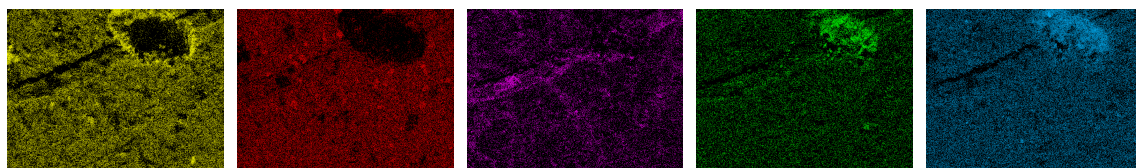
4.1.2 Microstructure of the freeze dried MXene

Figure 4.1 shows the morphology of the freeze dried material. Rather than dense particles, the sample exhibits an interconnected porous network with thin walls surrounding large pores. This morphology is characteristic of freeze dried suspensions,

as described above. Individual MXene sheets or well defined layered stacks could not be resolved at the magnifications used. Instead, the SEM primarily reveals the macroscopic architecture produced during freeze drying.

4.1.3 EDX analysis of the synthesized MXene

EDX analysis of the nominally pure MXene sample revealed the presence of fluorine, titanium, carbon, oxygen and aluminium, the maps displayed in Figure 4.2.



(a) Fluorine. (b) Titanium. (c) Carbon. (d) Oxygen. (e) Aluminium.

Figure 4.2: Figures of EDX mapping from a pure MXene sample. Reading showed high concentrations of fluorine, titanium and carbon and a surprising reading of aluminium.

The EDX analysis indicates the presence of 33.7 wt% F, 21.5 wt% Ti, 20.3 wt% O and 8.6 wt% Al. As seen in Figure 4.2, titanium was uniformly distributed throughout the analysed region. This is consistent with the presence of Ti-based MXene in a 0 wt% Al sample. Fluorine was also detected across the sample. This indicates fluorine surface terminations originating from the HF etching process. The relatively high fluorine content suggests that the synthesized MXene contains a considerable fraction of F terminations. A high fluorine content is generally associated with reduced electrical conductivity compared with other surface terminations, such as the oxygen terminations, and may have contributed to the relatively poor electrical performance measured for the pure MXene sample.

Oxygen was also detected throughout the analysed region. Since its distribution closely follows that of titanium, a substantial fraction of the oxygen is likely associated with oxygen surface terminations on the MXene rather than discrete oxygen particles. However, partial oxidation of the sample during processing or handling cannot be excluded and in the top right corner some localized oxide formation is visible.

Aluminium was also detected, although an ideal etching process would have had it removed. Several explanations are possible. Residual aluminium may originate from unetched MAX particles, residual aluminium containing reaction products or local regions where the etching reaction was incomplete. Since the aluminium appears relatively homogeneously distributed, widespread contamination is considered less likely. However, since SEM did not reveal obvious MAX particles, it is difficult to determine the dominant source of the residual aluminium.

Carbon was also detected but not discussed quantitatively because of the limited accuracy of EDX for light elements.

4.2 Fabrication of MXene-Al composites

The fabrication of the MXene-Al composites was successfully completed for all compositions. All composites could be successfully pressed into green bodies and all intended sintered samples completed the heating treatment without visible degradation. Samples with very low aluminium content, 10 wt% and 20 wt% however, did not handle keeping intact very long after pressing, even when sintered. The samples with 40 wt% and 60 wt% exhibited insufficient mechanical integrity upon added contact load, as well as the 80 wt% green body. Samples with higher aluminium contents however, showed improved structural integrity. This demonstrates the importance of the aluminium metal matrix for maintaining particle connectivity and mechanical integrity.

One other measure of fragility came with the hardness tests. Only the higher Al-content samples could be tested without breaking. In Table 4.1 a summarization of the samples and what characterization was made.

Table 4.1: Summary of fabricated samples and completed characterization.

Composition	State	Electrical characterization	Hardness characterization	SEM/EDX
0 wt% Al	Green	No	No	No
0 wt% Al	Sintered	Partial	No	Yes
10 wt% Al	Sintered	No	No	No
20 wt% Al	Sintered	No	No	No
40 wt% Al	Sintered	Partial	No	Yes
60 wt% Al	Green	No	No	Yes
60 wt% Al	Sintered	No	No	Yes
80 wt% Al	Green	No	No	Yes
80 wt% Al	Sintered	Yes	Yes	Yes
90 wt% Al	Green	Yes	Yes	Yes
90 wt% Al	Sintered	Yes	Yes	Yes
100 wt% Al	Green	Yes	Yes	Yes
100 wt% Al	Sintered	Yes	Yes	Yes

Because several low-aluminium compositions fractured during handling, the subsequent electrical and mechanical analysis mainly focuses on composites containing 80 wt% aluminium or higher.

4.3 Electrical characterization

4.3.1 Resistance as a function of contact load

Electrical resistance measurements were performed according to the procedure described in Chapter 3.6. The measured contact resistance was recorded as a function of applied contact load. Copper and silver reference samples were also tested to provide a comparison with highly conductive materials. The results can be seen in Figure 4.3.

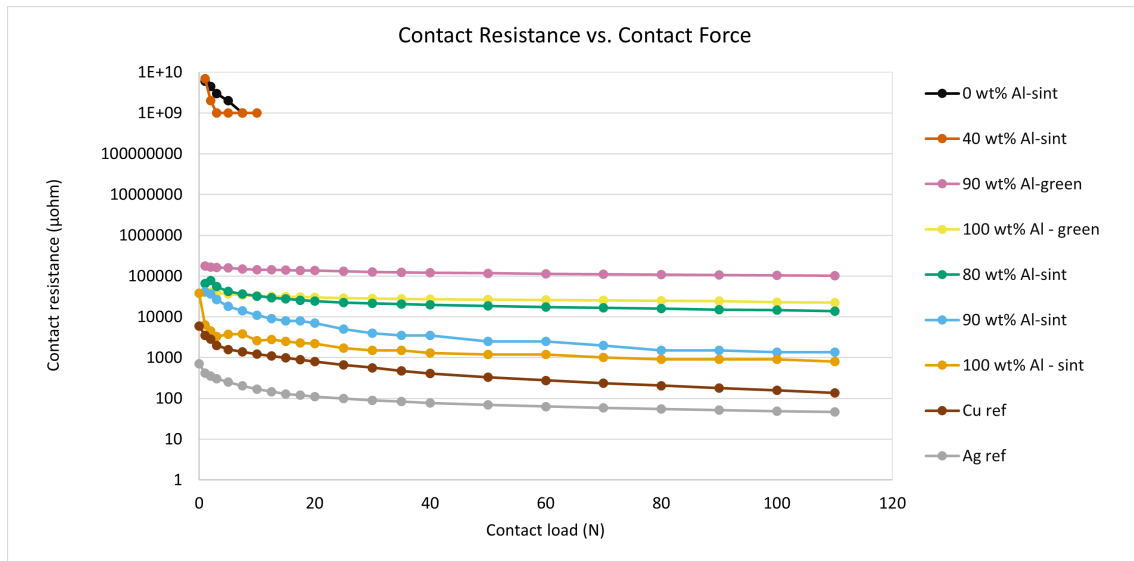


Figure 4.3: Contact resistance measurements over an increasing contact load. The data also includes two reference samples with copper and silver. The obtained data shows increasing aluminium content resulted in lower measured contact resistance, and that sintered samples perform better than green bodies.

For all samples, the measured contact resistance decreased with the increasing contact load before approaching a more stable value at higher loads. This trend is expected since the increasing contact pressure increases the effective contact area between the electrodes and the sample surface, and therefore reducing the contribution from contact resistance.

The results indicates that a clear trend can be observed where increasing aluminium contents resulted in lower measured resistance. Since aluminium is a highly conductive metal, the number of conductive pathways within the composite increases with increased aluminium content. On the other hand, lower aluminium content samples demonstrate higher resistance and weaker mechanical stability. The higher resistance of the low-aluminium composites suggests that a continuous metallic network was not fully established.

As foreseen, the reference samples copper and silver showed the lowest measured contact resistance throughout the entire loading range. These measurements there-

fore served as practical lower-resistance references and confirms that the setup and method can separate highly conductive materials with less conductive materials.

Both the 0 wt% Al and 40 wt% Al samples fractured before completion of the measurement series, as can also be observed in Figure 4.3. This gives an indication of poor mechanical integrity in MXene-rich compositions and this supports that the aluminium contributes substantially to the structural stability of the composites.

While aluminium content clearly strongly effects the resistance of the composites, the results also indicates a strong influence of the sintering process. The electrical properties from the sintering will therefore be discussed in the following section.

4.3.2 Effect of sintering

To evaluate the effects of the sintering process the electrical conductivity data obtained from the 90 wt% Al and 100 wt% Al samples were compared. In Figure 4.4 it is visible that there is a clear decrease in contact resistance between the green bodies and the sintered samples.

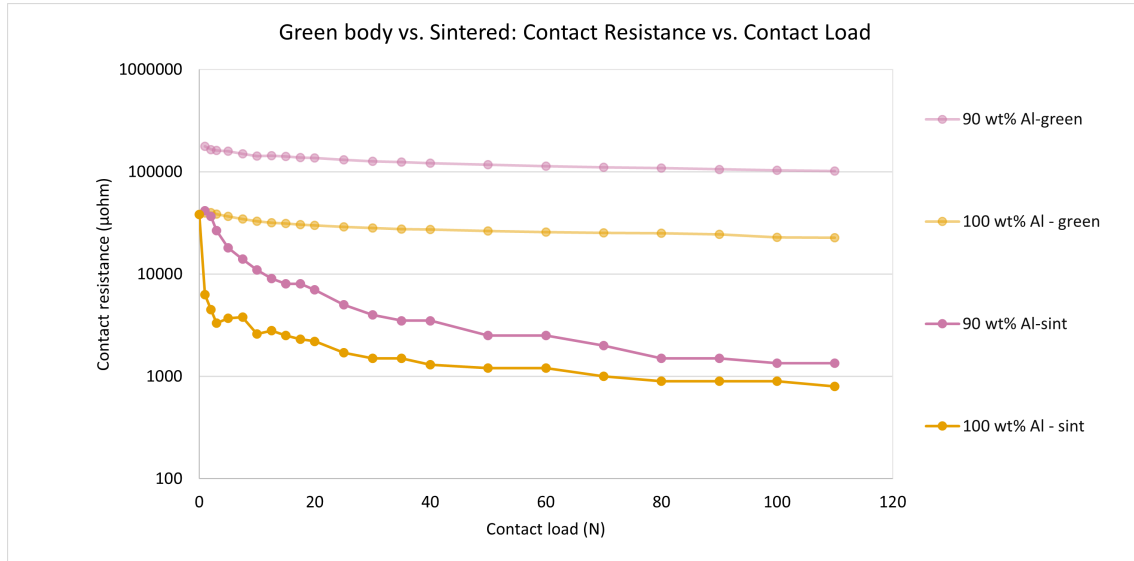


Figure 4.4: Graph of the green bodies and sintered samples. A clear decrease in contact resistance between the green bodies and sintered samples resulted in an improvement factor of 28-fold for the 100 wt% Al sample and 75-fold for the 90 wt% Al sample at 110 N contact load.

At maximum load, 110 N, the 90 wt% Al composites showed that sintering reduced the resistance from 101 500 $\mu\Omega$ to 1350 $\mu\Omega$, which corresponds to an approximate decrease of 98.7 %, which translates to a 75-fold improvement in electrical performance. For the 100 wt% Al samples the reduction went from 22 600 $\mu\Omega$ to 800 $\mu\Omega$, which is a decrease around 96.5 % and an approximate 28 times improvement in electrical performance. These results can be seen in Table 4.2.

Table 4.2: Table of four compositions, 100 wt% Al green and sintered, and 90 wt% Al green and sintered, compared to their contact resistance at 110 N contact load. Also displayed is the resistance reduction and improvement factor between the green bodies and sintered samples.

Composition	Green ($\mu\Omega$)	Sintered ($\mu\Omega$)	Reduction (%)	Improvement factor
100 wt% Al	22600	800	96.5	28×
90 wt% Al	101500	1350	98.7	75×

These results indicate that the effects of sintering are substantial and that the 90 wt% Al sample benefited more from sintering than the pure aluminium sample. This suggests that the MXene phase did not merely act as an inert filler, but also may have participated in the formation of conductive pathways in the composite. While the final resistance after sintering remained higher than that of pure aluminium the larger response to sintering indicates that the MXene containing microstructure underwent more significant changes during thermal processing.

The pronounced improvement of the 90 wt% Al composite further suggest that the MXene-containing composite initially had more poorly connected conductive pathways that improved significantly during sintering. During sintering, neighbouring aluminium particles form stronger interparticle contacts and may develop sintering necks, therefore reducing the number of poorly conducting interfaces and thereby helping electron transport throughout the composite. In addition, the MXene phase may contribute to the formation of conductive networks within the material. However, additional microstructural characterization is required to fully explain this behaviour. SEM imaging and EDX analysis were therefore performed to investigate the sintering effects on particle bonding, porosity and elemental distribution.

4.3.3 Effects of composition

One noticeable difference between the samples was that increasing aluminium content improved both the mechanical integrity and the electrical performance of the composites. The low-aluminium containing samples did either break before the measuring of contact resistance or during the applied load. This indicates that the aluminium fraction in the samples were insufficient and were unable to create a metal matrix connected enough to withstand mechanical burden. One other possible factor might be porosity in the samples. This would furthermore interrupt creation of a connected metal matrix. Porosity and possible cracking in the low-aluminium samples are discussed more in Section 4.5.2.

The connectivity of the metal matrix was visibly improved in the sintered samples. However, the low-aluminium samples were still fragile even after being sintered. By comparing the green bodies of the 60 and 80 wt% Al samples with the sintered, the green bodies were fragile but the sintered were not. The 40 wt% Al samples were fragile, even when sintered and the green body of the 90 wt% Al sample had good mechanical stability. These observations is naturally dependent on things like porosity and densification and in Section 4.5 the microstructure, such as porosity

and densification, are discussed as well as the aluminium dispersion in the samples, and some of their fragile behaviour can be explained there.

Upon the conductivity measurements some of the samples were at first difficult to get any form of reading from the micro-ohm meter. After some investigating the possible reason for this behaviour was concluded as oxide build-up. Aluminium rapidly forms a thin native oxide layer when exposed to air. Since aluminium oxide is electrically insulating, this layer can substantially increase the measured contact resistance. Light sanding removed much of the oxide layer, exposing fresh metallic aluminium and allowing stable electrical contact to be established.

4.4 Mechanical properties

4.4.1 Rockwell hardness

The measured values for the Rockwell hardness test ranged from 45 to 62 HV5 in the green body samples. The 80 wt% Al sample measured the highest hardness, whereas the 90 wt% Al measured the lowest. After sintering, the hardness of the pure aluminium sample decreased from 56 HV5 to 35 HV5, while the 90 wt% Al sample increased marginally from 45 to 50 HV5. The values were calculated as a mean value from multiple tests and is displayed in Table 4.3.

Table 4.3: Rockwell hardness test on five composites. The pre-selected values for the hardness was is in Vickers hardness scale HV5, however Rockwell hardness test were performed.

Composition	Hardness green (HV5)	Hardness sintered (HV5)
100 wt% Al	56	35
90 wt% Al	45	50
80 wt% Al	62	-

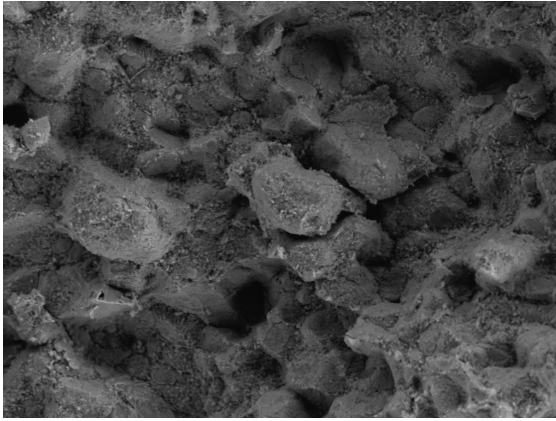
The hardness measurements did not declare a clear dependence on the composition of the composite. Some samples had very scattered readings, like the green body of the 90 wt% Al sample ranging between 35 HV5 to 59 HV5, and other had five consistent readings in a row, like the 100 wt% Al sample. The variability between measurements is likely due to local differences in porosity and microstructure. Therefore these hardness measurements are mainly used as a qualitative indicator of the mechanical integrity of the composites rather than for establishing quantitative trends between composition and hardness.

4.5 Microstructural characterization

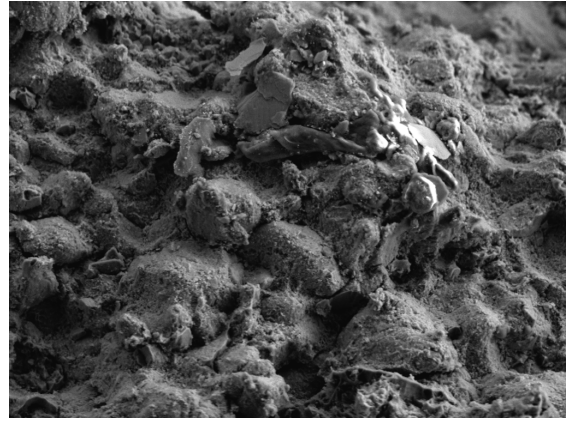
4.5.1 Green versus sintered microstructure

From the contact resistance measurement we learned that the electrical performance between the green body and the sintered 90 wt% Al sample were surprisingly high.

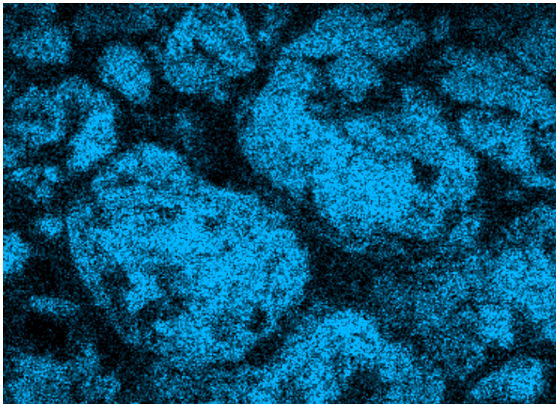
With an improvement factor being 75-fold it is very interesting to see what the samples look on the inside. In Figure 4.5a we see an SEM image of the 90 wt% Al green body and in Figure 4.5b we see the sintered version. The green body clearly separates the aluminium particles and this is even more visible in the EDX map of the aluminium distribution in Figure 4.5c. This result is expected since the composite at this stage is only mixed and pressed, and not treated with any thermal processing. In Figure 4.5b however, the particles are no longer distinctly isolated and visually more connected. While the SEM images suggest physical bonding between particles, the EDX maps in Figure 4.5d provide additional support where the aluminium distribution is more continuous between the particles after sintering. These observations are consistent with neck formation between neighbouring particles and probable cause for the improved electrical properties of the composite.



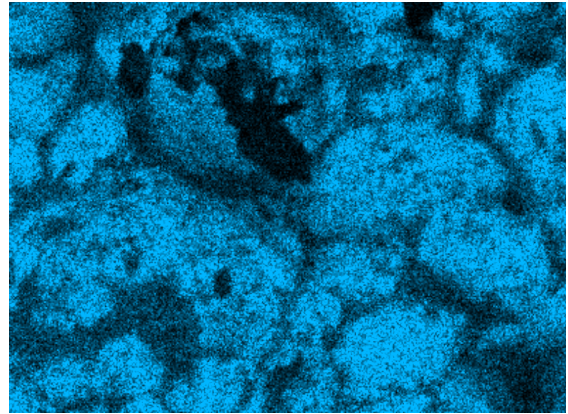
(a) SEM image of aluminium particles in a green body with 90 wt% Al, clearly separated from each other.



(b) SEM image of aluminium particles in a sintered sample with 90 wt% Al, showing signs of neck formation.



(c) EDX image of aluminium particles in a green body with 90 wt% Al, clearly separated from each other.



(d) SEM image of aluminium particles in a sintered sample with 90 wt% Al, showing signs of neck formation.

Figure 4.5: Comparison between SEM and EDX for a green body and a sintered sample with 90 wt% Al.

Besides neck formation, the sintered sample seems less porous than the green body.

This suggests that the sintering reduced the number of voids within the material and therefore increased the particle connectivity. The particles also appear being softer in shape and have fewer sharp edges. This also supports the occurrence of interparticle bonding caused by the sintering.

4.5.2 Porosity and cracking

The composites with higher amounts of MXene, such as the 10, 20, 40 and 60 wt% Al samples, became very fragile when handled in this project and had great difficulty or became impossible to run contact resistance measurements on. This was the result even when sintered for most of the low aluminium content samples. To investigate what this fragility comes from, SEM was made on the 40 wt% Al sample.

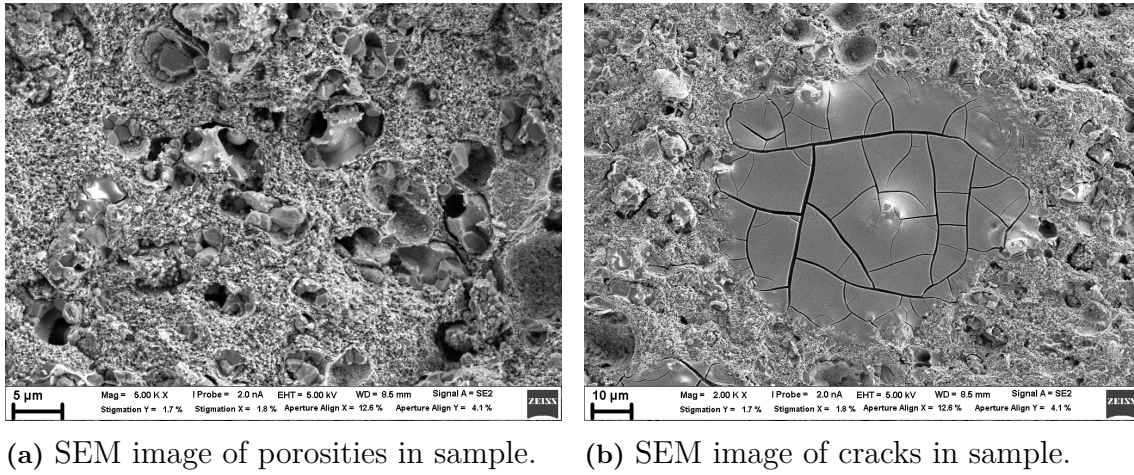


Figure 4.6: Two SEM images of the 40 wt% Al sample. These show both the porosity of the sample, but also imperfections like cracks on the surface.

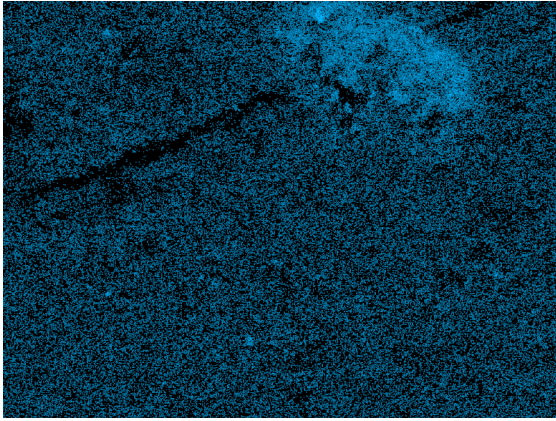
In Figure 4.6a and 4.6b two different detrimental microstructural features of the sample are shown. In Figure 4.6a many pores in the material are visible. The pores likely originates from an incomplete densification after freeze drying, pressing and sintering. With a porous sample, both the electrical and mechanical properties of the composite can be expected to be negatively influenced. Electrically, the voids will interrupt the conductive pathways for the electrons and neighbouring particles are less likely to form sintering necks, this since the particles are physically separated by the pores. Mechanically, they act as stress concentration sites that favour crack initiation and propagation. Such cracking can be seen in Figure 4.6b. Both these negative effects are consistent with the poor electrical performance and poor structural integrity of the lower aluminium content samples.

The occurrence of excessive amounts of porosity and cracking in the 40 wt% Al sample suggests that 40 wt% is an inadequate amount of aluminium to create a mechanically stable metallic network in the composite.

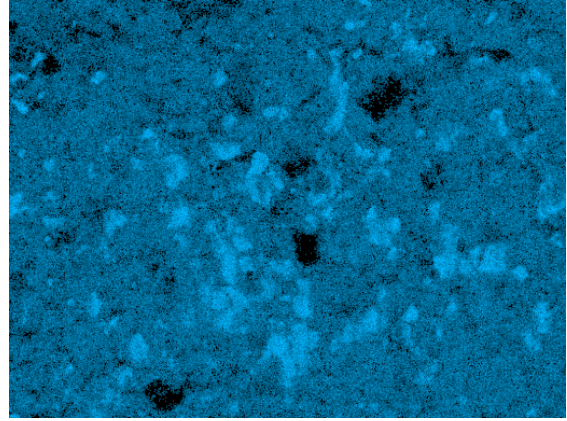
4.5.3 Elemental distribution

4.5.3.1 Aluminium distribution

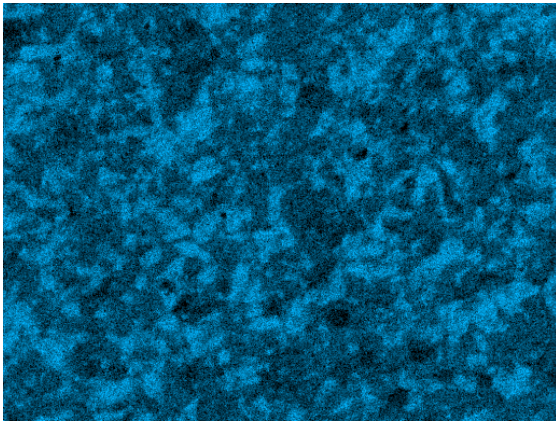
From Section 4.1 we established that, likely from an incomplete etching process, there still consists aluminium in the pure MXene sample. This conclusion was made based on the even distribution of the aluminium particles, so the risk of aluminium contaminations can be excluded. Figure 4.7 displays four distributions, these from 0, 40, 60 and 90 wt% Al samples. The 40 wt% and 60 wt% samples exhibit a less homogenous aluminium distribution than the 90 wt% Al sample. This may indicate incomplete dispersion of the aluminium powder during composite preparation or redistribution during later processing steps. This particle dispersion supports the impaired electrical performance of the samples as the connectivity of the aluminium is low. The 90 wt% Al sample however showed improved distribution which also improved the electrical performance of the composite. In a combined SEM and EDX analysis of a close-up of this sample, made in Section 4.5.1, physical connectivity between aluminium particles could actually be seen.



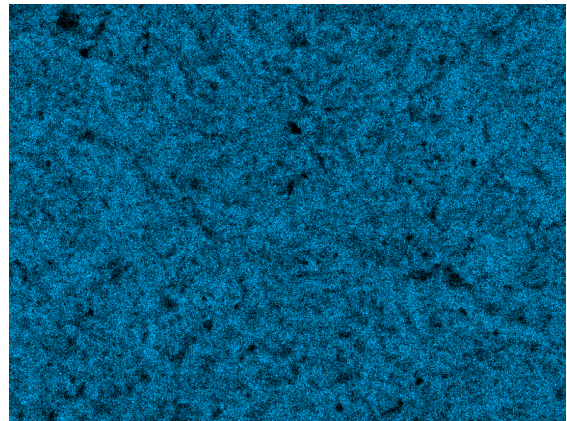
(a) Al distribution in 0 wt% Al sample.



(b) Al distribution in 40 wt% Al sample.



(c) Al distribution in 60 wt% Al sample.

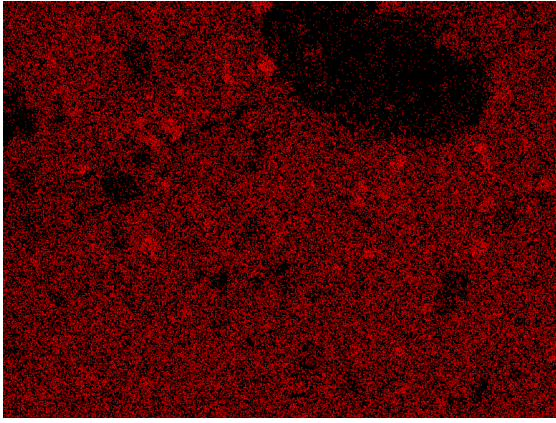


(d) Al distribution in 90 wt% Al sample.

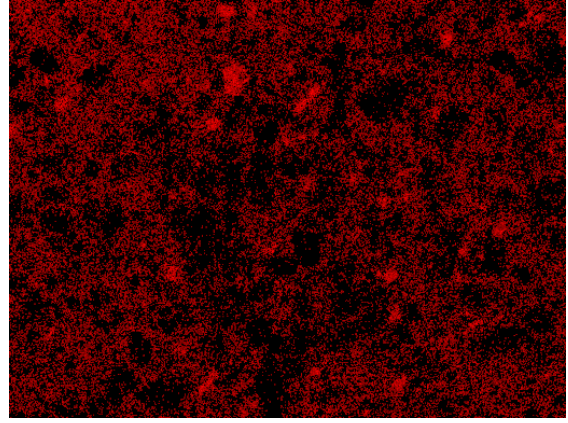
Figure 4.7: Aluminium distributions for the 40 and 60 wt% Al samples show poor distribution and inhomogeneity. In the 90 wt% Al sample there is good dispersion and in the pure MXene sample there are residual aluminium from the etching process present in the sample.

4.5.3.2 Titanium distribution

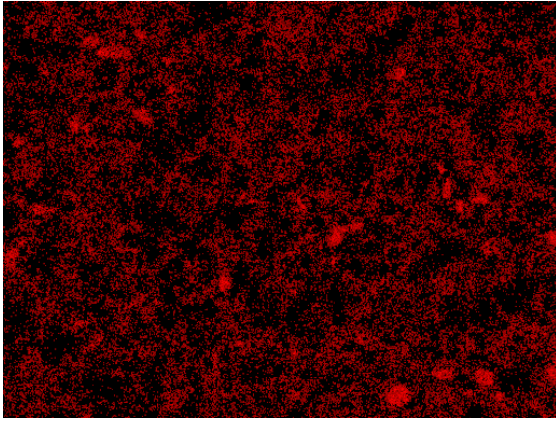
In Figure 4.8 the titanium distribution for the 0, 40, 60 and 90 wt% Al samples are visible. The titanium was used as an indicator of the MXene distribution within the composites, this since titanium only is present in the $\text{Ti}_3\text{C}_2\text{T}_x$ phase. As expected, the titanium signal decrease with lower MXene content samples, and shows the strongest signal for the pure MXene sample. Although the overall titanium content is lower in the aluminium-rich samples, an even distribution is still visible. This indicates that the MXene is evenly spread across the area and not located in secluded regions.



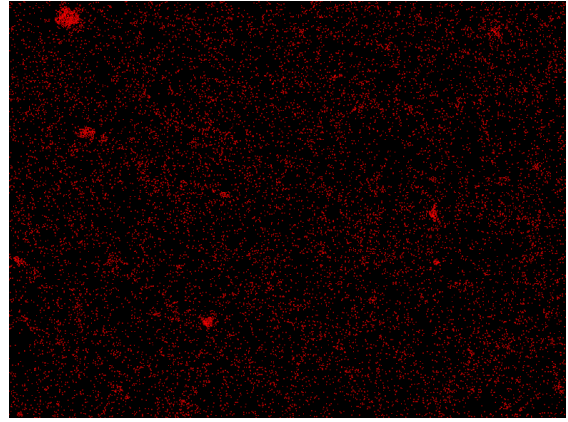
(a) Ti distribution in 0 wt% Al sample.



(b) Ti distribution in 40 wt% Al sample.



(c) Ti distribution in 60 wt% Al sample.



(d) Ti distribution in 90 wt% Al sample.

Figure 4.8: Titanium distribution, or rather the MXene distribution, for 40 and 60 wt% Al samples show inhomogeneous dispersion of MXene. The pure MXene show a good dispersion in the MXene-rich sample, and the 90 wt% Al sample show low Ti signal.

4.5.3.3 Oxygen distribution

Oxygen is one of the surface terminations expected in these samples. In Figure 4.9 the oxygen distribution of the 0, 40, 60 and 90 wt% samples are displayed. In the higher aluminium content samples the distribution is relatively homogenous, but for the lower aluminium content samples we can see a clear correlation of oxygen in

aluminium rich areas. In Figure 4.9a this is especially visible and might indicate the presence of aluminium containing oxide species associated with residual aluminium remains after etching. If the oxygen-rich areas corresponds to aluminium oxide formation, the results are consistent with the high contact resistance measurements since aluminium-oxide is electrically isolating. Localized oxygen enrichment might also indicate that the MXene sheets is partially oxidized and have formed TiO_2 -like species. These oxidations are expected to reduce the electrical performance of the composites.

Oxygen is an expected element in $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes due to the presence of the $-\text{O}$ and $-\text{OH}$ surface terminations formed during synthesis. Therefore, a homogenous oxygen distribution is not an unexpected part of any of these composites.

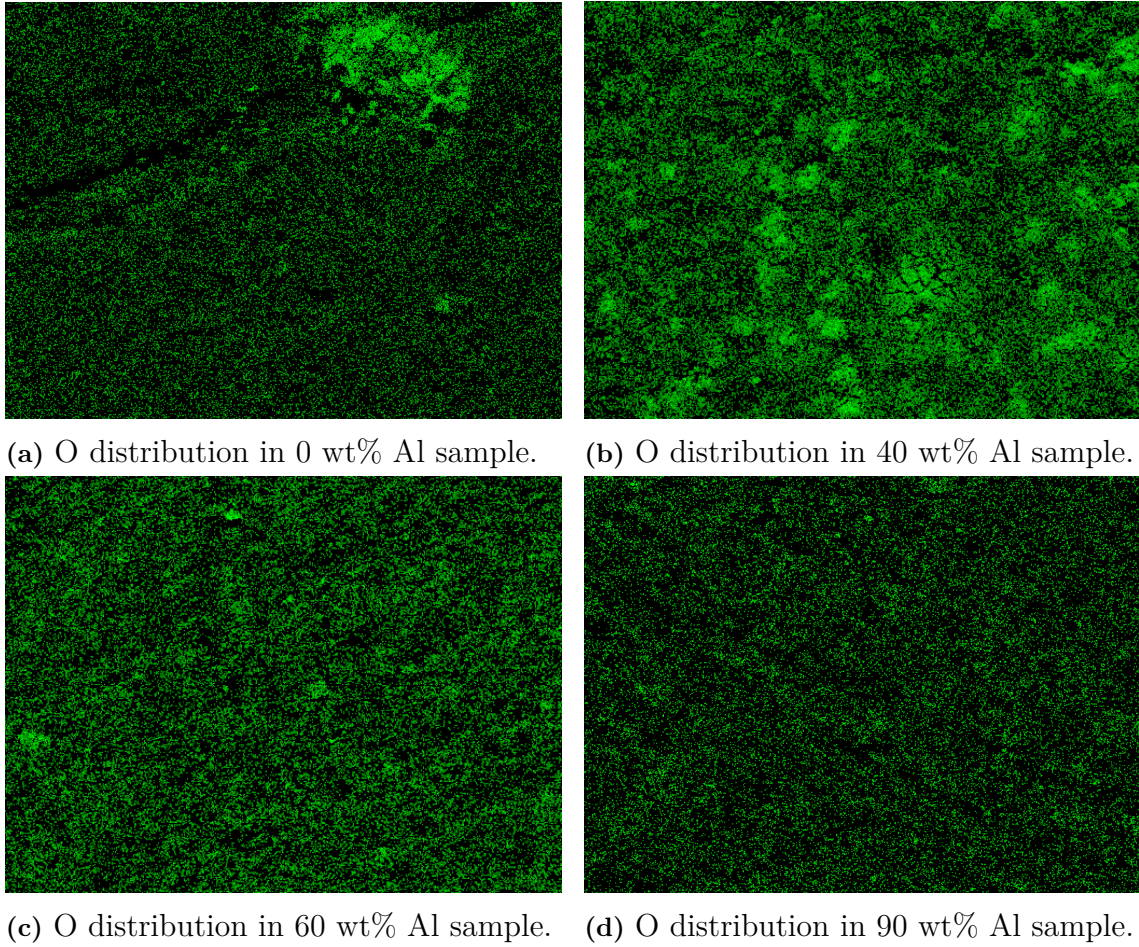


Figure 4.9: Oxygen distribution of pure MXene and 40 wt% Al samples show oxygen-rich regions. This displays signs of oxidation, either from Al or the MXene. The 60 and 90 wt% Al samples shows good dispersion, indicating that this oxygen are surface terminations of the MXenes.

4.5.3.4 Fluorine distribution

Fluorine is another surface termination expected in the composites. In Figure 4.10 the distributions for the samples of 0, 40, 60 and 90 wt% Al is shown. Just like for the titanium distribution, the amounts of fluorine decreases with increasing aluminium content. The fluorine distribution follows the titanium distribution of the composites, suggesting that they are connected to the MXene phase. This observation is consistent with fluorine acting as surface terminations of the $\text{Ti}_3\text{C}_2\text{T}_x$ MXene sheets.

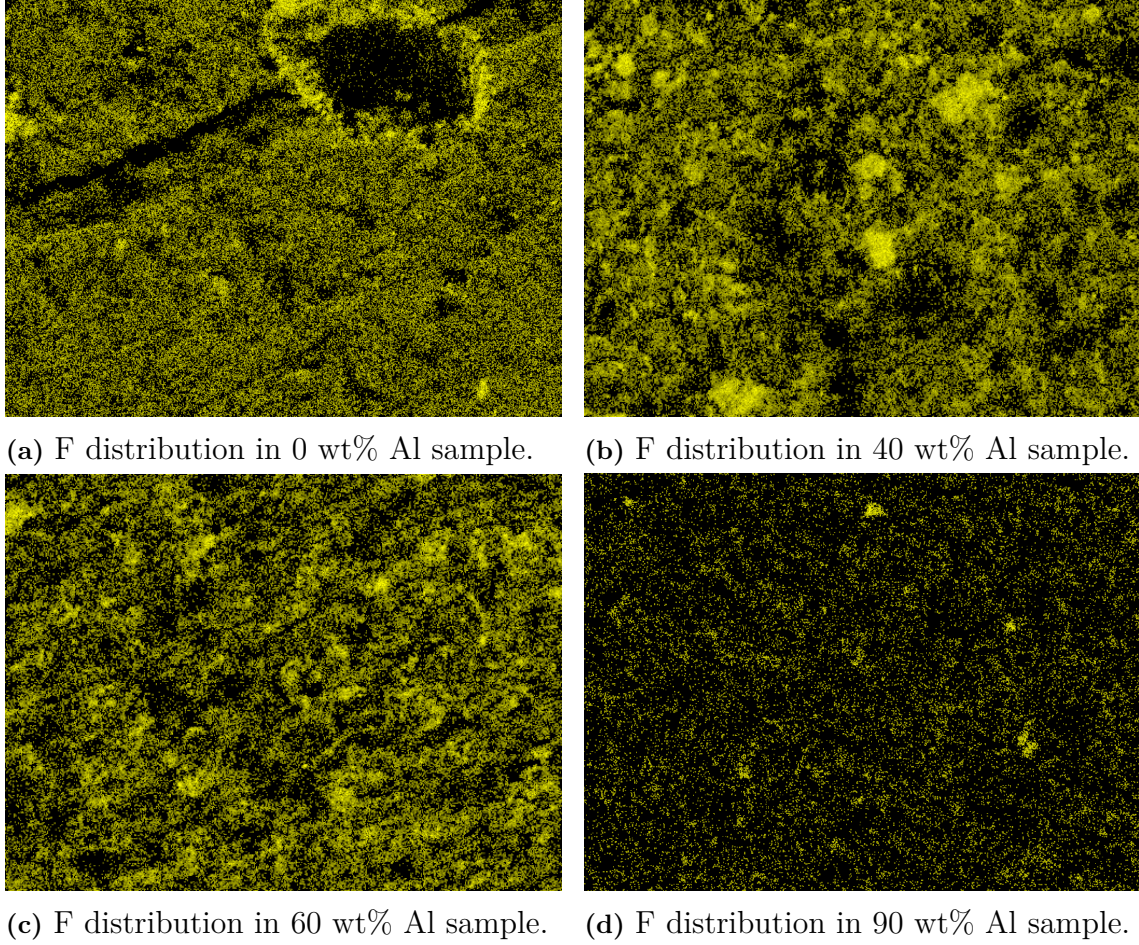


Figure 4.10: Fluorine distribution in all samples follow the titanium distribution, indicating that the fluorine acts as surface termination of the MXene. Low-aluminium samples show elevated signals of fluorine.

Surprisingly, the amounts of fluorine are higher than expected. In the purification process when synthesizing of the MXene, the specific surface terminations is randomized, and through in situ HF etching the fluorine amounts would be minimized. This however turns out to be not the case. This, in combination with the results about the remaining aluminium contents in the pure MXene sample, might indicate that the synthesizing process is not fully optimized and needs to be investigated further.

The higher amounts of fluorine in the lower-aluminium content samples are also consistent with the lower electrical performance on said samples.

4.6 Correlation between conductivity, hardness and microstructure

The conductivity and hardness results indicate that the composite properties are heavily controlled by the microstructural characteristics of the samples. Samples with high porosity and extensive cracking, such as the 0, 10, 20 and 40 wt% Al samples, exhibited low mechanical stability and high electrical resistance. At low aluminium contents, the composite structure was dominated by MXene-rich regions. Although MXene possesses high intrinsic conductivity, the porosity and limited particle connectivity of the sample resulted in high contact resistance and poor inter-particle bonding. This overall reduced the effective bulk conductivity of the samples. High aluminium content samples however, such as 80, 90 and 100 wt% Al samples, showed improved electrical performance and structural integrity. This behaviour is likely due to the improved particle bonding and the formation to a more continuous metallic network.

This improved particle bonding is attributed to the formation of sintering necks between neighbouring aluminium particles during heat treatment. These necks improved the electrical pathways between the particles which reduced the overall contact resistance between them and facilitated the general energy transport through the composite. In addition, a larger amount of aluminium in the samples reduces the number of poorly connected interfaces that the electrons travel, and this lowers the overall electrical resistance of the composite.

Furthermore, EDX analysis indicated that the presence of oxygen containing phases and fluorine rich surface terminations might contribute to the increase of electron scattering and interfacial resistance in the sample. Consequently, the electrical performance of the composites is controlled not only by the MXene's and aluminium's intrinsic conductivity, but also by the porosity, particle connectivity and chemical composition of the samples.

5

Conclusion

The aim for this project was to investigate the fabrication of aluminium/MXene composites and evaluate how varying aluminium contents and sintering processes influences electrical conductivity and microstructure of the resulting material.

The fabrication of the MXene $\text{Ti}_3\text{C}_2\text{T}_x$ from the MAX phase Ti_3AlC_2 was made through in-situ HF etching, washing, exfoliating and concentration. The synthesis produced a black MXene suspension that could successfully be pressed into freeze dried material. Through SEM the attempts of finding characteristic layered morphology were unsuccessful, however the characteristic network of sheets was visible in multiple SEM images. EDX displayed distributions of the MXenes main components Ti and C, as well as the surface terminations F and O. Also residual aluminium was confirmed through EDX. Some aluminium content was expected, however the amounts were more substantial and suggests incomplete etching. The residual aluminium together with the relatively high fluorine contents may have contributed to the poor electrical performance of both the pure MXene samples and the composites.

From the composite fabrication the results showed strong mechanical performance for the high-Al content samples and poor mechanical strength for the low-Al content samples. Upon further investigation through SEM imaging the low-Al content sample's fragility could be tied to poor particle connectivity through high porosity and cracking, as well as inhomogeneous aluminium distributions in the samples. This resulted in that some samples could not go through electrical characterization or hardness tests and broke either before or during tests. For the high-Al content samples the distribution of aluminium was improved and the porosity were lower as well, no visible cracking could be found and the overall strength was improved. For the high-Al samples a clear difference between sintered samples and green bodies could be seen. For the 60 and 80 wt% samples the green bodies were fragile and the sintered were not. This is likely due to improved particle connectivity through sintering necks and therefore decreased porosity of the samples.

From the electrical performance tests results showed increased performance with increased aluminium contents. The pure MXene sample showed very high resistance and by decreasing the MXene content in the composites the resistance decreased. The 80 and 90 wt% samples displayed the lowest resistance, however it did not surpass the performance of the pure aluminium sample. All the samples followed the same trend that the increased contact load decreased the contact resistance and stabilized at the contact load maximum at 110 N. Samples with lower aluminium

contents however, either broke before or during measurements giving non-existent or incomplete measurement series.

When comparing the green bodies to the sintered samples a clear improvement in contact resistance could be seen. For the 100 wt% Al sample the electrical improvement factor was 28-fold and for 90 wt% Al sample the improvement was 75-fold at the maximum contact load point 110 N. This result would suggest that the composite benefited more from the sintering than the pure aluminium sample, and also that the MXene in the composite acts as more than just a filler material. This suggests that the presence of MXene may influence the connectivity of the aluminium metal matrix after sintering.

Through characterization of the composites by SEM and EDX we could see the formation of sintering necks between green bodies and sintered samples in the 90 wt% Al samples. This is visible proof that the sintering increases the connectivity between neighbouring aluminium particles in the composites and correlates well with the improved electrical properties. The minimizing of porosities in the sintered samples compared to the green bodies may also indicate improved connectivity. In the 40 wt% Al sample porosities were visible even when sintered. These pores could also be connected to the high amounts of cracking also occurring in the sample. This increase in porosities and cracking displays poor connection between aluminium particles, even when sintered, and supports the poor electrical properties of the samples. EDX analysis for aluminium distribution also supports better electrical performance for high-aluminium contents, where the dispersion and homogeneity was much improved compared to low-aluminium composites. This is likely due to manual mixing developments whereas the high-aluminium samples were fabricated later in the project. EDX also disclosed high amounts of fluorine, a known surface termination of $\text{Ti}_3\text{C}_2\text{T}_x$. These high amount are likely also contributing to the overall lower electrical performance of the composites, this since fluorine disrupts the electron density of the MXene sheets.

From the hardness tests no clear connection between green bodies and sintered samples could be drawn. The 100 wt% aluminium sample indicated that the sintered hardness for the green body were higher than the sintered, however the results for the 90 wt% aluminium sample were the opposite. This in combination with some samples having very scattered readings does indicate that the test should not be interpreted as evidence of a systematic hardness trend.

Overall, the fabricated Al/MXene composites did not outperform pure aluminium in terms of electrical conductivity. Within the processing conditions investigated in this work, no direct electrical advantage of using $\text{Ti}_3\text{C}_2\text{T}_x$ as reinforcement phase in a aluminium metal matrix composite could therefore be stated. Nevertheless, the study provides valuable insight into how aluminium content, porosity, particle connectivity and sintering influence the electrical and mechanical behaviour of these composites. The identified limitations also provide a clear basis for future optimization of both the MXene synthesis and composite fabrication.

6

Future Work

This project has shown that aluminium/MXene composites can be fabricated and both the sintering process and aluminium content strongly influence the electrical and mechanical performance of the samples. However, several aspects require further investigation to better understand the material system.

The starting point for any future work on this project is the optimization of the MXene synthesizing. EDX analysis indicated residual aluminium in the pure MXene samples and relatively high fluorine contents. This suggests that the etching process was not fully optimized and can be looked into further. Future work should therefore explore alternative etching conditions, washing procedures and delamination methods to reduce the amount of residual aluminium and to modify the surface terminations of the MXene.

Microstructural characterization should also be further investigated, closely related the synthesizing process. Higher magnification SEM or transmission electron microscopy (TEM) could be used to view individual MXene sheets and the level of delamination achieved. X-ray diffraction (XRD) could also be used to determine whether residual MAX phase remains after synthesis and identify possible oxide phases formed during processing.

The low aluminium samples had high porosity and poor mechanical strength, therefore can further improvements in the fabrication process be made. Improvements, such as in powder mixing, freeze drying or pressing, may increase the densification and reduce cracking. The sintering process could also be investigated. Hot pressing or spark plasma sintering (SPS), which is more commonly used in powder composite fabrication, could be investigated to determine whether they improve densification and electrical conductivity.

A

Raw electrical resistance data

Table A.1: Data from the contact resistance vs. contact load measurement for various samples. For samples with multiple measurements an average is displayed.

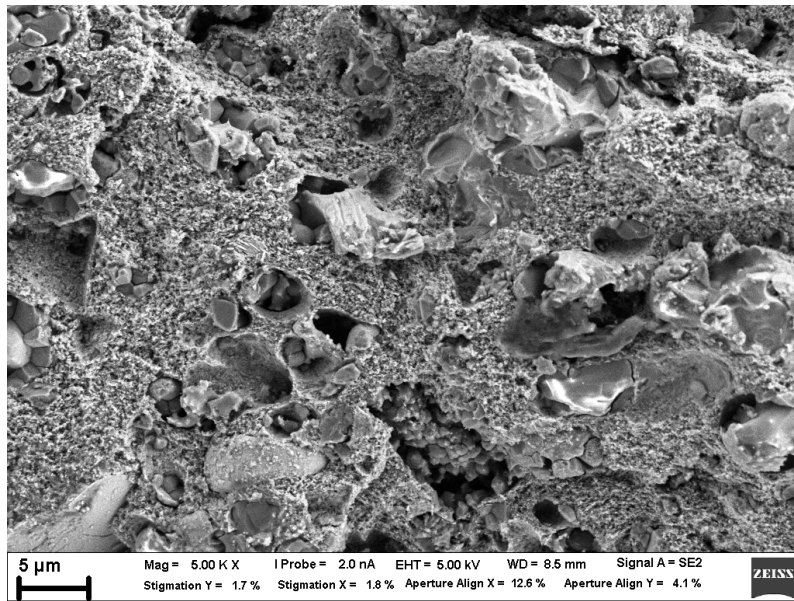
Load (N)	100 wt% Al Green ($\mu\Omega$)	100 wt% Al Sint ($\mu\Omega$)	90 wt% Al Green ($\mu\Omega$)	90 wt% Al Sint ($\mu\Omega$)	80 wt% Al Sint ($\mu\Omega$)
0	-	38100	-	-	-
1	39400	6300	178000	41500	66200
2	40000	4500	165000	36500	77250
3	38600	3300	162000	26500	55850
5	36500	3700	158000	18000	41900
7.5	34400	3800	149000	14000	36400
10	32700	2600	142500	11000	32350
12.5	31800	2800	143000	9000	29600
15	31200	2500	141000	8000	27650
17.5	30500	2300	138000	8000	25650
20	29800	2200	136000	7000	24150
25	28900	1700	130500	5000	22500
30	28200	1500	126000	4000	21450
35	27500	1500	124000	3500	20550
40	27200	1300	121500	3500	19800
50	26300	1200	117000	2500	18600
60	25800	1200	113500	2500	17450
70	25200	1000	110500	2000	16500
80	25000	900	108500	1500	15850
90	24400	900	105500	1500	15100
100	22900	900	103500	1350	14550
110	22600	800	101500	1350	13900

Table A.2: Data from the contact resistance vs. contact load measurement for various samples. For samples with multiple measurements an average is displayed. Here two reference samples, silver and copper, is included.

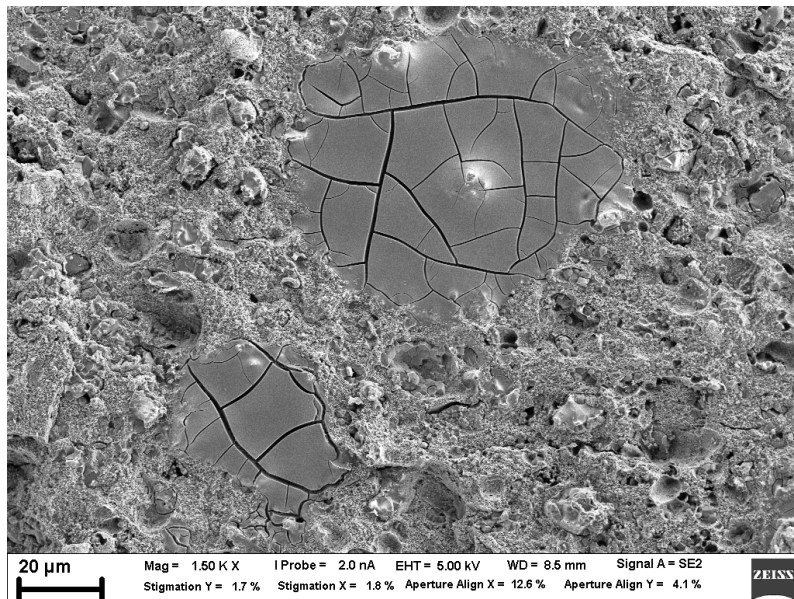
Load (N)	60 wt% Al Sint ($\mu\Omega$)	40 wt% Al Sint ($\mu\Omega$)	Pure MXene Sint ($\mu\Omega$)	Silver reference ($\mu\Omega$)	Copper reference ($\mu\Omega$)
0	-	-	-	700	5988
1	-	7×10^9	6×10^9	414	3501
2	-	2×10^9	4.5×10^9	350	2835
3	-	1×10^9	3×10^9	306	2004
5	-	1×10^9	2×10^9	250	1588
7.5	-	1×10^9	1×10^9	202	1382
10	150000	1×10^9	-	169	1224
12.5	-	-	-	147	1096
15	-	-	-	128	990
17.5	-	-	-	119	887
20	-	-	-	110	797
25	-	-	-	98	664
30	-	-	-	90	565
35	-	-	-	83	473
40	-	-	-	77	412
50	-	-	-	69	329
60	-	-	-	63	276
70	-	-	-	58	235
80	-	-	-	55	205
90	-	-	-	51	179
100	-	-	-	49	157
110	-	-	-	46	137

B

Additional SEM images



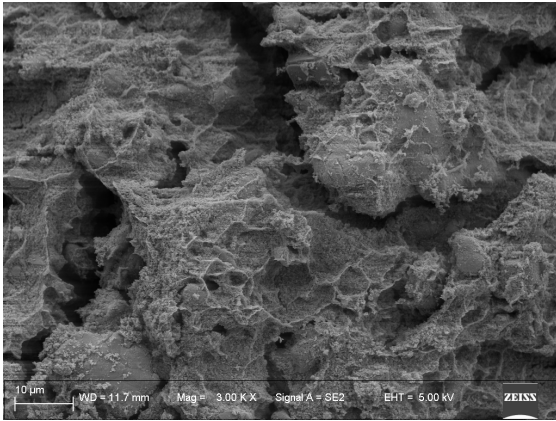
(a) Porous structure in sample.



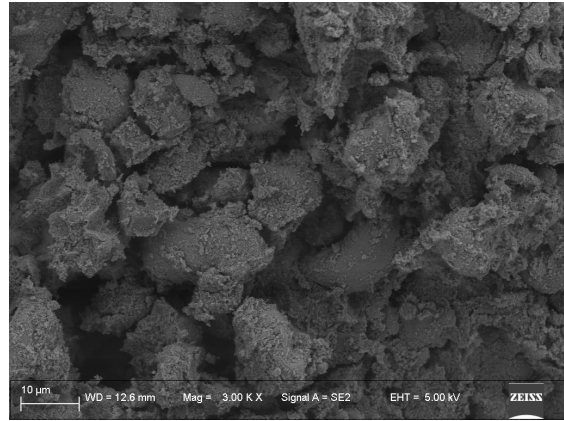
(b) Cracking visible in sample.

Bilaga B.1: SEM images of pores and cracks in the 40 wt% Al sample.

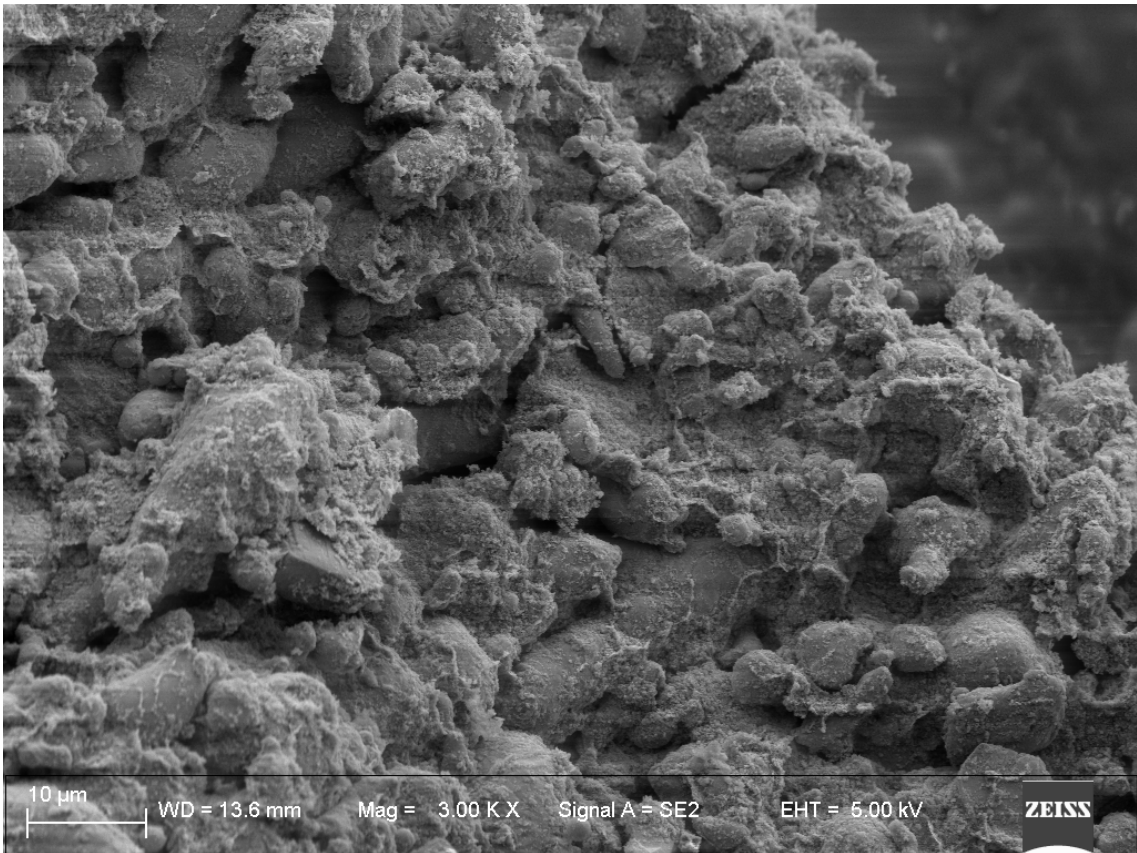
B. Additional SEM images



(a) 40 wt% Al composite powder.



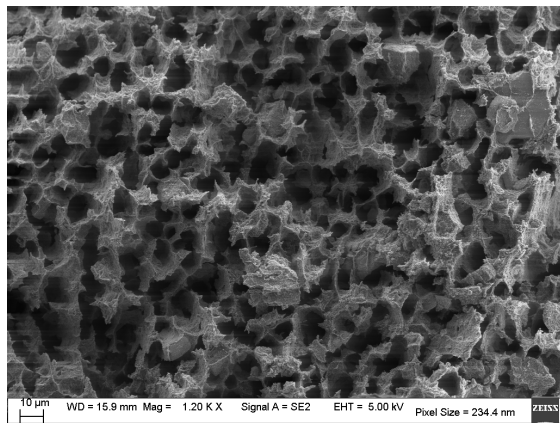
(b) 60 wt% Al composite powder.



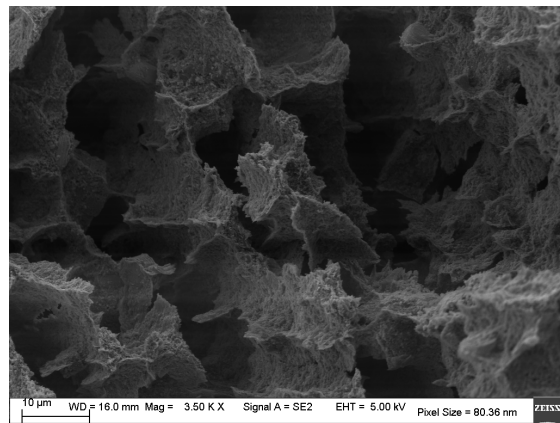
(c) 80 wt% Al composite powder.

Bilaga B.2: SEM images of three composite powders, all with different content of aluminium vs. MXene.

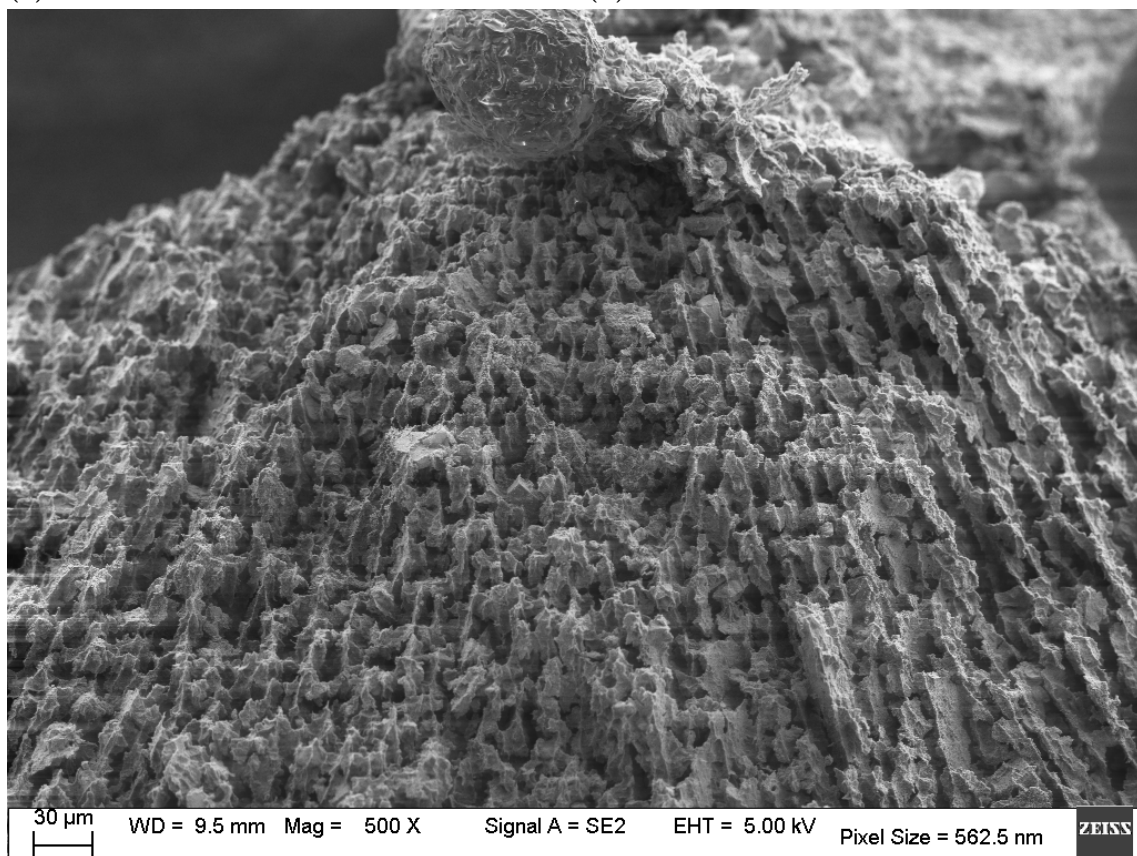
B. Additional SEM images



(a)



(b)

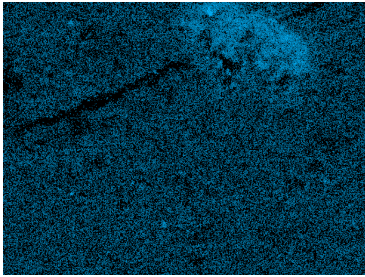


(c)

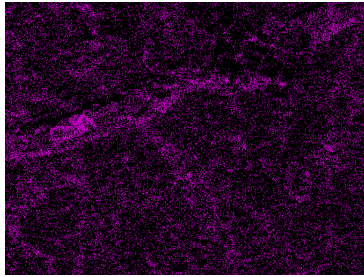
Bilaga B.3: SEM images of a freeze dried structure of a pure MXene sample.

C

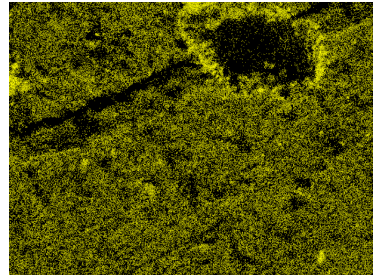
Additional EDX maps



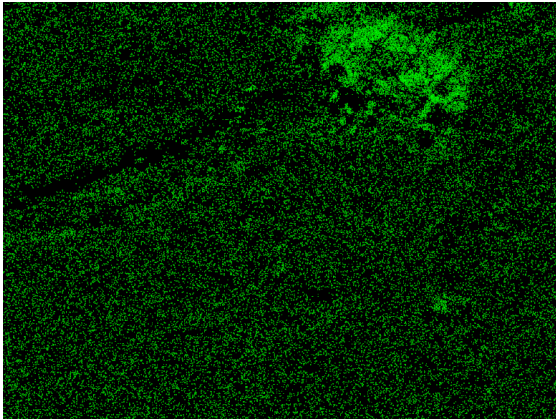
(a) Al distribution



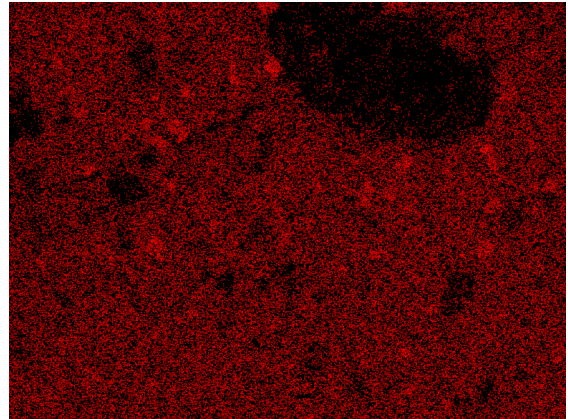
(b) C distribution.



(c) F distribution.

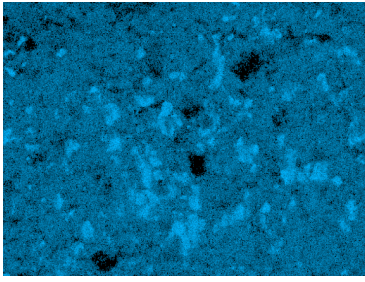


(d) O distribution.

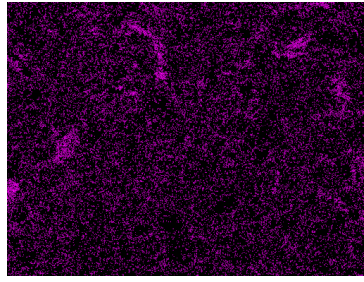


(e) Ti distribution.

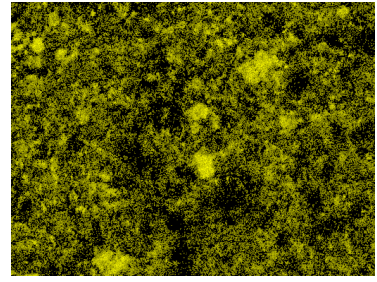
Bilaga C.1: EDX mapping of the pure MXene sintered sample.



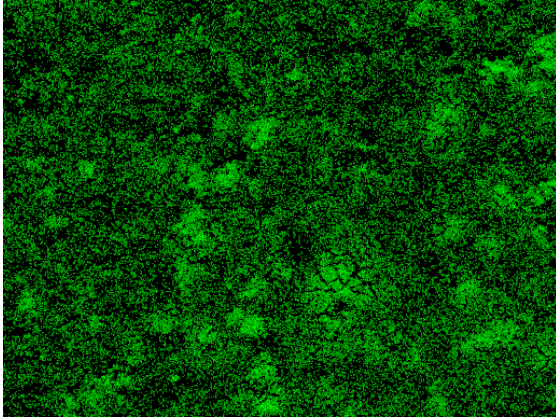
(a) Al distribution.



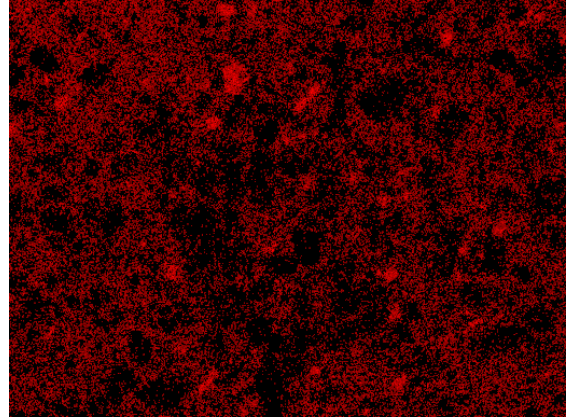
(b) C distribution.



(c) F distribution.

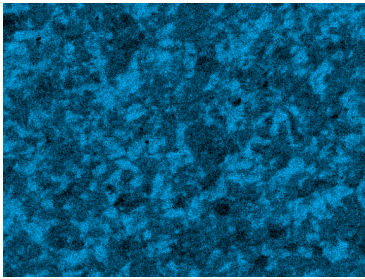


(d) O distribution.

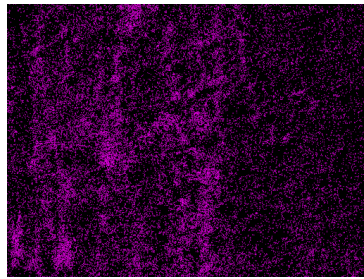


(e) Ti distribution.

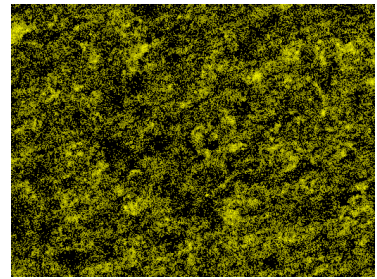
Bilaga C.2: EDX mapping of the 40 wt% Al sintered sample.



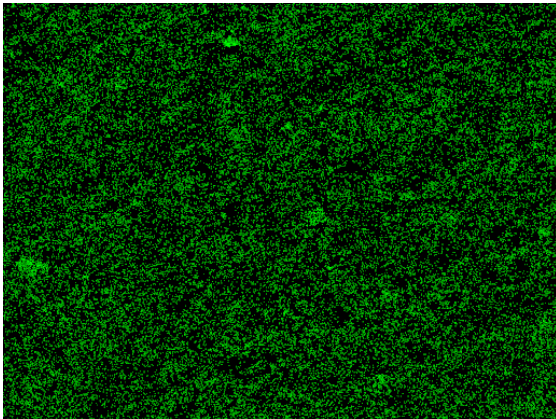
(a) Al distribution.



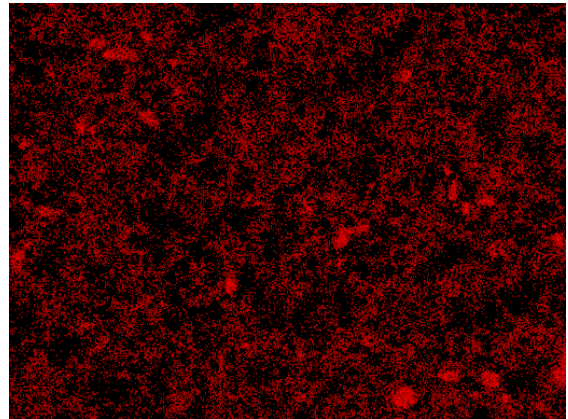
(b) C distribution.



(c) F distribution.

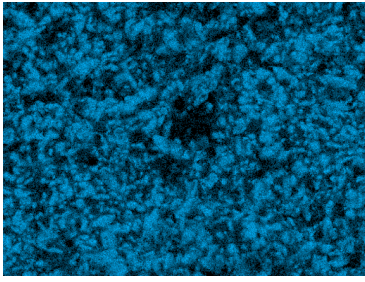


(d) O distribution.

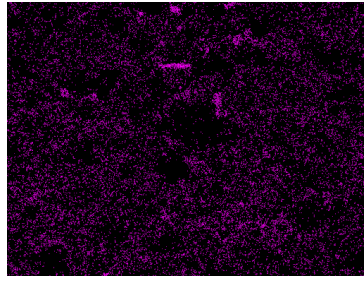


(e) Ti distribution.

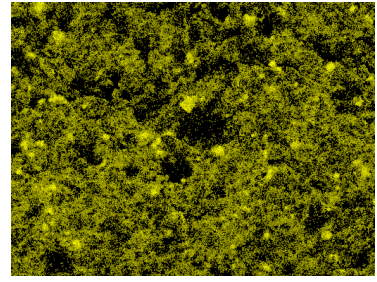
Bilaga C.3: EDX mapping of the 60 wt% Al sintered sample.



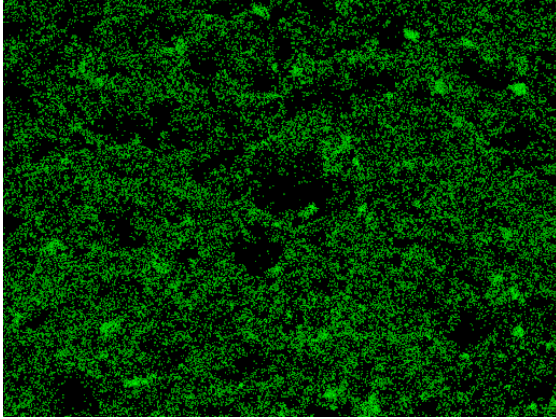
(a) Al distribution.



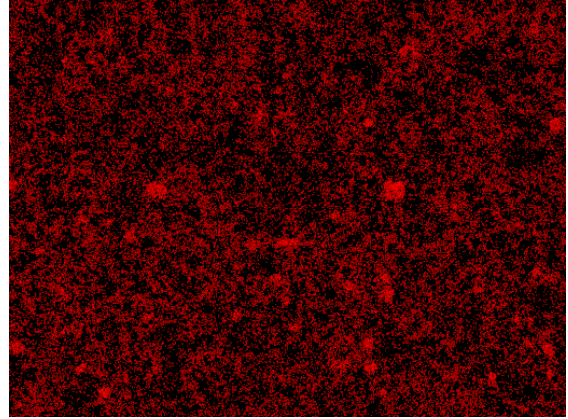
(b) C distribution.



(c) F distribution.

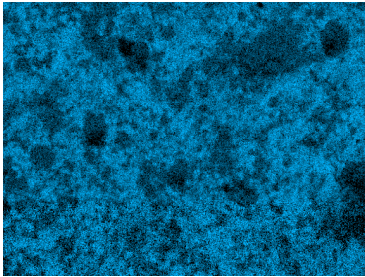


(d) O distribution.

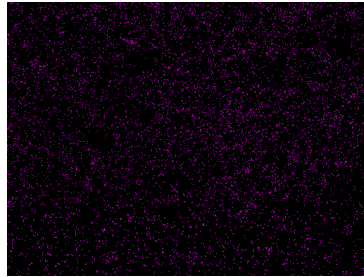


(e) Ti distribution.

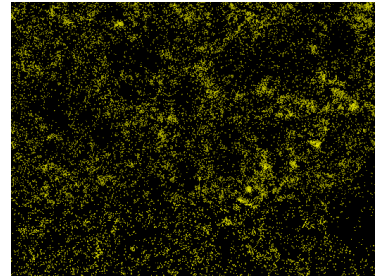
Bilaga C.4: EDX mapping of the 60 wt% Al green body sample.



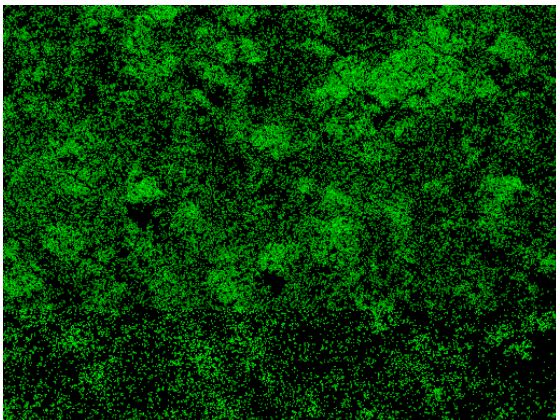
(a) Al distribution.



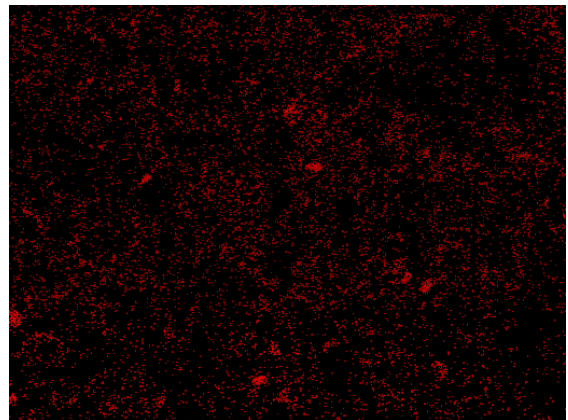
(b) C distribution.



(c) F distribution.

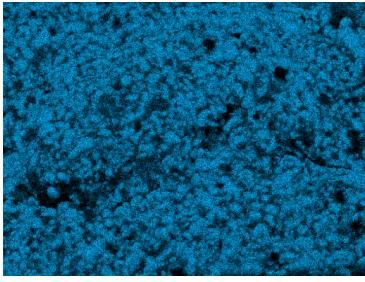


(d) O distribution.

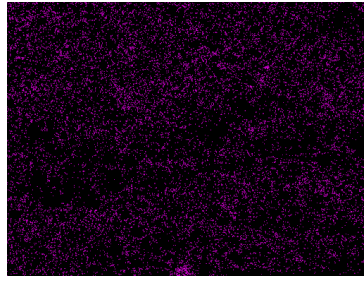


(e) Ti distribution.

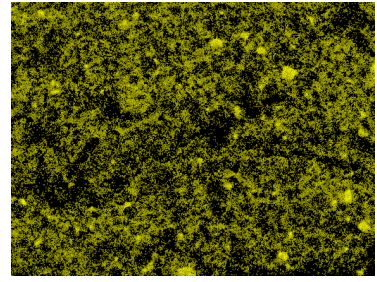
Bilaga C.5: EDX mapping of the 80 wt% Al sintered sample.



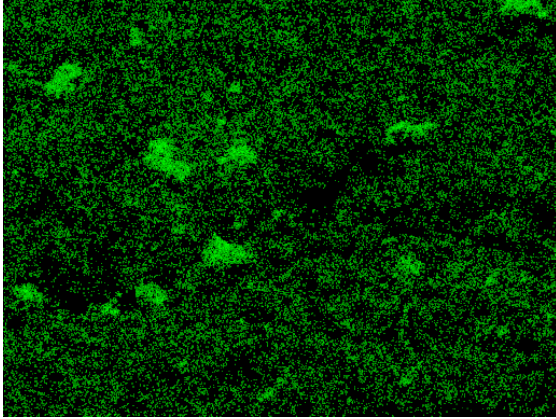
(a) Al distribution.



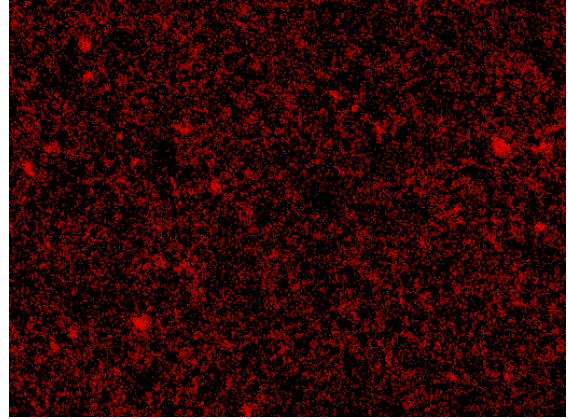
(b) C distribution.



(c) F distribution.

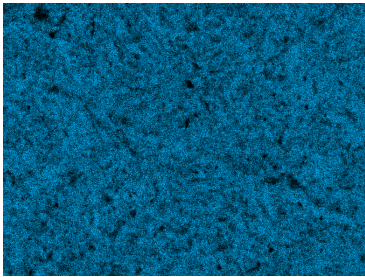


(d) O distribution.

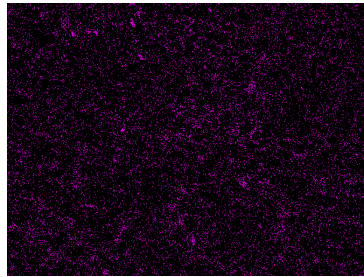


(e) Ti distribution.

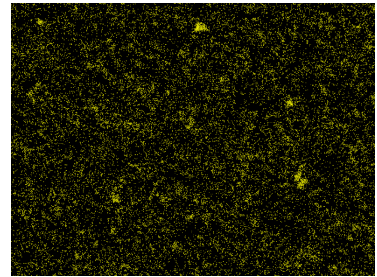
Bilaga C.6: EDX mapping of the 80 wt% Al green body sample.



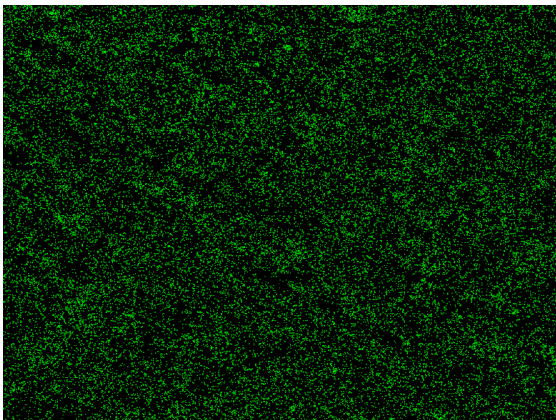
(a) Al distribution.



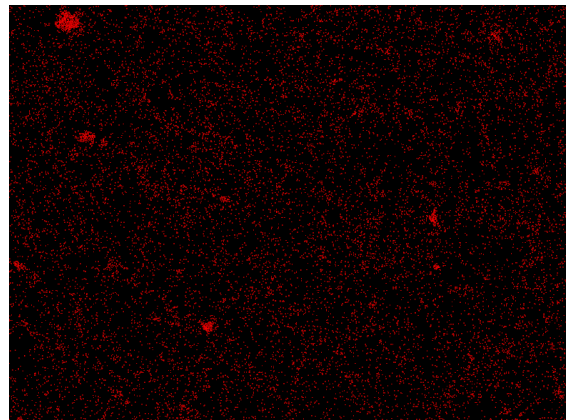
(b) C distribution.



(c) F distribution.



(d) O distribution.



(e) Ti distribution,

Bilaga C.7: EDX mapping of the 90 wt% Al sintered sample.

D

Hardness data

Table D.1: Data from hardness test. The test were made with Rockwell mechanics, however the received values are in Vickers scale (HV5).

Test number	100 wt% Al Green (HV5)	100 wt% Al Sint (HV5)	90 wt% Al Green (HV5)	90 wt% Al Sint (HV5)	80 wt% Al Sint (HV5)
1	64	35	51	43	62
2	53	35	35	51	64
3	56	35	39	43	63
4	55	35	35	46	67
5	57	35	59	42	58
6	56		35		
7			47		
8			48		

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