

ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL

**MATERIAL PROPERTIES OF THERMOPLASTIC MATRIX CARBON FIBER
REINFORCED HIGH PERFORMANCE COMPOSITES USING AUTOMATED
FIBER PLACEMENT**



M.Sc. THESIS

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Polymer Science and Technology Programme

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ

**TERMOPLASTİK BAZLI KARBON FİBER İLE GÜÇLENDİRİLMİŞ VE
OTOMATİK FİBER YERLEŐTİRME İLE SERİLMİŐ KOMPOZİT
MALZEMELERİN MALZEME ÖZELLİKLERİNİN İNCELENMESİ**

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To my dearest mother,



FOREWORD

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ABBREVIATIONS

AFP	: Automated Fiber Placement
CFR	: Carbon Fiber Reinforced
DMA	: Dynamic Mechanical Analysis
DSC	: Differential Scanning Calorimeter
HM	: High Melt
LM	: Low Melt
NDI	: Non-Destructive Inspection
PMCs	: Polymer Matrix Composites
Prepreg	: Preimpregnated



SYMBOLS

ΔH_{cc}	: Heat of cold crystallinity
ΔH_f	: Enthalpy of fusion
$\Delta H_{100\%}$: Crystalline melt energy for 100% crystalline matrix
$C\%$: Crystallinity percentage
T_d	: Decomposition Temperature
T_m	: Melting temperature
T_g	: Glass Transition Temperature



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MATERIAL PROPERTIES OF THERMOPLASTIC MATRIX CARBON FIBER REINFORCED HIGH PERFORMANCE COMPOSITES MANUFACTURING USING AUTOMATED FIBER PLACEMENT

SUMMARY

Technological developments lead a way through change on material performance requirements. Material science has been one of the oldest areas of interest of humankind since ancient history, even historical milestones has been identified according to the materials used during the time interval. Throughout the years, material science has been evolved and improved such that, nowadays people are able to tailor the material to get desired property of final product. Material engineers deals with finding solutions to make desired final property products [28,31].

Aerospace is one of the engineering fields that requires strong, lightweight, resistant and flexible materials. This is a must since all aircrafts must be lightweight relatively to the other transportation machines. To answer high performance requirements studies on composite materials have significantly increased during the last decades. Composite materials consist of two or more distinguished material having individual properties, to be able to create a composite material an interface between individual components must take place. Composite materials should have improved final properties than the individual components. Composite materials can be divided into sub classes according to the reinforcement and the matrix phase. Matrix phase holds the reinforcement together and protects the reinforcements from external damages, which can be physical, mechanical or environmental conditions. Matrix materials can be polymer, metal, ceramic, reinforcement on the other hand, can be fibers, structures or particles.

One of the most commonly used materials for the engineering applications is carbon fiber reinforced polymer matrix composites on critical parts of design for. For the matrix phase, thermoplastic or thermoset matrix are suitable options for such applications. Thermoplastic matrix has several advantages over thermoset matrix for being recyclable, lighter, durable and easier to storage with respect to thermoset matrix.

In this study, unidirectional carbon fiber reinforced thermoplastic matrix composite panels consisting of 11 plies are laid down with automated fiber placement (AFP) robot with $[[0^\circ/90^\circ]_2 0^\circ]/90^\circ$ s orientation to address a typical design approach. AFP is an additive manufacturing method of producing high quality, near absent flaw final product. It requires less time to manufacture parts with AFP than conventional methods. Impregnation means to wet fibers with matrix, prepreg is shortened name for pre-impregnated. To be able to manufacture a composite part, prepregs are laid down on to each other. Conventional way of manufacturing a composite material is hand lay-up of prepregs. If the reinforcement phase is a continuous fiber, then the stacking of prepregs can be named after orientation of fiber.

There are two different options of matrix used for this study, one having relatively higher melting temperature than the other. Different matrix material is used to address, whether melting temperature change causes any difference in thermo-mechanical properties or not. In this study 8 panels are manufactured, they are laid down with different lay-up speeds with average of 100 mm/sec and 400 mm/sec. Change of lay-up speed gives an idea of how fast the production rate can be, in order not to lose thermo-mechanical strength and crystallinity of the material.

For the thermoplastic materials, consolidation takes place under certain circumstances. Consolidation is a process of solidification of the polymer. In this study, half of the identical panels are post consolidated in the autoclave. By doing so, the effect of the post consolidation is investigated by post consolidating one of two set of the identical panels in autoclave, others left as in situ. The objective is to investigate the trend of approaching post consolidated performance level without any further need of post consolidation by changing the process parameters.

In order to be able to evaluate the outcomes of parameter, material and process change, differential scanning calorimetry (DSC), microscope, gas pycnometer and dynamic mechanical analysis (DMA) tests are conducted. Various results are investigated with respect to crystallinity, defect formation, void content, and mechanical performance of the panels.

Results has shown that there is no significant effect of lay-up speed and melting temperature of the matrix on crystallinity, whereas post consolidation has a strong influence on both degree of crystallinity and thermo-mechanical properties. Post consolidated panels have 25000 MPa of storage modulus while in situ panels have 15000 MPa. The results are also elaborated with the void content which is relatively decreases with the post consolidation treatment.

TERMOPLASTİK BAZLI KARBON FİBER İLE GÜÇLENDİRİLMİŞ VE OTOMATİK FİBER YERLEŞTİRME İLE SERİLMİŞ KOMPOZİT MALZEMELERİN MALZEME ÖZELLİKLERİNİN İNCELENMESİ

ÖZET

Teknolojik gelişmeler ile birlikte malzemedeki beklenen performans isteklerinin de değışmesi ve geliştirilmesi bir ihtiyaç olmuştur. Malzeme bilimi insanlık tarihinde her zaman yeri olan en eski bilim dallarından biridir, tarihsel dönemlerin izahı ve tanımlanması ilgili dönemde ağırlıklı olarak kullanılan malzemedeki ilham almıştır. Yıllar içinde malzeme bilimi gelişerek değışmiştir, bugünlerde, bilim insanları malzemeleri son üründe istenen performans özelliklerine göre tasarlayıp üretebilmektedirler. Malzeme mühendisleri bu noktada, istenilen özelliklere sahip son ürünün imal edilebilmesi için karşılaşılan sorunlara çözüm aramaktadırlar.

Mühendislik alanları arasında havacılık, yüksek performans isteri bakımından önde gelen alanlardan biridir, bu istekler, dayanım, hafiflik, esneklik ve mukavemet olarak özetlenebilir. Yüksek mühendislik isteklerine cevap verebilmek için, kompozit malzemeler üzerine yapılan çalışmalar son yıllar içerisinde kayda değer biçimde artmıştır. Kompozit malzemeler, birbirinden farklı bireysel özelliklere sahip, bir ya da daha fazla malzemenin arayüz oluşturmasıyla oluşur. Kompozit malzemeler her bir katılımcı malzemedeki daha gelişmiş ve farklı özelliklere sahiptir. Kompozit malzemeler matris ve güçlendirici fazlarına dayandırılarak sınıflandırılabilir. Matris fazı, güçlendirici fazı bir arada tutar ve dışarıdan gelecek darbe ve etkilere karşı korur. Matris fazı polimer, metal ya da seramik olabilir, güçlendirici faz ise fiber, yapısal ya da parçacık şeklinde olabilir.

Karbon fiber destekli polimer malzemeler kritik parçaların mühendislik uygulamalarında en çok tercih edilen tasarım malzemelerinden biridir. Bu gibi uygulamalarda matris malzemesi için termoplastik ya da termoset malzemeler kullanılabilir. Termoplastik malzemeler yeniden kullanılabilirlik, kolay tamir, hafiflik, dayanıklılık ve saklama koşulları bakımından termoset malzemelere oranla bazı avantajlar sunmaktadır. Bununla birlikte termoplastik malzemelerin proses sıcaklıkları termoset malzemelere göre daha yüksek olduğundan proses parametrelerinin bu yönde belirlenmesi gerekmektedir. Termoset polimer matris kompozit malzemeler kimyasal yapıları gereği kürleşme reaksiyonu geçirirler. Kürleşme otoklav adı verilen basınç ve sıcaklığı kontrol edilebilen endüstriyel makinelerin içerisinde gerçekleşir. Kompozit malzemenin otoklav içerisinde kürleşmesi saatler süren bir işlemdir ve öncesinde ince işçilikli hazırlıklar gereklidir. Bu hazırlıklar kısaca şu şekilde özetlenebilir; termoset bazlı kompozit malzemeler odsa koşullarında muhafaza edilemediğinden dondurucularda saklanır. Malzemenin kullanılabilir hale gelebilmesi için öncelikle dondurucudan çıkarılarak oda koşullarında bekletilir. Oda koşullarına gelen malzeme, otoklava girmeye uygun tepsi/takımlar üzerine serilir, vakum torba içerisine yerleştirilir, kaçak olmaması için mühürlenir. Otoklavda herhangi bir sebepten dolayı vakumda kaçak yalanması durumunda parçalar kullanılamaz hale

gelebilir. Termoplastik malzemeleri kimyasal yapıları gereği k rlenme reaksiyonuna ihtiya duymazlar. Termoplastik malzemelerin karmařık yapıda i ie gemiř polymer zincirlerinden oluřur. Bu zincirler lineer yapıdır ve uygun sıcaklık ve basın altında birbirleri  zerinde hareket ederler. Sonu olarak termoplastik malzemeler kimyasal deęil fiziksel bir deformasyona uęradıklarından termoset malzemelerde olduęu kadar otoklavda k rlenmesine gerek olmaz. Termoplastik malzemeler konsolidasyona uęrar. Konsolidasyon polymer zincirlerinin ilk teması ile aray z oluřması ve akabinde zincirlerin kaynařması olarak tanımlanabilir.

Kompozit malzemeler geleneksel y ntemler ile prepreglerin elle serilip otoklavda k rlenmesi ile imal edilir. Teknolojik geliřmeler ile geleneksek y ntemler olabildięince yerini otomasyona dayalı, hatayı minimize eden ve evre dostu y ntemlere bırakmaya bařlamıřtır. Termoplastik malzemeler geri d n řt r lebilir oluřları, kolay tamir edilebilirlięi ve kısa s ren konsolidasyon ihtiyaları bakımından termosetlere kıyasla daha verimli bir alternatif olma durumundadır. Bununla birlikte malzemelerin elde serim yerine, otomatik robotlar yardımı ile serilmesi hata oranını minimize etmekte ve verimi arttırmaktadır. Fiber fazı reine ile ıslatılır, aray z oluřturulur, sonrasında bu plakalar katmanlar halinde  st  ste serilir. Kompozit malzemeler geleneksel y ntem ile elle serilir, otomatik fiber yerleřtirme metodu uzun ve zahmetli olan bu y ntemin yerini almak  zere geliřtirilmiřtir.

Otomatik fiber yerleřtirme bir eklemeli imalat metodudur, bu y ntem sayesinde y ksek kalitede ve neredeyse kusursuz  r nler imal edilebilmektedir. Robota beslenen slit řeklindeki prepreg malzemeler katman katman uygun takım  zerine serilir. Robotun komutları bilgisayar destekli tasarım yazılımları aracılıęı ile yazılır. Robot alıřtırılmadan  nce malzeme beslemesi yapılmadan serim sim lasyonu yapılabilir. Robotun malzemeyi takıma seren ucunda metal, silikondan gibi malzemelerden oluřabilen bir silindir bulunur. Malzemenin konsolide olması iin gerekli basın bu silindir sayesinde uygulanır. Sıcaklık iinse farklı enerji eřitleri kullanılabilir. Bunlardan en yaygın ve etkili olan enerji kaynaęı verimlięi y ksek olması bakımından lazer destekli olanlarıdır.

Otomatik fiber yerleřtirme verimli olmasının yanı sıra hata oranını d ř rmesi bakımından da tercih edilme sebebidir. Otomatik fiber yerleřtirme neredeyse kusursuz para  retilme potansiyeline sahiptir. Bununla beraber, her imalat y nteminde olduęu gibi parada istenmeyen hatalar ile karřılařılabilir, bunlardan bazıları bořluklu yapılar, slitlerin katlanması, kat kaybı, ya da y zeydeki dalgalanmalar olarak sıralanabilir. Hataların yakalanması iin de yine otomasyon ile eřitli y ntemler geliřtirilmektedir.

Bu alıřmada, tek y nl  karbon fiber takviyeli termoplastik matris kompozit malzemeler toplamda 11 katman olacak řekilde lazer destekli otomatik fiber yerleřtirme robotunda tipik bir tasarımı adreslemek adına $[[0^\circ/90^\circ]_2 0^\circ]/90^\circ$ s y n nde serilmiřtir. alıřma kapsamında 8 adet panel  retilerek; farklı polymer matris malzeme kullanımının, robotun serim hızı deęiřiklięinin ve serime ilave olarak otoklavda ileri konsolidasyonun malzemenin son  zellikleri  zerindeki etkileri incelenmiřtir. Paneller 100 mm/sn ve 400 mm/sn ortalama hız ile serilmiřtir. Serim hızı deęiřiklięi ile, termomekanik  zellikler ve kristallenme y zdesini olumsuz etkilemeden ideal  retim hızına dair fikir vermesi amalanmıřtır.

Termoplastik malzemelerin konsolidasyonu sıcaklık, basın gibi belli fakt rlerin bir araya gelmesi ile oluřur. Konsolidasyon polymerin katılařarak son formunu alması s recine verilen addır. Bu alıřmada,  retilen panellerin yarısı otomatik fiber serme robotunda serildikten sonra otoklavda konsolide edilmiřtir, dięerleri serim sırasında

yerinde konsolide olmak üzere ilave prosese tabi tutulmamıştır. Proses parametreleri ve matris malzeme değişikliği ile ilave konsolidasyona gerek olmadan yerinde konsolidasyona hangi şartlar altında yaklaşıldığı ortaya konmaya çalışılmıştır.

Otomatik fiber yerleştirme robotu parametreleri, matris malzeme ve proses değişikliklerinin son ürüne etkilerinin incelenmesi için diferansiyel taramalı kalorimetre, mikroskop, piknometre ve dinamik mekanik analiz testleri icra edilmiştir. Sonuçlar, kristallenme miktarı, paneller içerisindeki boşluk araştırması ve mekanik performans yönünden incelenmiştir.

Sonuç olarak, serim hızının ve matris malzemenin erime sıcaklığının malzemenin kristallenme yüzdesinde ciddi bir etkiye sahip olmadığını anlaşılmıştır. Bununla beraber, otoklav konsolidasyonunun son ürünün termo-mekanik ve fiziksel özellikleri üzerinde ciddi bir etkiye sahip olduğunu ortaya koymuştur. Otoklavda konsolide olan paneller 25000 MPa modül değerine sahipken bu değer otoklavda konsolide olmayan panellerde 15000 MPa civarında kalmıştır. Sonuçlar aynı zamanda malzeme içeriğindeki boşluk miktarı ve yoğunluk ölçümleri ile kıyaslanmıştır. Malzeme içeriğinde bulunan boşluk miktarı ile termo mekanik özellikleri doğrudan ilişkilidir, termo-mekanik özellikleri yüksek olan panellerin, boşluk oranının düşük olduğu gözlemlenmiştir. Bu noktada, otoklav konsolidasyonunun malzemenin kristallenme yüzdesini ciddi miktarda arttırdığı ve otoklavda uygulanan basınç ile birlikte malzeme içeriğindeki boşluk miktarını azalttığı görülmüştür. Kristallenme derecesi yüksek ve boşluk miktarı düşük olan paneller termo-mekanik testlerde daha başarılı sonuçlar vermiştir.



1. INTRODUCTION

Composite materials can be made up of two or more different material having distinctive characteristics, which can be related with physical and chemical properties. To be able to define a material as composite there should be an interface created between the components. Composite materials are tailored to build desired final property needed which creates higher performance materials than individual components [7, 44].

Composites materials can be classified according to two phases which are matrix and reinforcement phase. Matrix phase can also be called as matrix and reinforcement can be called as fibers in this content. Fiber provides strength and stiffness. Matrix protects the fiber, passes load in between fibers and plays role like an adhesive to hold the fibers organized and protects the fibers from both mechanical and environmental damage.

Polymeric matrix composites (PMCs) may be divided into three groups according to their thermal and structural characteristics: thermosetting, thermoplastic, and elastomeric materials. Thermosetting matrix goes under curing mechanism which is defined as creation of crosslinks between polymer chains by the help of an energy source. On the other hand, thermoplastic matrix may be reshaped while it is heated to a semi-fluid state and then becomes rigid when cooled [4, 40]. Thermoplastics polymers offer several mechanical characteristics like toughness, resistance and the ability to recycle. An example for a thermoset matrix with high performance properties is epoxy, for thermoplastic matrix polyether ketone family would be a suitable alternative. Glass fibers, ceramic fibers, polymeric fibers, or carbon fibers can all be used to strengthen PMCs. Since it has distinctive performance characteristics, carbon fiber reinforced PMCs are mostly employed in high performance engineering applications [6, 8, 25].

Fiber laminate is used to create composite materials. Laminate refers to a stack of lamina with different fiber orientations. Fibers are impregnated (wetting with the matrix) in matrix to make preregs. According to the nature of the matrix composite material undergoes either consolidation or curing process. Thermoplastics polymers

are not cured as in the case of thermosetting polymers. Therefore, prepregs made with a thermoplastic matrix are quite stiff or boardy. Thermosets get transition of the liquid to solid state [9, 10].

Composite parts can be manufactured by hand lay-up of prepregs which starts with prepreg removal and follows; release agent application to the mold, cutting of plies, vacuum application, coverage of laminates, lay-up of plies, cure cycle in autoclave (for thermosets) and demold. Autoclave can be considered as an oven in which both temperature and pressure is controlled. In an autoclave chemical and physical reactions takes place at the same time. As can be concluded both hand lay-up and post processing in the autoclave is relatively long processing in comparison to automated fiber placement (AFP). AFP gives the ability to manufacture high quality parts that is why it is one of the most advanced methods of manufacturing. Basically, AFP works with a robotic arm which lays down the prepreg material layer by layer. Since no curing reaction is needed for thermoplastic matrix composites by adjusting the process parameters of AFP aerospace grade parts can be manufactured with in situ or near in situ consolidation (vacuum bag only, oven) [10, 19, 22, 39].

1.1 Purpose of Thesis

In this graduate study, comparison of two different thermoplastic matrix is made (the only difference is high melt and low melt properties), carbon fiber reinforced (CFR) PMCs parts are manufactured with automated fiber placement. Parts are laid down with different speeds and some of the parts are post consolidated after automated fiber placement some are left as in situ.

It is aimed to evaluate the changing of process parameters of AFP, polymer matrix material and post consolidation on the final property of the manufactured part. One set of each identical parts are post consolidated with autoclave to compare their thermo-mechanical and physical performance. Matrix material characteristics and effect of lay up speed in the final property is also examined. Samples from the manufactured panels are taken to make differential scanning calorimeter, dynamic mechanical analysis and microscope analysis. Results are investigated in terms of crystallinity and thermo mechanical properties, void content.

2. LITERATURE REVIEW

2.1 Polymeric Composite Materials

One of the most common used structural materials for engineering application is polymeric matrix materials (PMCs). Polymeric matrix materials have prevailing properties for being cost effective that is why they are not only used for research and development activities. Polymeric matrix composites comply great with high performance fibers. High performance fibers can be made of carbon, boron or aramid. Composite materials can be divided into sub classes according to the matrix material, polymeric matrix material is a class of composite material in which polymer matrix can be in class of thermoplastic or thermoset. Thermoplastic matrix go under curing mechanism in which polymer solidifies with the application of heat and pressure. Curing can also be called as crosslinking which occurs within the polymer chains. Curing requires huge amount of thermal energy which brings the problem of operational cost and residual stress inside the manufactured part. In addition, long cure cycles may cause defects within the part such as delamination, waviness, warpage.

Curing of thermosetting materials occurs generally in autoclaves. Autoclave is a closed cylindrical shaped vessel in which temperature and pressure is controlled. Figure 2.1 shows a representation of autoclave treatment [42].

Panels having high performance final physical and mechanical properties can be manufactured in an autoclave because heat and pressure application can be properly adjusted and controlled with this process. Application of heat and temperature helps bonding of prepreg plies to each other, cures the polymeric material and eliminates trapped air. For the thermosetting matrix composites chemical reaction takes place in the autoclave while in case of thermoplastic autoclave is used for better consolidation between the interlaminar bonds of plies. Capacity of an autoclave is variable they can be designed smaller for research and development activities as well as mass production for high technology application. In those cases dimensions of the autoclave can be enormous which brings relatively higher investment cost.



Figure 2.1 : An illustration for the vacuum bag in an autoclave.

Curing reaction, matrix flow and the transfer of heat energy occurs in an autoclave. Figure 2.2 (a) and (b) illustrates a typical bagging of composite material prior to autoclave treatment. parts are bagged to separate the gas that is used for pressure application from the composite part, vacuum application is also possible thanks to bagging. Figure 2.2 (c) shows an example of lay-up of prepregs with different oriented fiber prepregs. Figure 2.2 (d) gives an actual prepreg microcut analysis cured in an autoclave [7].

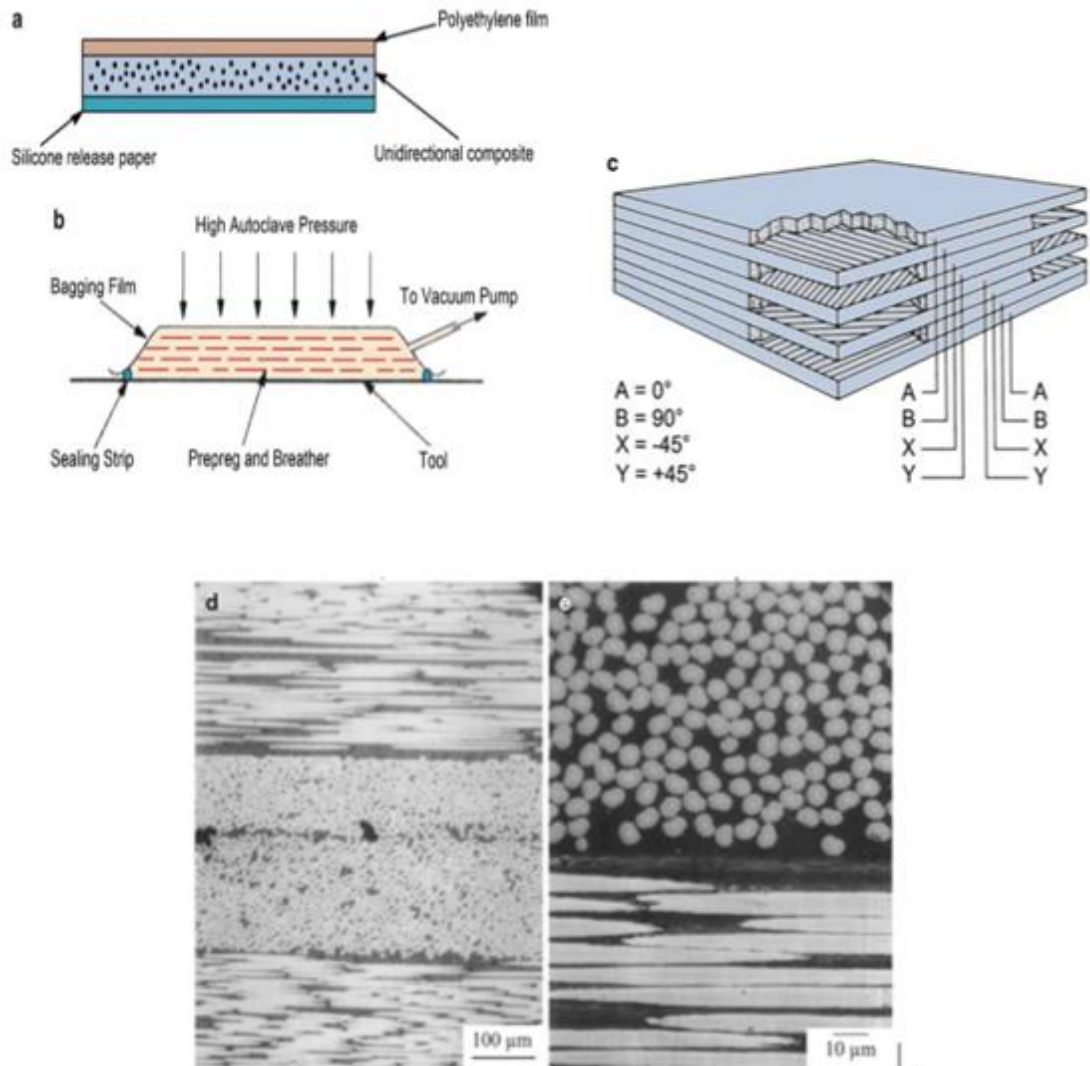


Figure 2.2 : (a) view of a prepared prepreg (b) view of autoclave treatment (c) different orientation of fibers (d, e) microcut analysis.

2.2 Thermoplastic Matrix Composites

Another class of polymeric matrix composites are thermoplastics which has several advantages with compared to thermoset matrix composites. In the most simply form the main variance of the thermoplastic and thermosetting polymers is caused by their chemical nature. As it has been explained thermosetting polymers can create crosslinks within their polymeric chains while thermoplastic polymers are not. Since thermoplastic polymers have linear chains that is why they are not cured with the application of heat and pressure but they go under physical transformation [30]. As a

result, for the curing of thermoset materials much more longer and complex curing cycles are required than thermoplastic materials. There are advantages of thermoplastic matrix composites with compared to thermosetting matrix materials include:

- Special storage requirement (freezer etc.) is not needed.
- Manufacturing is more flexible and efficient in terms of time and cost.
- Thermoplastics have unlimited shelf life, which is environmentally friendly.
- Chemical resistance is high.
- In order to joint different parts heat application can be enough.
- Parts can be reshaped, repaired, recycled.
- Their mechanical properties (toughness, impact resistance etc.) are better than thermosetting materials.

In Table 2.1 properties of thermosetting and thermoplastic materials are given. As it can be concluded from the table, all the mechanical properties show better performance in thermoplastics than thermosetting [7, 15]. Besides from the advantages listed above, thermoplastic materials also have some drawbacks with compared to thermosetting material. They can be listed as follow:

- The processing temperature of thermoplastics is higher than thermoplastics.
- The structure of thermoplastics is stiffer (lack of tack property) which can be a problem during manufacturing.

Table 2.1 : Properties of thermosetting and thermoplastic polymers.

Property	Epoxy	PEEK
Tensile Strength (MPa)	35-85	92
Flextural Modulus (GPa)	15-35	40
Density (g/cm ³)	1.38	1.30
Continuous Service Temperature	25-85	260-425
Water absorption (24 h %)	0.1	0.1

To be able to call a composite part as qualified, there should be efficient amount of contact and between the layers and the tows, void should be eliminated. In general, to be able to achieve a good quality consolidation, 6-12 MPa of pressure and temperature above melting temperature (275-350 °C) is applied. Parts are left at dwell in the set temperature for about half hour which is way more less than a thermosetting material. One of the drawbacks of thermoplastic material is their higher melt viscosities which makes it difficult to impregnate than thermosetting material. Consolidation mechanism of thermoplastic materials can be further explained by creation of bonds between the interfaces. This bonding firstly occurs when two adjacent plies come into intimate contact and then bonding occurs in the interface called autohesion. In the autohesion phase, chain like molecules' segments diffuse across. Figure 2.3 shows the consolidation phenomenon of thermoplastics [20].

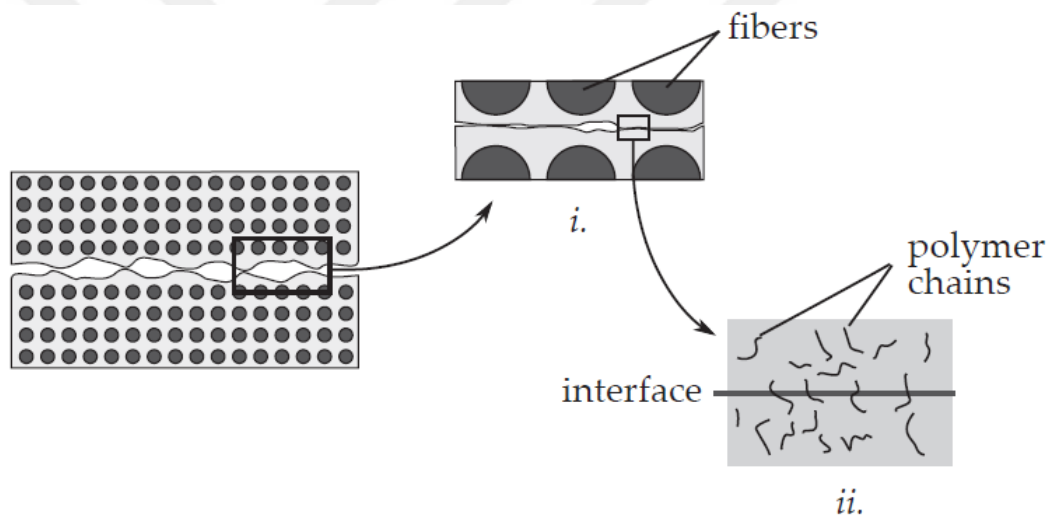


Figure 2.3 : Consolidation mechanism (i) intimate contact (ii) autohesion.

2.3 Carbon Fiber Reinforced Polymer Composites

The history of carbon fiber reinforced material usage goes back as 1950s and has been matured enough to be used as structural material in the 1980s. the main area of engineering using carbon fibers was aerospace industry and it is followed by sporting goods. Carbon fiber reinforced materials mainly used as load carrying structural parts as well as their excellent thermal and relatively high electrical conductivity made them to be used in the areas that accumulated static electrical charge. Parts that are coated with carbon fiber are used as shield against electromagnetic field. Classification of carbon fiber can be done according to their mechanical properties and orientation of

fibers. Prepregs can be made as woven or unidirectional, according to the place of part to be used in the final product, design can be tailored. Table 2.2 shows various properties of carbon fiber reinforced polymeric composite materials [4].

Table 2.2 : Properties of carbon fiber reinforced thermoplastic and thermosetting composite materials.

Prepreg Material	UD Thermoset Carbon / Epoxy	UD Thermoplastic Carbon / PEEK	Fabric Thermoset Carbon / Epoxy	Fabric Thermoplastic Carbon / Polyimide
Fiber-Volume Fraction (%)	55-65	57-63	57-63	58-62
Processing temperature (°F)	250	550	250	660-730
Tensile Modulus (Msi)	15-22	26	8-9	10-18
Tensile Strength (ksi)	180-320	410	75-124	130-155
Compressive Strength (ksi)	160-250	206	65-95	130
Maximum Service Temperature (0°F, dry)	180-250	350	-	500-600
Shelf Life (0°F dry, months)	6-12	Indefinite	6	12
Out Time at Room Temperature (days)	14-30	Indefinite	10	Indefinite

2.3.1 Prepreg hand-lay-up

Composite materials consist of stack of prepreg plies. Those plies can be laid by hand. Hand Lay-up is a labor intensive yet simple process. Figure 2.4 shows a representation of hand lay-up [4].



Figure 2.4 : Representation of prepreg hand lay-up.

Flow chart of prepreg Hand- Lay-up and bagging procedure prior to post curing/consolidation can be schematically shown as in Figure 2.5.

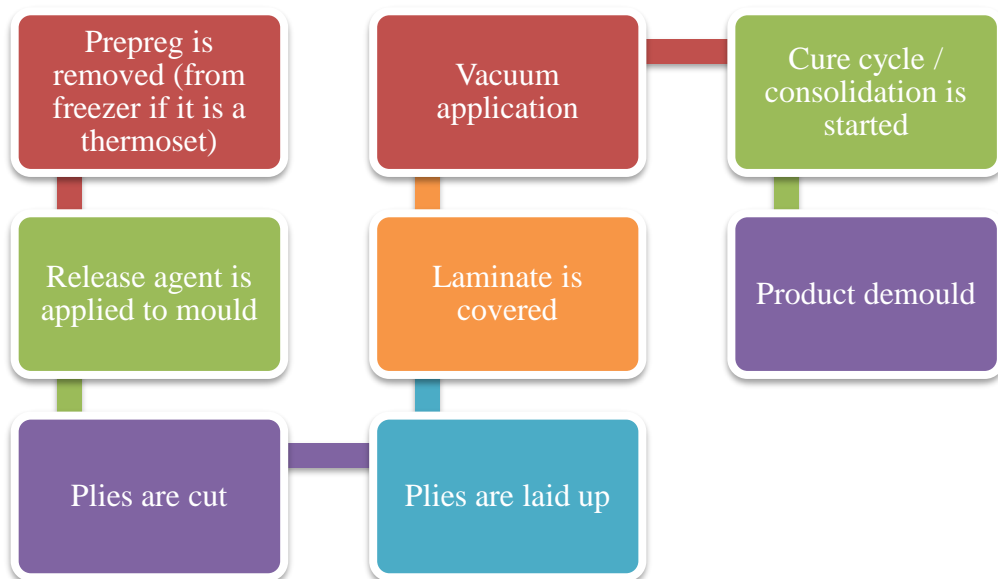


Figure 2.5 : Flow chart of prepreg hand lay-up process.

There are some advantages and disadvantages of prepreg hand lay-up process, advantages can be summarized as; flexible design allowance which results in easier manufacturing of complex items and equipment investment cost is minimum. Disadvantages of prepreg hand lay-up can be listed as, being labor intensive process, quality of manufactured part extremely dependent on person who is working and also it is not a suitable process for mass production, product uniformity can alter and finally scrap material amount is high.

2.3.2 Automated tape placement (ATL) and automated fiber placement (AFP)

Technological development has made it necessary to automate nearly any process in industry. Thanks to automation defect rates are reduced and productivity, efficiency increases [5, 14, 24]. For the composite part manufacturing, there are two important automation techniques; ATL and AFP that eliminates human error factor. Automated machines are large sophisticated robotic machines that requires high capital investment cost. Both thermosetting and thermoplastic matrix composited can be laid with ATL and AFP.

2.3.2.1 Automated tape laying

In automated tape placement technique, fibers are in the form of tape which can be laid down in a decided direction. ATL technique is commonly used for especially aerospace applications, with ATL not the single fiber tows but form of tape is placed continuously [2]. This process generally used for thermosetting materials but can also be used for thermoplastics. Placement starts after computer aided design program is prepared. Using the CAD data part is manufactured on to a mold layer by layer [12]

2.3.2.2 Automated fiber placement

Automated fiber placement is a technique that individual fiber tows are placed. Thanks to individual fiber placement, AFP allows placement of complex parts, scrap rate is also reduced because placement takes place in more detailed form. Fiber tows (preimpregnated fibers with matrix) are fed from the robotic arms spool on to the surface of the mold. In general width of a tow is 3 mm and a tow consist of fibers. An average strand consists of nearly 12000 filaments impregnated with matrix. AFP

allows changing lay-up speed according to the needed portion of part, such as curvatures. A compaction roller presses the fiber onto the mold that the placement is done on to. Thanks to compaction roller, entrapped air and minor gaps are eliminated within the part. Figure 2.6 gives an illustration of AFP machine working principle [3].

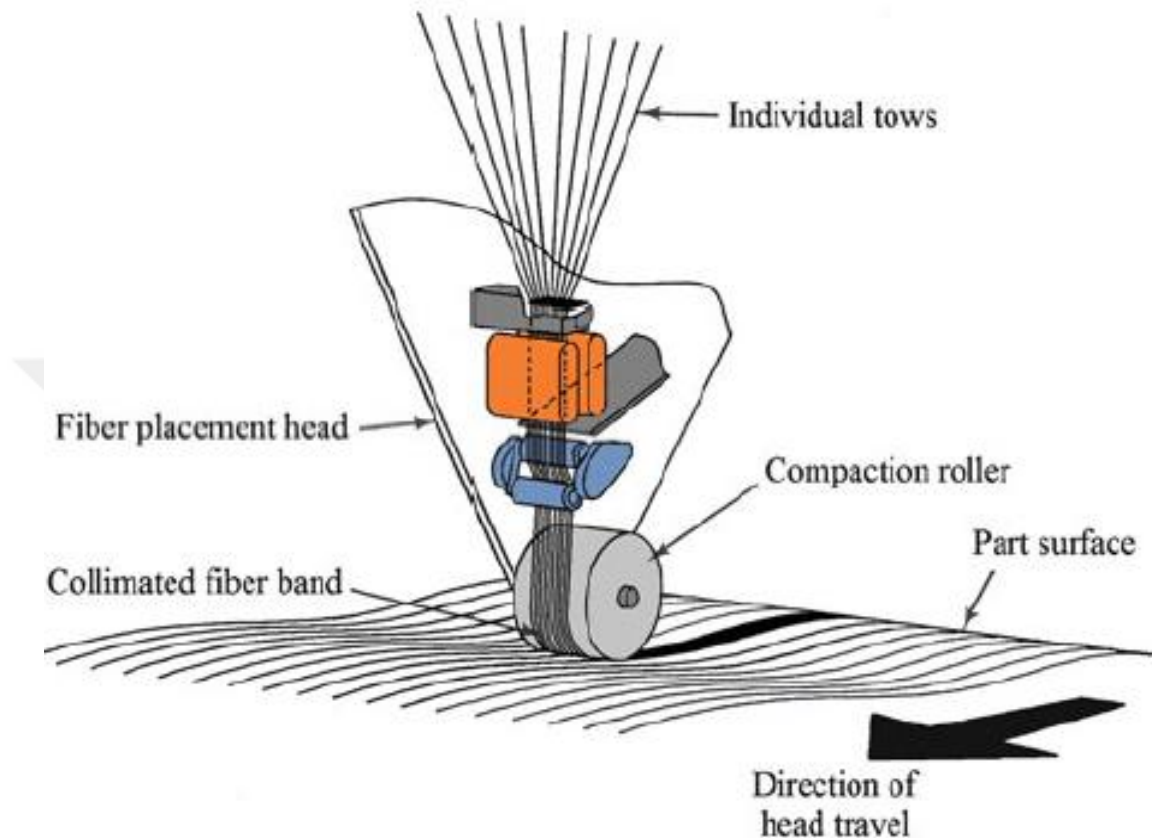


Figure 2.6 : Representative sketch of automated fiber placement.

2.3.2.3 Future of composite part manufacturing

Composite materials play an important role in engineering, however their relatively high material and manufacturing cost is the main obstacle to bring them to common usage. There are a lot of research and development activities on going to reduce the cost and improve the manufacturing process. Automated fiber placement takes the place of hand lay-up for this purpose. AFP like processes result in increased productivity and thermoplastic composites are a good option considering requirement of shorter consolidation cycle times. Figure 2.7 shows the trend of number of research on automated manufacturing methods such as AFP, ATL and Filament Winding (FW) [18]. It can clearly be concluded that during the last decades, automated processes has drawn attention of researchers significantly.

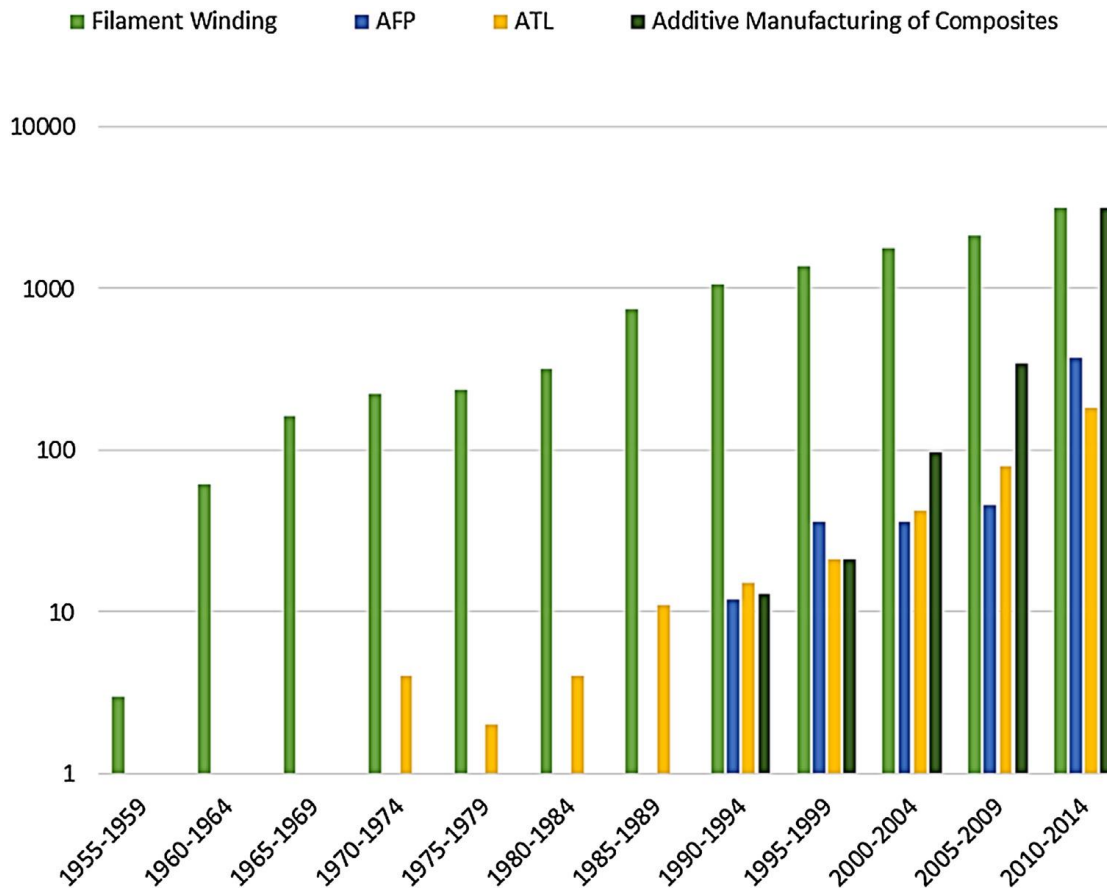


Figure 2.7 : Graph of number of published papers over the years with respect to FW, ATL and AFP.

2.4 In Situ Consolidation

Thanks to thermoplastic matrix composite materials chemical structures curing reaction is not needed. This property brings an advantage to AFP process, full in situ consolidation concept aims to consolidate the part while it is being placed onto mold. The main struggle with the in situ concept is that, final part quality is needed to meet post consolidated product performance such as (low void content, porosity, high crystallinity amount and autohesion). Moreover, for a successful in situ consolidation fed raw material property is also an important parameter because raw material quality can change from supplier to supplier. In conclusion, in situ consolidation aims to consolidate the part as it is placed onto mold.

AFP technology consists of subcomponents which are given in Figure 2.8. There is also software equipment which controls the whole system (motion of the robotic arm and process parameters like speed and force, direction of fiber) [27].

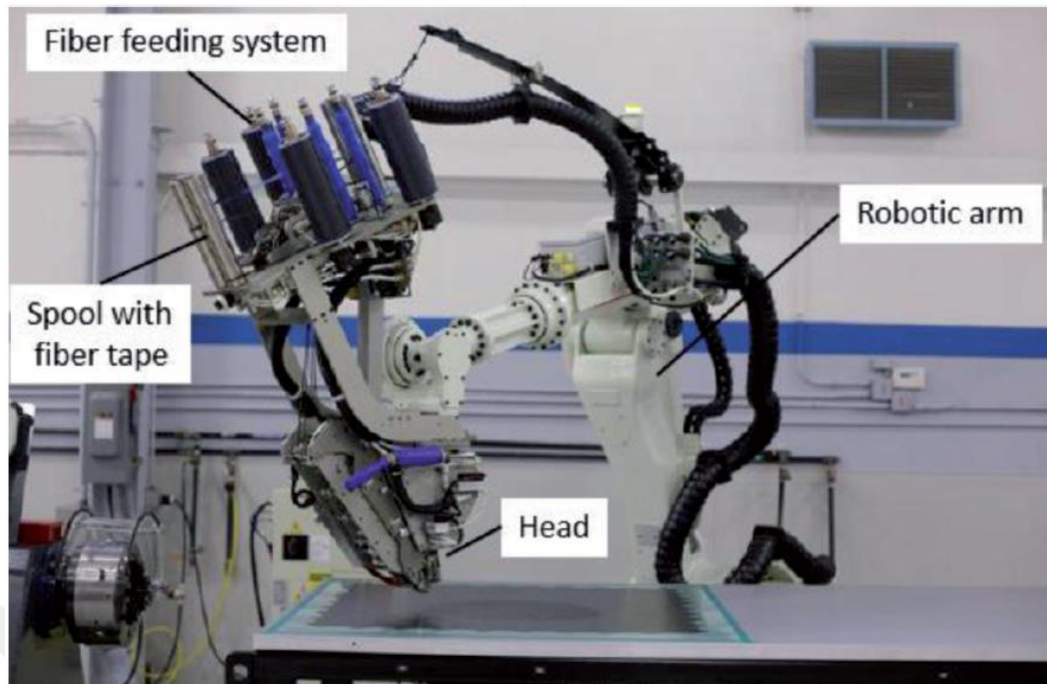


Figure 2.8 : Automated fiber placement machine.

2.5 Interlaminar Bonding of Thermoplastic Composites

In order a thermoplastic composite part to be fully consolidated there should be proper amount of close interaction, which is directly linked with the pressure and heat application. In the place of first interaction of polymer layers diffusion takes places in between the layers. Diffusion is caused by thermal energy. Thermoplastic materials are in the form of entanglement in their natural form, and they do not easily move. When there is a heat application polymer chains starts to move. Movement of chains is linked with the temperature increase.

2.6 Type of Heat Sources

The main objective of AFP is to achieve in situ consolidation. In situ consolidation can only be achieved by sufficient amount of heat, pressure application above the melting temperature of composite matrix. AFP system can use different type of heat sources to decrease the operation cost and optimize the process requirement. Some of those heat sources are; ultrasonic, direct flame, infrared heating, flash lamps and lasers [8, 32, 34]. Figure 2.9 shows the diagram of criteria like cost, feasibility and power output with respect to different heat sources. According to efficient score diode laser heat source on top of the list [16].

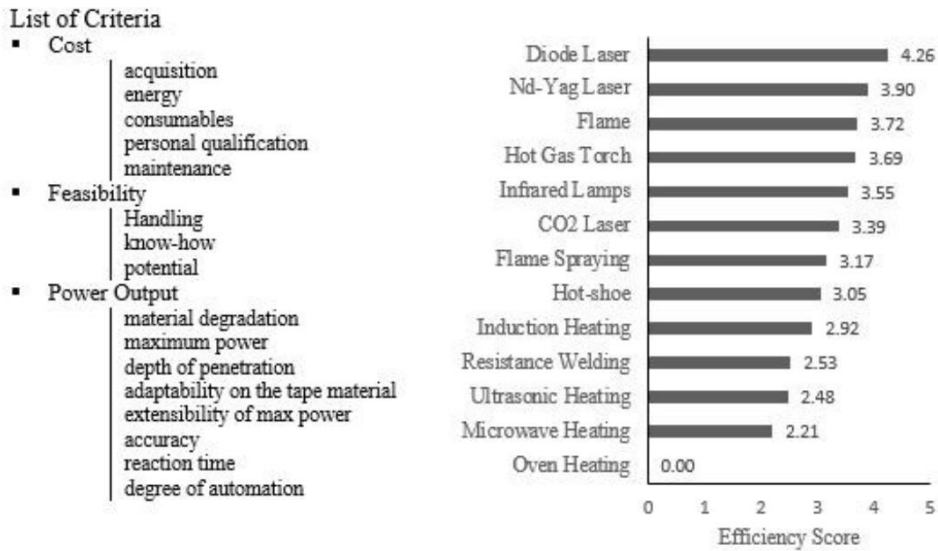


Figure 2.9 : Investigation of AFP/ATL heating sources.

Laser assisted AFP technique has higher heat intensity and it heats faster than other methods. However, it is an expensive process, physically occupies large space and special healthy and safety precautions should be taken in the use of laser assisted AFP equipment. The main issue with the heat source is about uniform application of heat [43].

Laser beams are well controlled that is why parts with uniform quality can be manufactured. Figure 2.10 shows a schematical representation of AFP system, heat is directly applied at the point where the first contact with the tool is made.

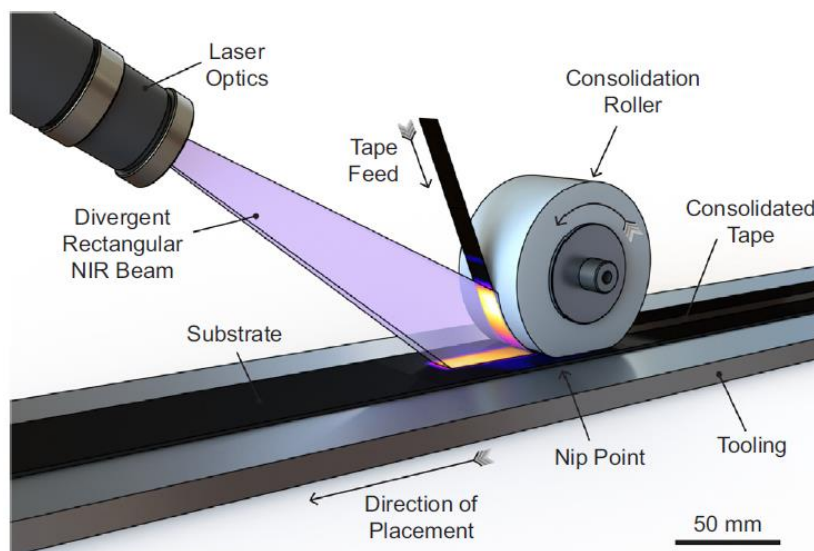


Figure 2.10 : Schematic of AFP heat source and nip point.

In conclusion laser is a heat source with the capability of high heat intensity, it also has high precision of applying heat to a specific region which in return reduces internal stress and material defects.

2.7 Compaction Roller and Speed

In AFP systems, besides from the applied heat, pressure is an important parameter. Compaction force is applied with the compaction rollers placed on tip of the robotic arm. Roller material can be steel or silicon. Silicon rubbers can be used for the parts with complex shapes because of their ability of deformability [1]. They also apply pressure with longer distribution which helps the intimate contact creation by giving more time). Time of cooling also important for polymeric matrix composite parts, if a steel roller is being used than more cooling is required for the nip point. Figure 2.11 gives an example of conformable silicon compaction roller.

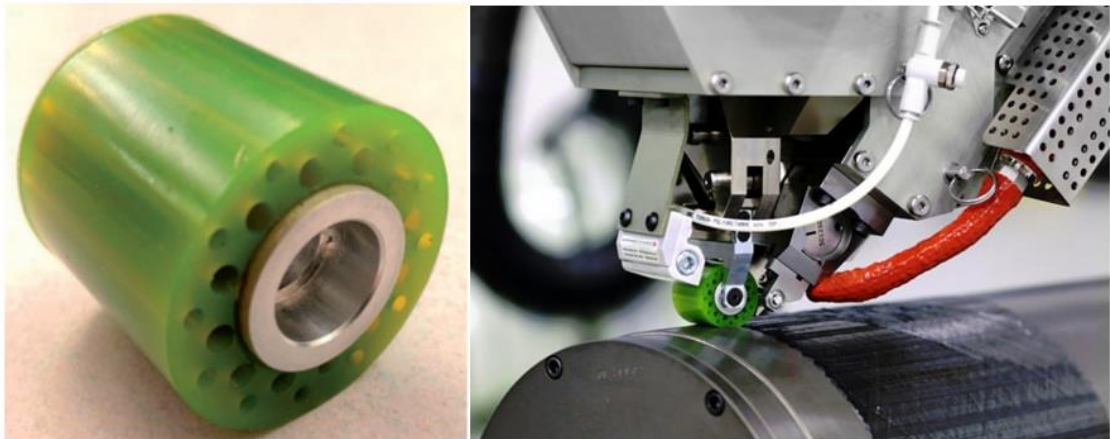


Figure 2.11 : Conformable compaction roller example.

2.8 Characteristic of Composite Materials

There are several factors that alter property of composite materials. Those properties mainly affected by the matrix viscosity. That is why impregnation is one of most critical phase of thermoplastic processing. Characteristic of tape that is fed to the AFP machine is related with internal void content, fiber volume ratio, impregnation and surface roughness. For the full in consolidation, all the parameter changes must be

taken into consideration. Figure 2.12 shows how can fiber distribution and surface roughness can alter from supplier to supplier.

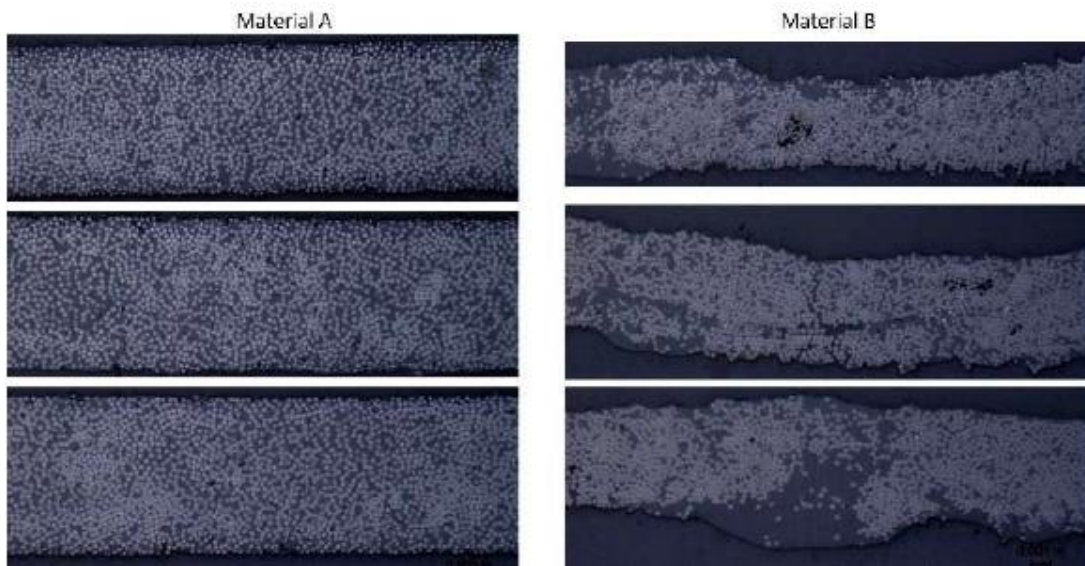


Figure 2.12 : Matrix and fiber distribution of same composite material between suppliers.

For the quality part production impregnation, consolidation and crystallinity are needed to be main concerns. Impregnation cannot be controlled during part manufacturing; however, consolidation and amount of crystallinity can be controlled.

By changing the AFP parameters, temperature, pressure and the time that those two parameters applied can be controlled. If the pressure is set, for the appropriate amount of intimate contact and bonding, high temperature or longer time is needed. Moreover, mechanical properties are highly affected by the crystallinity. If the degree of crystallinity increases then the strength and stiffness increases, but low degree of crystallinity increases fracture toughness and resistance. Crystallinity is directly linked to the thermal history of the polymer. If the cooling rate is high than there is less time given to the polymer chains to get in order (became crystalline), therefore cooling rate should be considered accordingly. It should also be kept in consideration that if the lay-up rate is low than the material will be exposed to the high energy for a longer time which may cause material degradation [37]. Another important parameter for mechanical properties is void content, internal void content can be reduced by applied compaction force amount, AFP and ATL processes have ability to control those parameters.

2.9 Defect Formation During AFP Process

AFP may result in part manufacturing with defects besides from being one of the major composite manufacturing techniques [13]. Those defects are required to be captured during placement and manually corrected for the quality of produced part. Defects are one of the biggest issues coming with automation, since they are hard to define and classify. Some of the defects are shown and identified in table 2.3 [21].

Table 2.3 : Some of defects occurred during AFP and their identification.

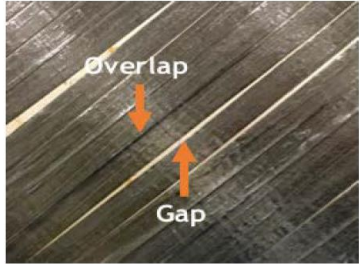
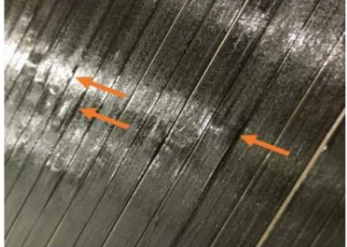

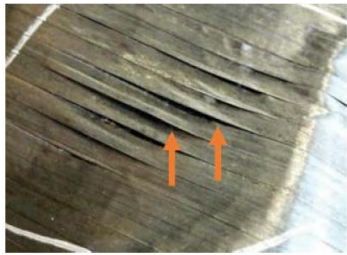



Identification / Outcome	Appearance
<p data-bbox="560 860 746 898">Gap / Overlap</p> <p data-bbox="320 936 991 1043">Gaps results in matrix rich regions and causes crack propagation. Overlaps creates rough surfaces which lowers compressive strength.</p>	
<p data-bbox="608 1211 703 1245">Pucker</p> <p data-bbox="280 1283 1038 1357">Puckers causes delamination, in some cases pucker can be flattened by upcoming layer.</p>	
<p data-bbox="600 1503 711 1536">Wrinkle</p> <p data-bbox="280 1574 1038 1648">If wrinkles are not covered with the upcoming layer, then folded tows may result in strength loss.</p>	
<p data-bbox="600 1771 711 1805">Bridging</p> <p data-bbox="280 1843 1038 1962">These defect shows that compaction force is not suitable or compaction roller is not in best contact with the part. Bridging can cause matrix rich areas or delamination.</p>	

Table 2.3 (continued) : Some of defects occurred during AFP and their identification.

Identification / Outcome	Appearance
<p data-bbox="406 629 564 660">Folded Tow</p> <p data-bbox="209 703 762 846">Folded tows may result in serious damage, because thickness increases right next to decreased thickness, those regions create low strength area that can cause failure.</p>	
<p data-bbox="443 1137 528 1169">Splice</p> <p data-bbox="213 1211 762 1317">Splices cause in thickness change but they are not considered as much significant for stiffness change.</p>	
<p data-bbox="387 1599 584 1630">Foreign Object</p> <p data-bbox="218 1673 756 1742">In order to avoid foreign object defect manufacturing must be cleaned regularly.</p>	

2.10 Inspection of Defects

AFP robotic technology has a promising potential because of giving advantage of efficient mass production, however this does not mean that process is defect free. The most common defect seen in the parts manufactured with AFP is gaps and overlaps that generally caused by width change of tows or machine tolerances. Figure 2.13 shows such defects created on the manufactured part [33].

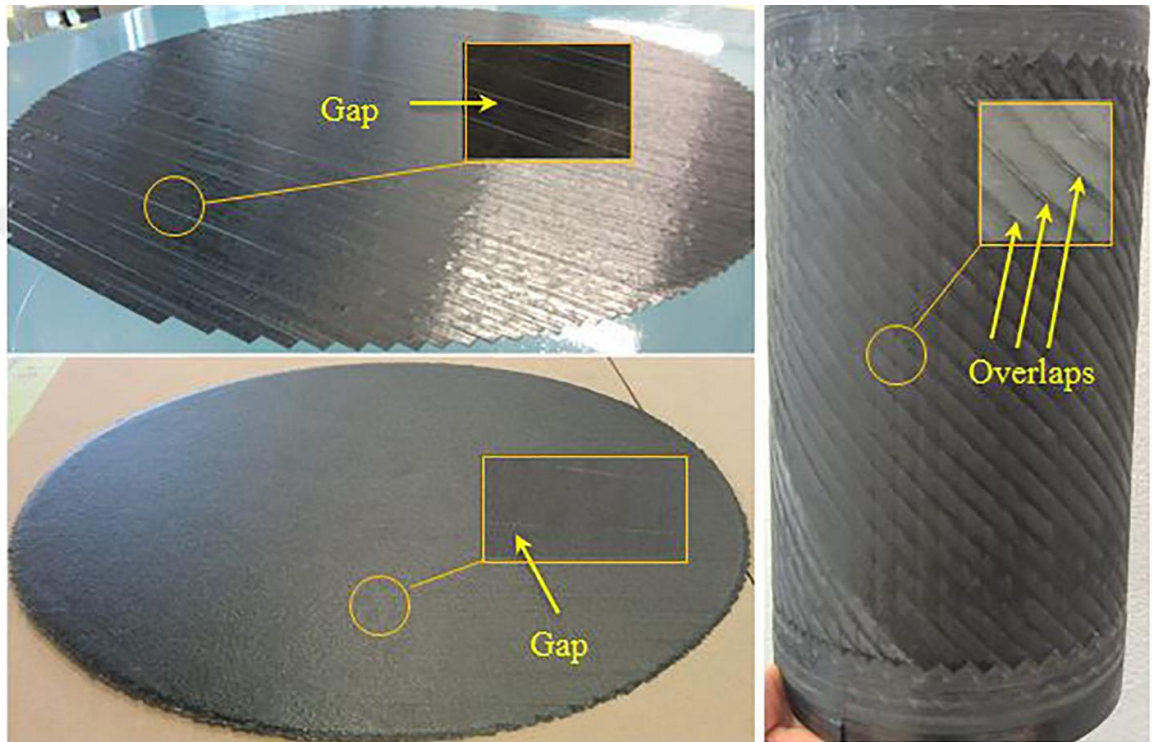


Figure 2.13 : An example of gaps and overlaps during automated fiber placement.

There are several studies carried out on investigation of defects [46] Ebrahim et al. (2017) has studied on glass fiber composite preregs, with addition of Fiber Bragg Grating (FBG) [33]. During the manufacturing some defects like, gaps and overlaps are intentionally created in the part. Lay-up process is monitored afterwards, to be able to investigate the effect of optical signal wavelength on the FBG sensor. Figure 2.14 illustrated a representative picture of FBG sensor working principle.

The results of FBG sensors have shown that there is significant wavelength change when the defect is captured.

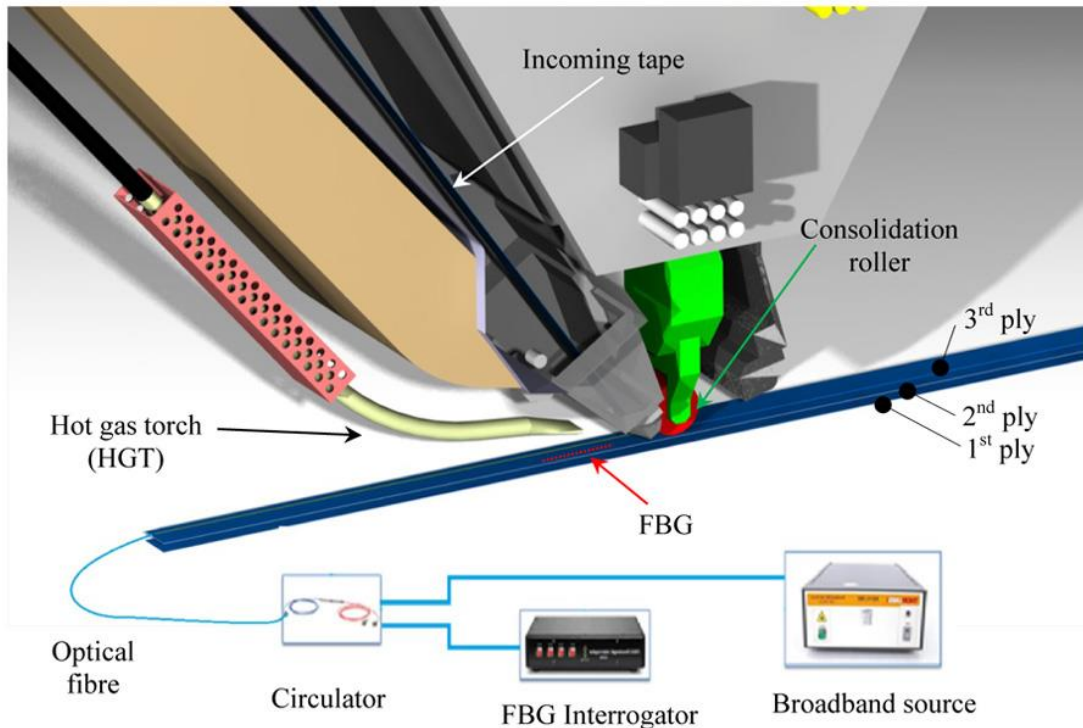


Figure 2.14 : Fiber bragg grating sensor.

Shadmehri et al. (2015) has used a laser inspection technique to detect potential defects. The system works with a laser projection and visual inspection in parallel. Figure 2.15 shows a representation of the working principle [35].

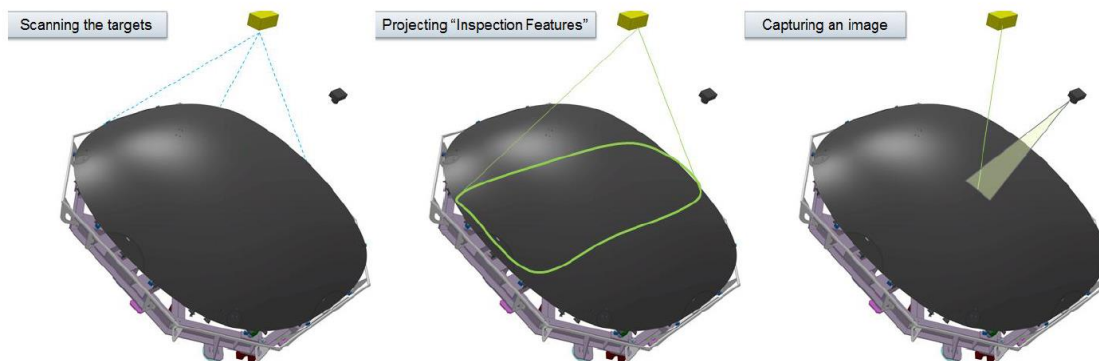


Figure 2.15 : Laser inspection system.

Thanks to laser inspection system acceptable limits for gaps and overlaps are identified. If the gaps exceed the acceptable limits than laser system can detect the defect as seen in the Figure 2.16. When the defect is inspected necessary caution is needed to be taken which can be a repair or replacing the part depending on how severe the defect is. Determination of the severity of the defect is decided with respect to controlled experiments and research activities.

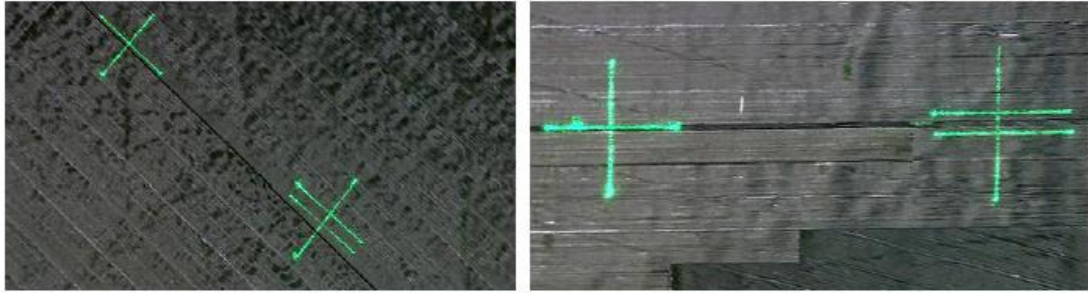


Figure 2.16 : Laser inspection for gaps between the tows.

Another defect besides from the gaps and overlaps are fiber waviness. This defect should be eliminated. Since defects remarkably reduces the mechanical properties of produced part. Krämar et al. (2020) has used a camera for capturing the waviness formation. Experimental setup for their study is given in Figure 2.17 [28].

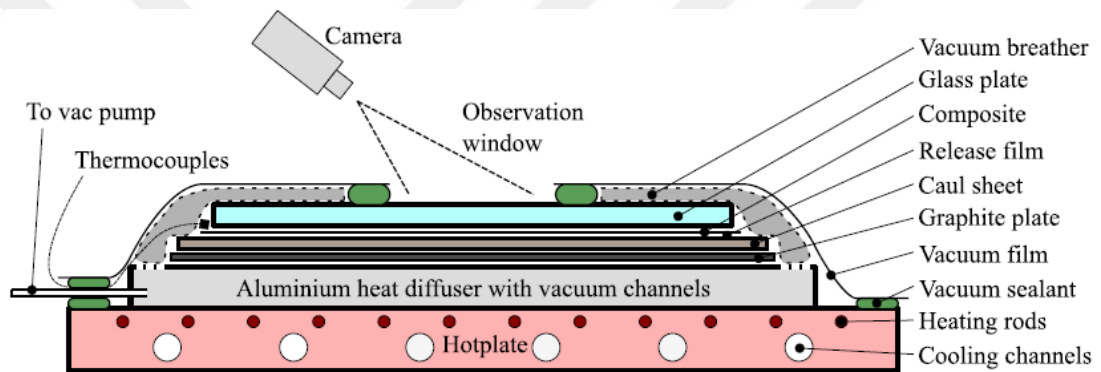


Figure 2.17 : Defect capturing mechanism with camera.

It has been observed that waviness occurs in the cooling stage, from maximum process temperature to crystallization temperature. Figure 2.18 shows formation of waviness. If the process temperature gets higher than the waviness formation increases, or the degree of the waviness.

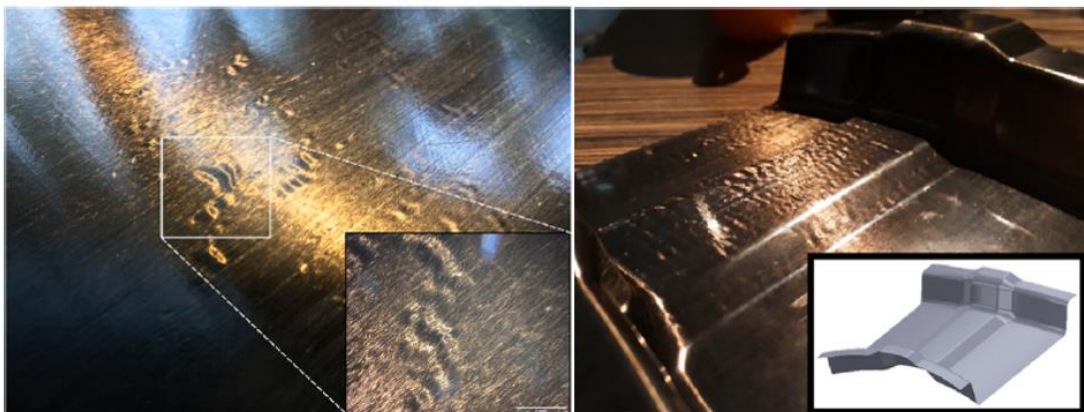


Figure 2.18 : Formation of waviness.

It has been concluded that driving force waviness formation is caused by tool shrinkage, ply and tool friction creates a wave [28].

Another way of defect monitoring is to capture difference in temperature. Chadwick and Willmeroth (2019) has studied defect detection by the help of a thermal camera, temperature values are recorded. Figure 2.19 shows a representation of the system [6].

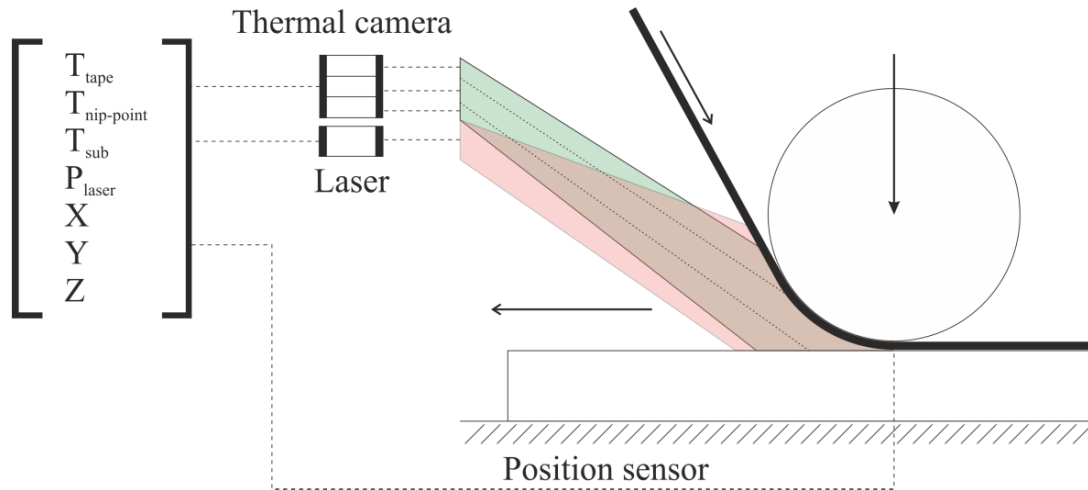


Figure 2.19 : Real time temperature monitoring during AFP manufacturing

Detected defects are identified in accordance with the temperature change. Table 2.4 shows how the defects are determined.

Table 2.4 : Common defects and recorded temperatures.

Defect	Effect on recorded temperature
Laser power overshoot	Sharp increase
Insufficient laser power	Sharp decrease
Foreign Material	Sudden decrease or increase
Missing tow material	Ambient value zero

2.11 Effect of Defects on Mechanical Properties

Defect formation and their effect on the final part manufactured can be severe. That is why there is remarkable amount of study carried out to investigate the outcomes of defects [17]. Croft et. al (2010) have studied on common defects [11]. They have tested strength property on those intentionally defected regions. Tests are conducted in both lamina and laminate level.

Test are conducted in two different configurations. One of them is on the lamina level and the other one is on laminate level. Tension, compression and IPSS tests are conducted for the first set. open hole tension (OHT) and compression tests are conducted for the laminate level. A comparison in between each specimen is conducted with respect to a part which is free from defects. Tension and in plane shear specimen microcut view is given in Figure 2.20.

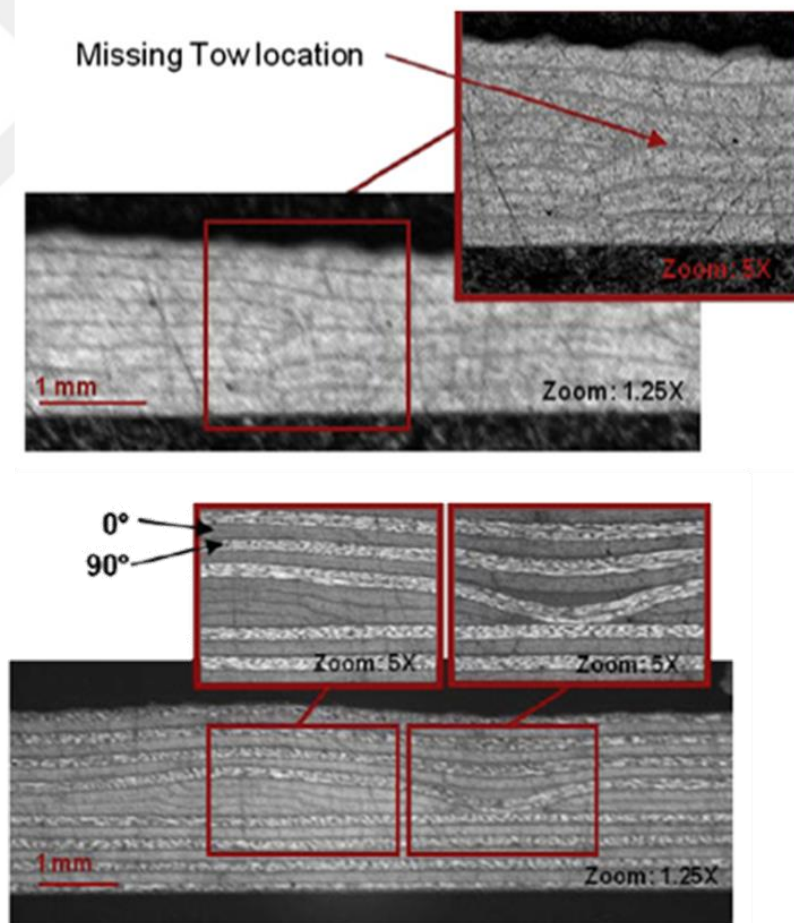


Figure 2.20 : Tension specimen with gap (left), IPSS specimen with gap and overlap (right).

Test results are given in the Figure 2.21 and tabulated in Table 2.5. Basically results are indicates that if a defect improves a mechanical property, results in a decrease or disadvantage in another property. Therefore, it is difficult to take advantage of these improvements even if there is [11].

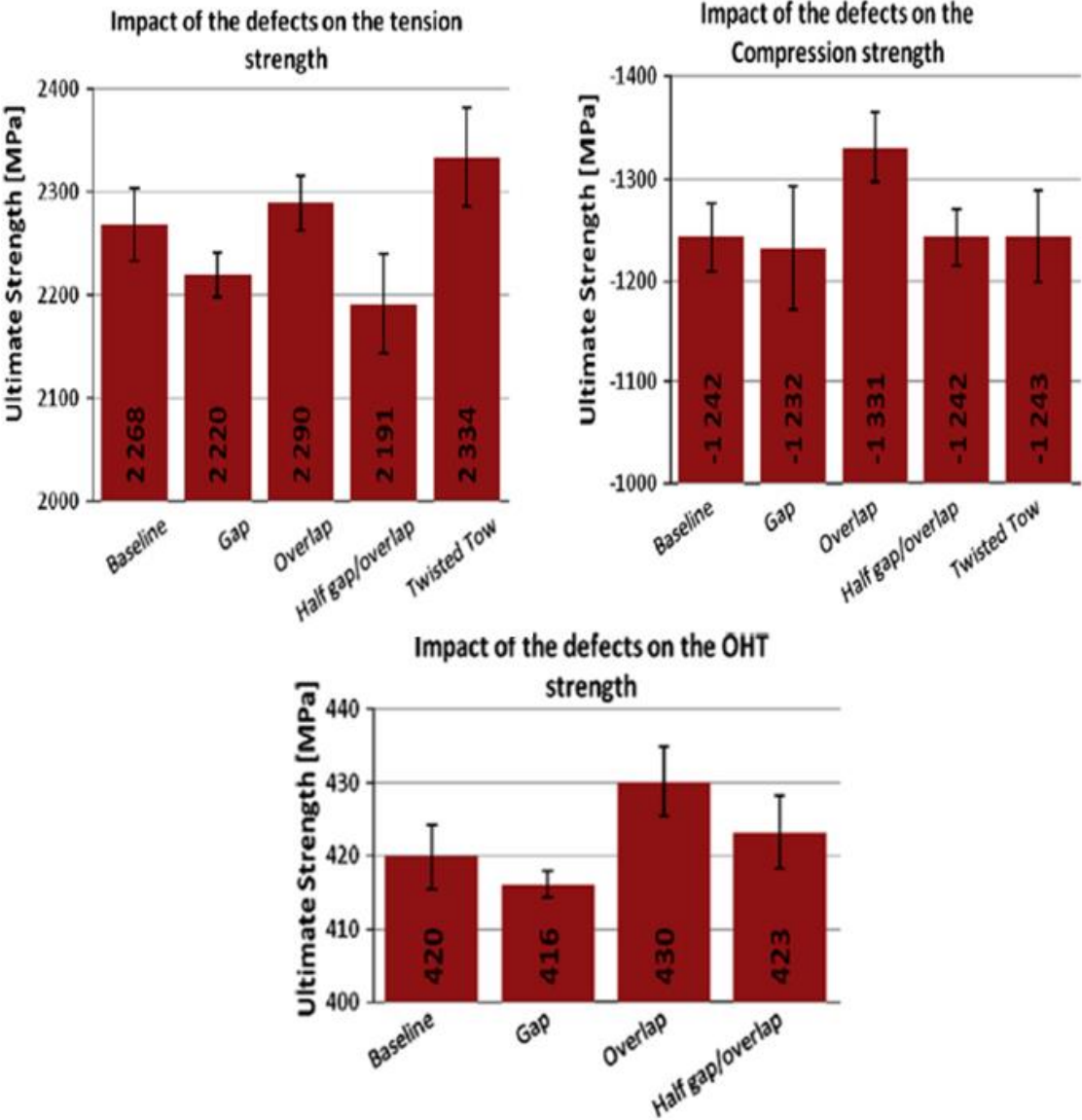


Figure 2.21 : Test results with respect to reference.

Moreover, like in the case of open hole compression test, improvement can cause delamination which is the accumulation of damage. In such cases panels can be needed to be repaired. Because when the repair is not conducted then the mechanical properties of the manufactured part drops significantly. In some cases a repair can not be the ultimate solution or may not be sufficient enough to use the part in structural design location, in such situations related part might not be used at all. This means that near flow production increases the quality and also reduces the manufacturing cost.

Table 2.5 : Comparison of performance with respect to different defects.

		Gap	Overlap	Half Gap/ Overlap	Twisted Tow
	Tension	↔	↔	↘	↗
	Compression	↔	↗	↔	↔
In-Plane Shear	Length	↔	↔	↘	↘
	Width	↗	↘	↘	↘
	OHT	↔	↔	↔	
OHC	Length	↗	↗	↗	
	Width	↘	↘	↘	

↗ $\geq 3\%$ increase up to 13%

↔ $\pm 3\%$ variation

↘ $\geq 3\%$ decrease up to 13%

In conclusion, results that are collected from this study should be enriched with further investigation to be able to show effect of cumulative and single defects on final product. This should be kept in mind that defects are an important parameter for the part's mechanical performances.

2.12 Investigation of In Situ Consolidation Performance

In addition to the mechanical performance criteria of a final part, some engineering applications such as aerospace requires a part having certain surface finish quality. In automated fiber placement applications, a good consolidation property is not the only necessity but also surface smoothness is important.

Shadmehri et al. (2018) studied on the effect of the 'repass' treatment to be able to investigate the surface finish quality [36]. Repass treatment is defined as movement of compaction roller without placing additional tows on to previously laid plies. By doing so, additional heat and force is applied to the part. Figure 2.22 shows that repass treatment decreases the roughness significantly both on perpendicular and along to the fiber direction. Repass treatment is applied for once for some panels and twice for the others, to be able to compare with the reference autoclave treated panels are used.

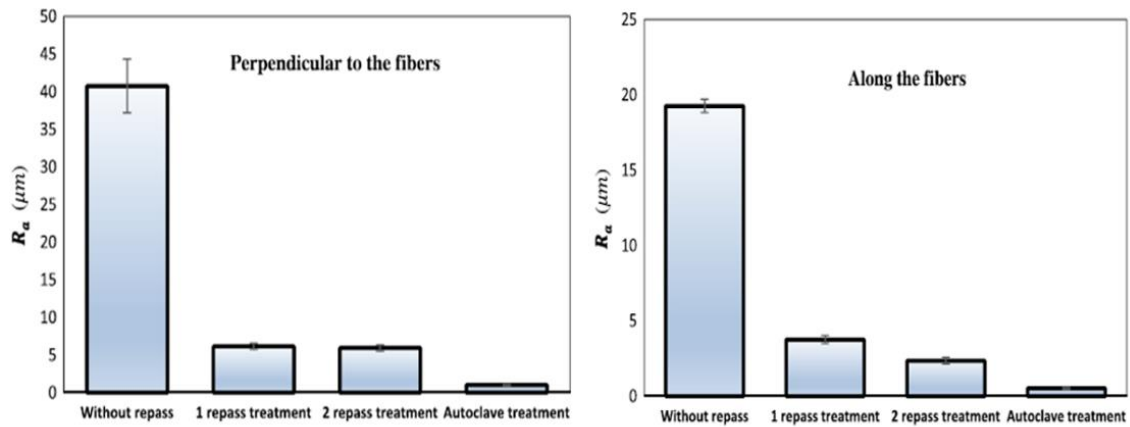


Figure 2.22 : Roughness measurement results in corresponding fiber direction.

The effect of repass and autoclave treatment also investigated with the micru-cut analysis. Microscope results are given in Figure 2.23 which indicates that void content decreases with repass treatment.

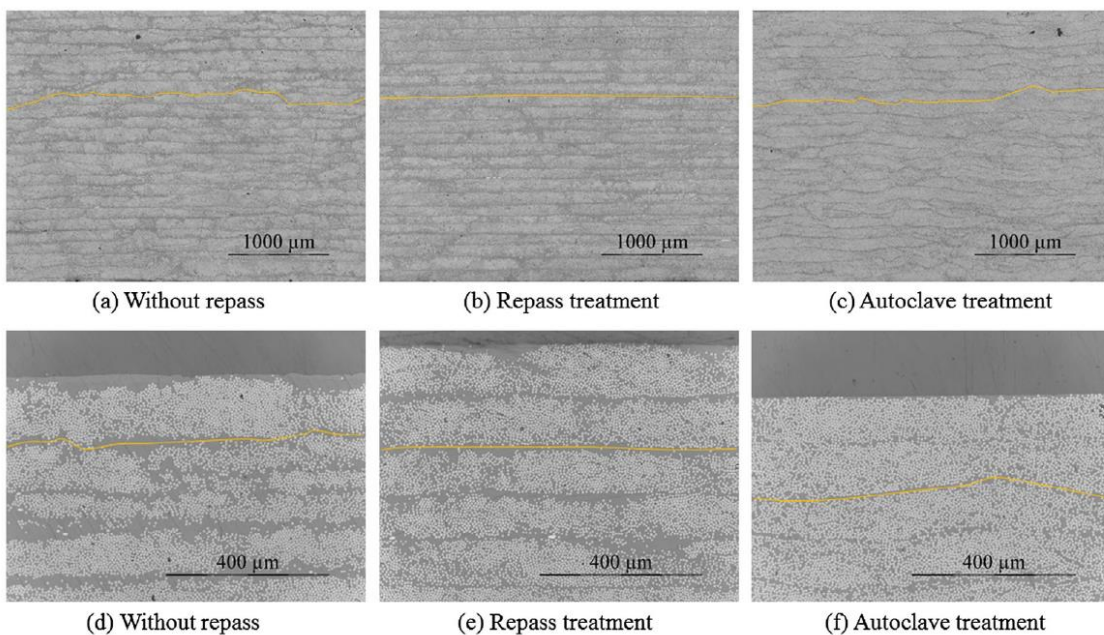


Figure 2.23 : Microscope results of panels with/without repass and autoclave treatment.

Table 2.6 shows results for void content. It is no surprise that autoclave treated panels are the ones with lowest void content. The void content for the in situ panels are significantly decreased with the repass treatment. Results have shown that second repass treatment did not have much influence on the void content as much as first repass treatment.

Table 2.6 : Void content results.

Sample Condition	Void Content (%)	CV'
Without Repass	0.42	0.56
1 Repass treatment	0.28	0.46
2 Repass treatment	0.23	0.51
Autoclave treatment	0.03	0.83

CV'= Coefficient of Variable.

It can be seen from the Table 2.7 that degree of crystallinity also effected by the repass treatment. Since repass treatment is a fast process rate of cooling was high for polymer chains to get in crystal form that is why degree of crystallinity is decreased by the repass treatment. Autoclave is an environment with controlled temperature and pressure that is why crystallinity is much higher in autoclave level panels.

Table 2.7 : Degree of crystallinity results.

Sample Condition	Crystallinity (%)	SD*	Tm (°C)	SD*
Without Repass	30.4	1.3	342.6	0.2
1 Repass treatment	26.1	1.3	342.8	0.2
2 Repass treatment	23.7	0.2	342.6	0.3
Autoclave treatment	35.9	0.3	344.0	0.2

Note: The weight ratio of matrix is taken to be 32 %.

*SD= Standard Deviation.

In another study, flat coupons that are carbon fiber reinforced polymer composites are manufactured with AFP and compared with the coupons that are manufactured with proven manufactured methods like autoclave treatment. Table 2.8 shows the outcome of mechanical properties and how much change is observed with compared to

autoclave level coupons. In this study panels manufactured with automated fiber placement and autoclave has different fiber volume content that is why higher tensile and compression modules results of taken with AFP panels (having higher fiber volume content). To be able to make a fair judgement material property should have been the same [23].

Table 2.7 : Mechanical Properties of part with AFP and autoclave consolidation.

		AFP	Autoclave (A)	AFP/A
Tensile Modulus (GPa)	Average Value	165	136	122 %
	Standard Deviation	0.23	3.61	
	Coefficient of Variation (%)	0.14	2.66	
Tensile Strength (MPa)	Average Value	2420	2446	99%
	Standard Deviation	93.09	44.34	
	Coefficient of Variation (%)	3.84	1.81	
Compressive Modulus (GPa)	Average Value	144	123	117%
	Standard Deviation	7.08	0.40	
	Coefficient of Variation (%)	4.92	0.32	
Compressive Strength (MPa)	Average Value	1055	1407	75%
	Standard Deviation	37.70	9.44	
	Coefficient of Variation (%)	3.57	0.67	
Shear Modulus (GPa)	Average Value	5.3	5.5	96%
	Standard Deviation	0.28	0.20	
	Coefficient of Variation (%)	5.28	3.64	
Shear Strength (MPa)	Average Value	101	80	120%
	Standard Deviation	4.59	1.27	
	Coefficient of Variation (%)	4.54	1.59	

In another study mechanical properties of different coupons manufactured AFP having different laser power setting and two different fiber ply orientation is investigated by Kok et. al (2015) [26, 41]. Study is focused on the interlaminar bond strength (ILSS) because ILSS measures the load transfer between the layers. Figure 2.24 fracture

toughness values for different laser power configurations is given. Results has shown that a sharp decrease for the lowest and highest power setting is get. The maximum average fracture toughness is made a peak for 575 W.

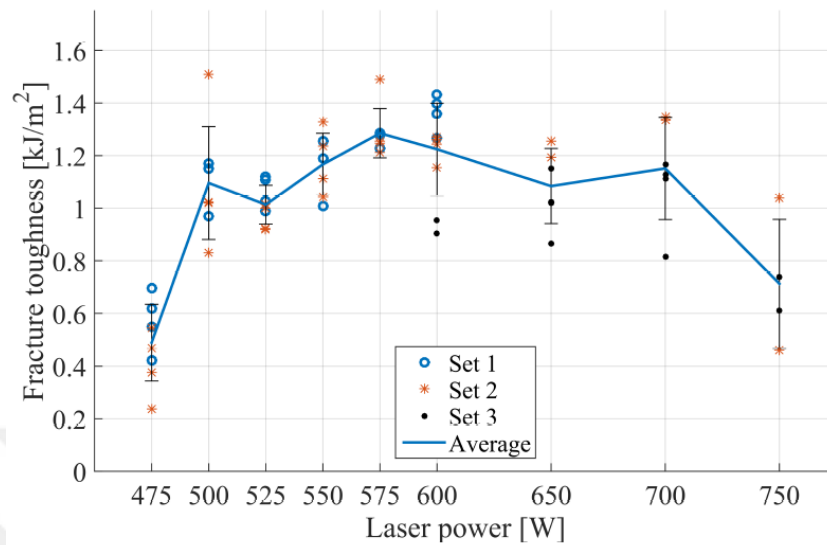


Figure 2.24 : Fracture toughness with different laser powers.

In another study, Eimanlou et. al (2018) investigated the effect of placement rate and laser power on the ILSS property. In Figure 2.25 results are shown. It can be seen that maximum ILSS value is reached in 100 mm/sec lay-up speed, but ILSS is decreased at high laser powers which can be explained by the degradation of the polymer matrix. The study shows that optimum laser power changes with different lay-up speeds.

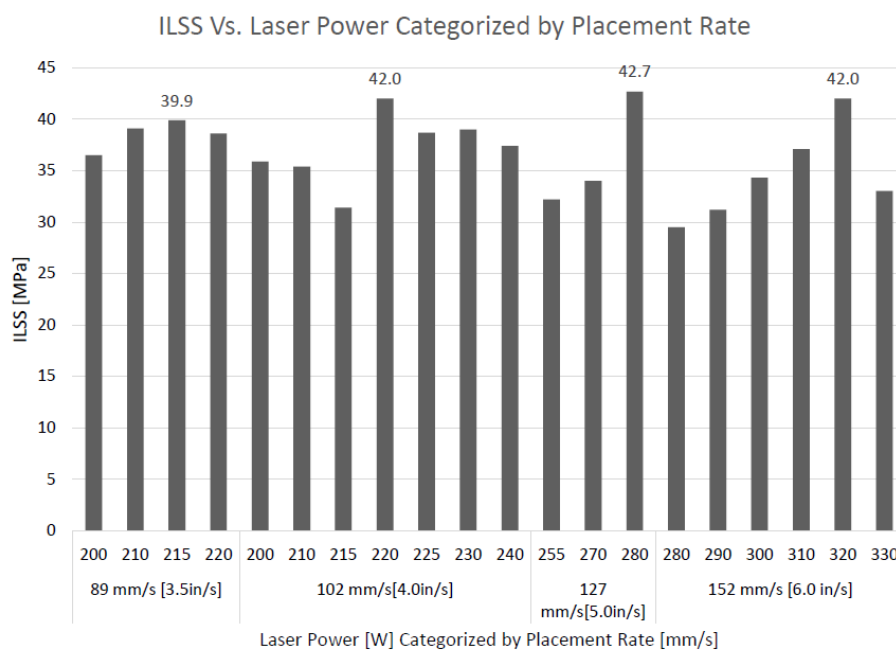


Figure 2.25 : ILSS results for different lay-up speed and laser power.



3. EXPERIMENTAL

3.1 Materials and Method

Material used in this study is Carbon fiber reinforced polyaryletherketone family semicrystalline polymer is used in slit tape form and laid down with Automated Fiber Placement, and also a similar matrix with relatively lower melting temperature is used to compare thermos-mechanical properties. In total 8 panels are manufactured, 2 of them laid down with average of 100 mm/sec speed, 2 of them are laid down with 400 mm/sec. Table 3.1 represents the configuration of manufactured parts. In every set, one of the panels are post consolidated in autoclave. Heat source for automated fiber placement robot is laser with nip point temperature of average 450 °C, compaction force of 500 N, roller stiffness of 60 Shore A and 4 tows are laid down at the same time. Post consolidated panels are consolidated at the autoclave for 400 mins. Heat up and cooling rate is set to 5 °C, pressure is applied as 8 bar, temperature is hold at average of 350 °C for 30 mins.

Table 3.1 : Configuration of manufactured panels.

		AFP Speed	
		Low Speed	High Speed
High Melt	In Situ	Panel Number 1	Panel Number 3
	Autoclave	Panel Number 2	Panel Number 4
Low Melt	In Situ	Panel Number 5	Panel Number 6
	Autoclave	Panel Number 7	Panel Number 8

3.2 Theory and Calculation

Studies have shown that for the manufacturing of the thermoplastic composite parts, three main steps are needed to complete: impregnation, consolidation and crystallinity [9]. Automated Fiber Placement process gives ability to control consolidation and crystallinity, however impregnation is depended on the material fed. Because of the fact that consolidation and crystallinity is the result of heat application, compaction force, time and lay-up rate has been changed to observe the result of giving polymer chains less time to get oriented, which is expected to alter crystallinity amount [16, 20]. Consolidation is related with the temperature and pressure applied, to be able to investigate performance of in situ consolidation, identical panels are autoclave treated. Studies have shown that even repress treatments on the panels manufactured by AFP has strong influence on crystallinity, more remarkably post consolidation has strong influence on final mechanical properties [23, 37].

In addition to the time that is needed to achieve complete consolidation, power of laser source has strong effect on the final product. Kok et al. have showed that at optimum temperatures which means less than degradation temperature but as high as possible, internal void content is decreased and mechanical performance is improved [26]. In this study, in order to observe effect of the post consolidation and speed of lay-up, samples are taken from the panels and DSC test is conducted to show amount of crystallinity, microcut analysis performed to show internal void distribution, gas pycnometer test is done to emphasize compatibility of the density with the void content. Finally, DMA test is conducted to show effect of post consolidation and lay-up speed on the thermo-mechanical properties.

3.2.1 Differential scanning calorimeter

Crystallinity calculation for the sample is done with the DSC measurement. Equipment view is given in Figure 3.1. DSC Equipment used in this study is Mettler Toledo DSC 3+. Test is conducted with the order described below [38].

1. 25.0°C, 5.0 min, N₂ 50.0 ml/min
2. 25.0..400.0°C, 10.00 K/min, N₂ 50.0 ml/min
3. 400.0°C, 5.0min, N₂ 50.0 ml/min
4. 400.0..25.0°C, -10.00 K/min, N₂ 50.0 ml/min

5. 25.0°C, 5.0min, N₂ 50.0 ml/min
6. 25.0..400.0°C, 10.00 K/min, N₂ 50.0 ml/min



Figure 3.1 : Differential scanning calorimeter test equipment.

Matrix content is taken as 34% and $\Delta H_{100\%}$ 130 J/g. Test condition is given in Table 3.2.

Table 3.2 : Test condition for differential scanning calorimeter test.

Test Condition	
Isothermal waiting conditions	at 25 °C 5 min
Temperature range	25 °C...400 °C
Temperature scanning rate	10 °C/min
Test atmosphere	Nitrogen 99,999 % purity
Gas flow rate	50 ml/min
Sample holder	Aluminium standard, 40 μ l

DSC test is conducted with double cycle. The first cycle gives the idea for the process parameter change, while the second cycle can be investigated for the material change properties.

Calculation of the crystallinity is conducted with respect to the equation explained in the equations (3.1) and (3.2) below.

Heating cycle analysis:

$$(\Delta H_f - \Delta H_{cc}) \times RC = A \quad (3.1)$$

$$C\% = \frac{\Delta H_{100\%}}{A} \quad (3.2)$$

ΔH_{cc} : Heat of cold crystallinity

$\Delta H_{100\%}$: Crystalline melt energy for 100% crystalline matrix

RC : Matrix content (34%)

3.2.2 Dynamic mechanical analysis

Mechanical performance of the panels are investigated with DMA analysis. Equipment view is given in Figure 3.2. Equipment used in this study is Mettler Toledo DMA1.



Figure 3.2 : Dynamic mechanical analysis test equipment.

Test condition for the DMA is given in Table 3.3.

Table 3.3 : Test condition for dynamic mechanical analyser.

Test Condition	
Clamping geometry/type	Single cantilever bending
Free length	17,0 - 17,5 mm
Frequency	1 Hz
Heating rate	3 °C/min
Amplitude	15 µm
Clamp torque	25 cNm
Specimen length	35,0 ± 0,2 mm
Specimen thickness	10,0 ± 0,2 mm
Temperature range	25...300°C
Preload	N/A
Offset control	None

3.2.3 Gas pknometer

Void content of the panels are investigated with automatic density analysis. Equipment view is given in Figure 3.3. Equipment used in this study is Automatic Density Analyzer, Anton Paar Quantachrome ULTRAPYC 1200e.



Figure 3.3 : Gas pknometer test equipment.

Test condition is given in Table 3.4. Cooling system is started prior to the test for at least 1 hour in 23 °C.

Table 3.4 : Test condition for density.

Test Condition	
Cell size	Large cell
Test temperature	23 °C
Type of gas used	Helium (at least 99,99 % purity)
Purge time	1 min
Target pressure	17.0 psig
Runs	10
Number of runs averaged	8

3.2.4 Microscope analysis

For the microscope analysis samples are cut from the original panel with water jet. Equipment view is given in Figure 3.4. Materials and equipment used for this study is PAresi KM-B Matrix + Flacon catalyser, Struers, Nikon eclipse LV100ND with Clemex vision lite. Samples are places on to 40 mm moulds through their cut section. Afterwards, matrix is pured in to the mould, waited for 30 mins and removed from the mould.



Figure 3.4 : Microscope analysis test equipment.

After the matrix impregnated sample is taken out of the mould they are sanded and polished. When the samples are polished enough and having a pure surface they are rinsed with distilled water and ethyl alcohol and than dried.

At the end samples are ready to be examined under the microscope 25X/50X/100X/200X/500X maginified views are photographed





4. RESULTS AND DISCUSSION

DSC test results have shown that lay-up speed has no significant effect on the crystallinity, whether parts are post consolidated in autoclave or left as in situ, crystallinity has not shown any remarkably change.

Graphs of first heating cycle is given in Figure 4.1 and Figure 4.2, second heating is related with the properties of the material, that is why only first heating cycles are investigated to observe effects of process parameters change.

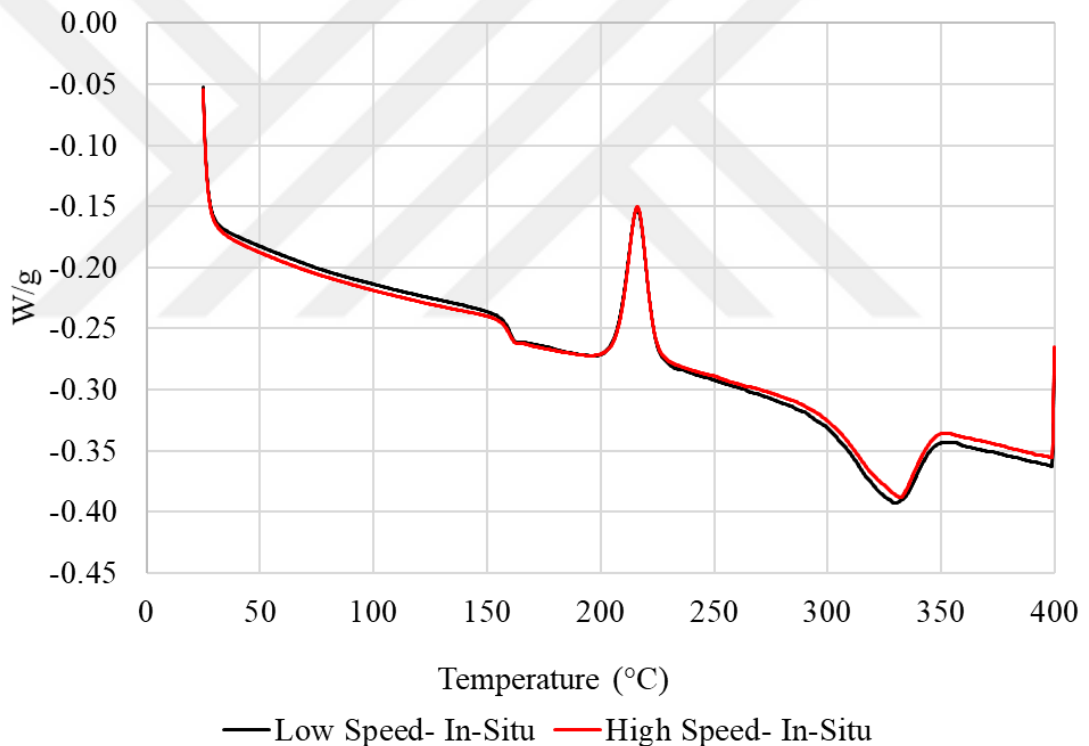


Figure 4.1 : First heating cycle for in situ panels with respect to speed change.

It has been concluded that lay up speed do not have a significant effect on the crystallinity degree. The first peak in the graph is cold crystallinity peak which is remarkable high in both lay up speeds which refers that there has not been significant amount of consolidation occurred in the in situ treated parts. After the autoclave treatment it is expected not to see cold crystallization peak in the differential scanning

calorimeter inspection. Figure 4.2 validates the idea of complete consolidation after autoclave treatment.

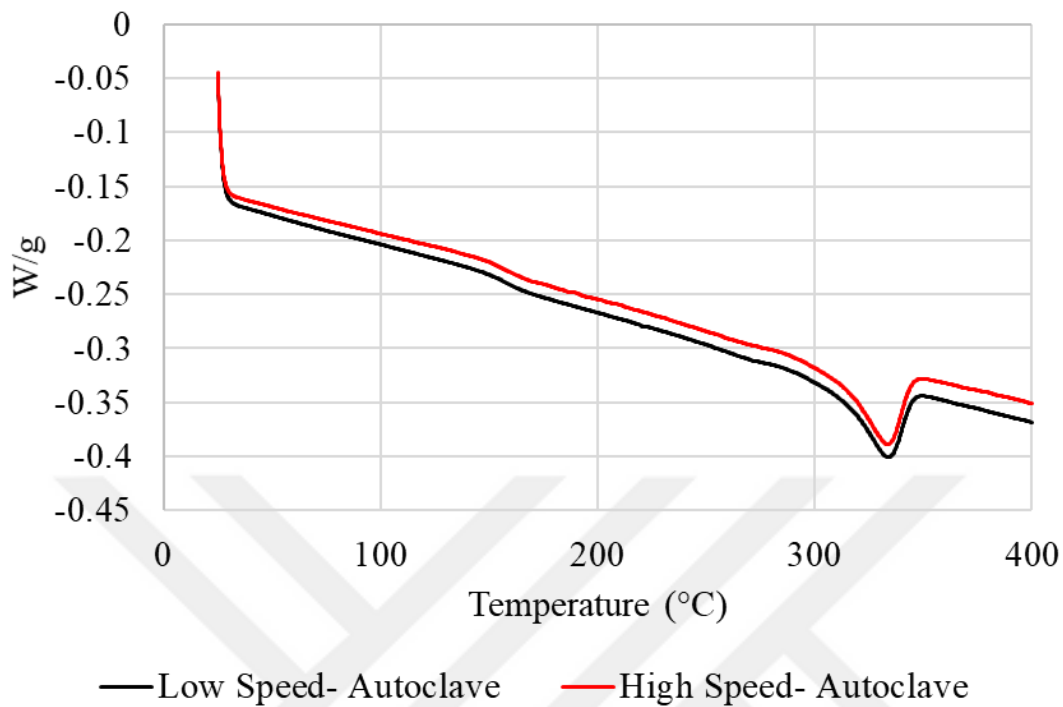


Figure 4.2 : First heating cycle for autoclave consolidated panels with respect to speed change.

After autoclave treatment thermal history of the part is erased, that is why crystallinity becomes independent from the lay-up speed. Table 4.1 shows crystallization percentages for all the panels.

Table 4.1 : Crystallization percentages for all panels.

Effect of Velocity on crystallization (%)			
Low Speed-In Situ	6.23	High Speed-In Situ	5.79
Low Speed-Autoclave	28.79	High Speed-Autoclave	29.31

As it can clearly be seen in Table 4.1 and Figure 4.3 autoclave treatment improved crystallinity percentage significantly.

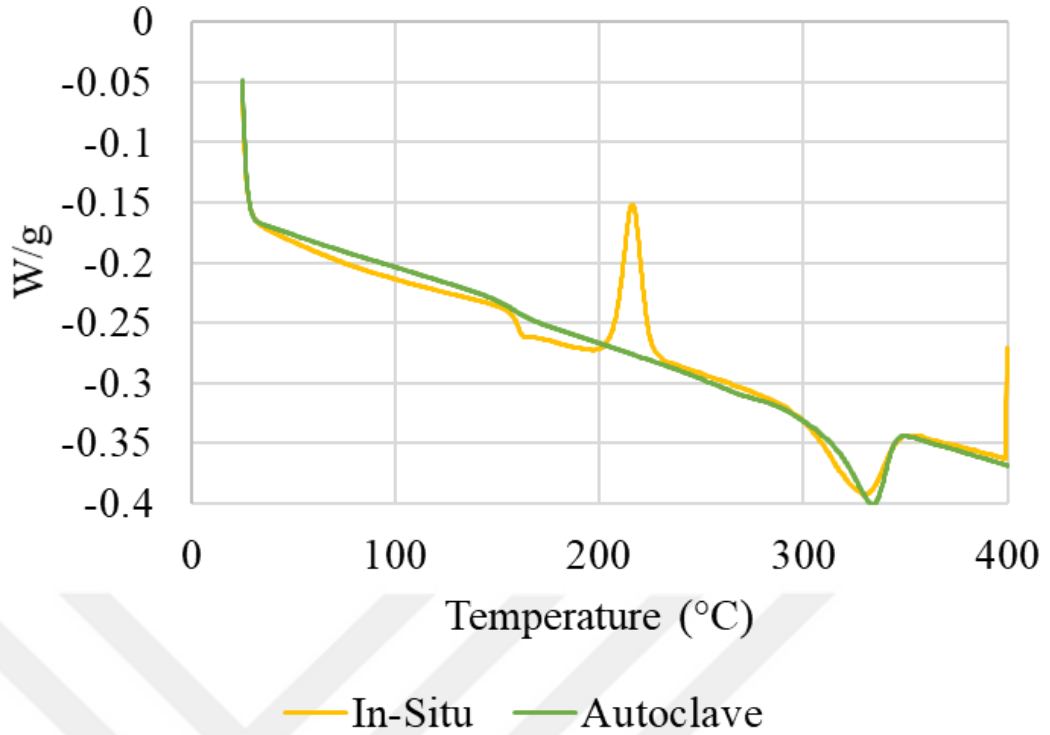


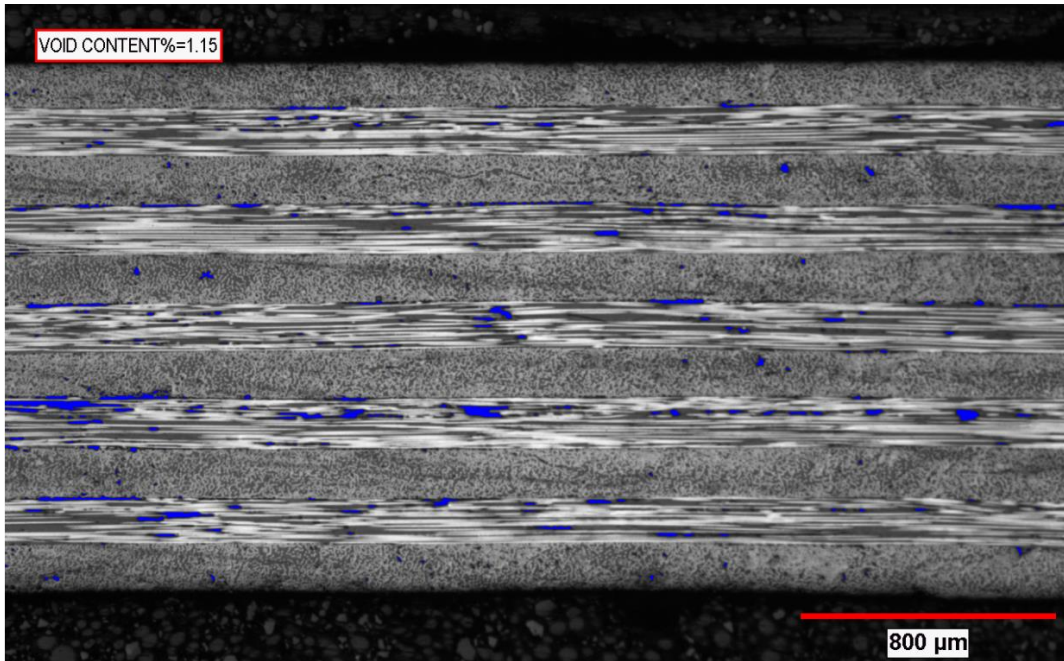
Figure 4.3 : First heating cycle for in situ and autoclave consolidated and low speed parts

Gas pycnometer analysis is conducted to observe if there is a significant effect of process parameter on the density. Results are tabulated in Table 4.2, showing that lay-up speed has no effect on the density while autoclave treatment increases the density.

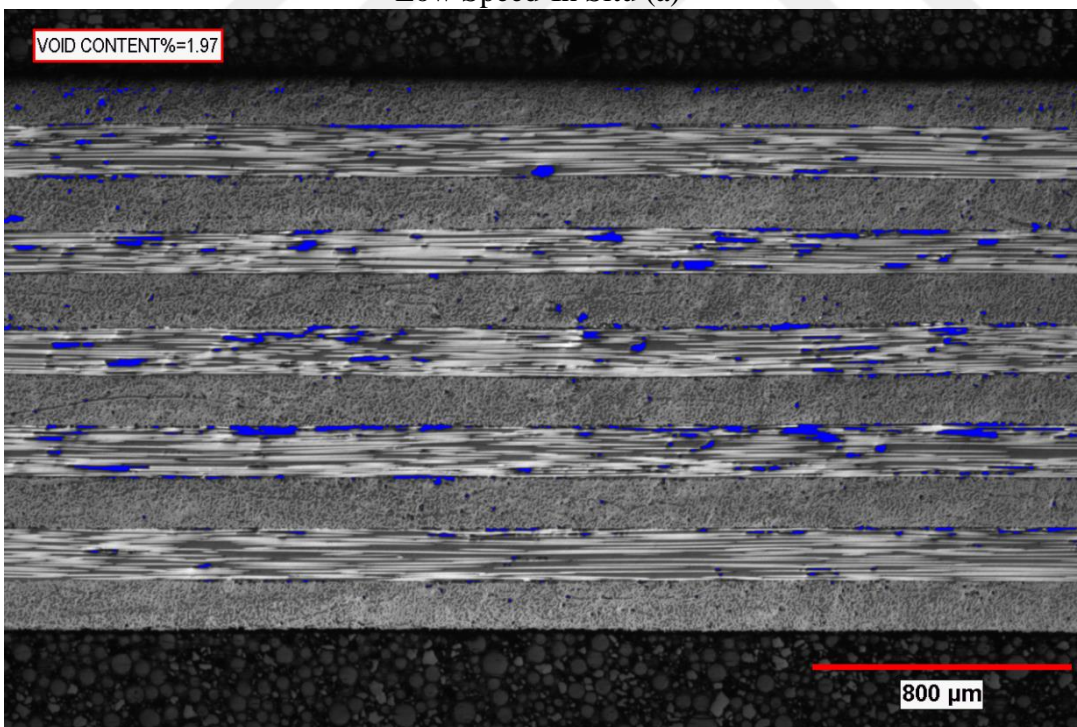
Table 4.2 : Density results with respect to autoclave treatment and lay-up speed.

Effect of Velocity and Post Consolidation on density (g/cc)			
Low Speed-In Situ	1.52±0.0008	High Speed-In Situ	1.53±0.0010
Low Speed-Autoclave	1.54±0.0014	High Speed-Autoclave	1.56±0.0017

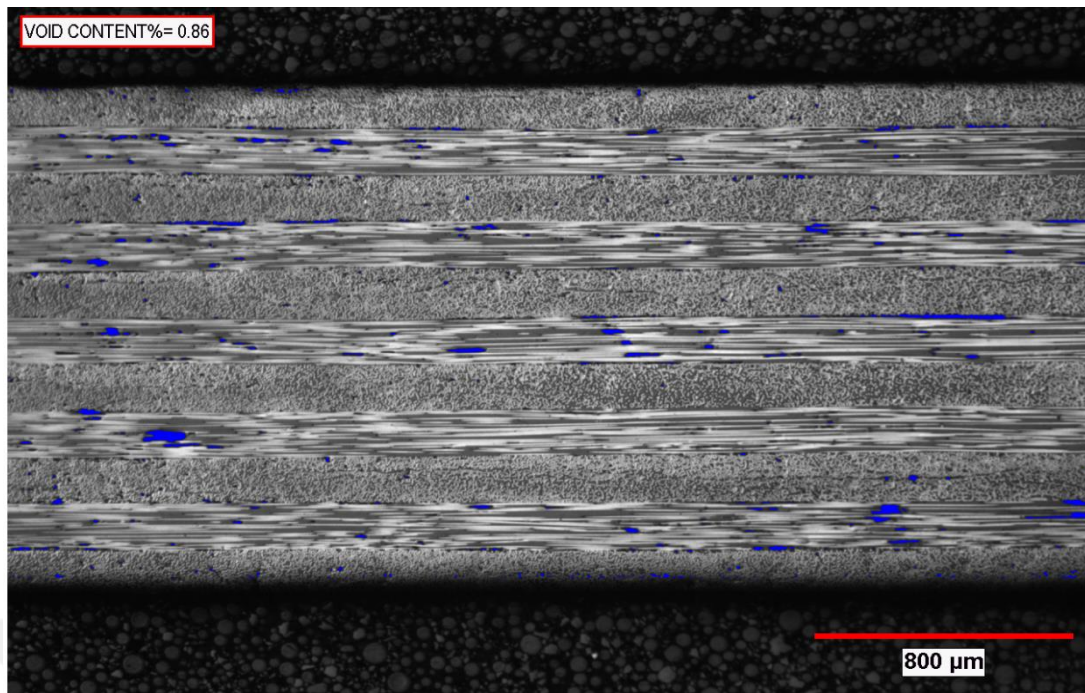
Figure 4.4 gives microcut analysis results which complies with the gas pycnometer test, showing that autoclave treatment reduces internal void content, while lay-up speed has no remarkable effect on the void content.



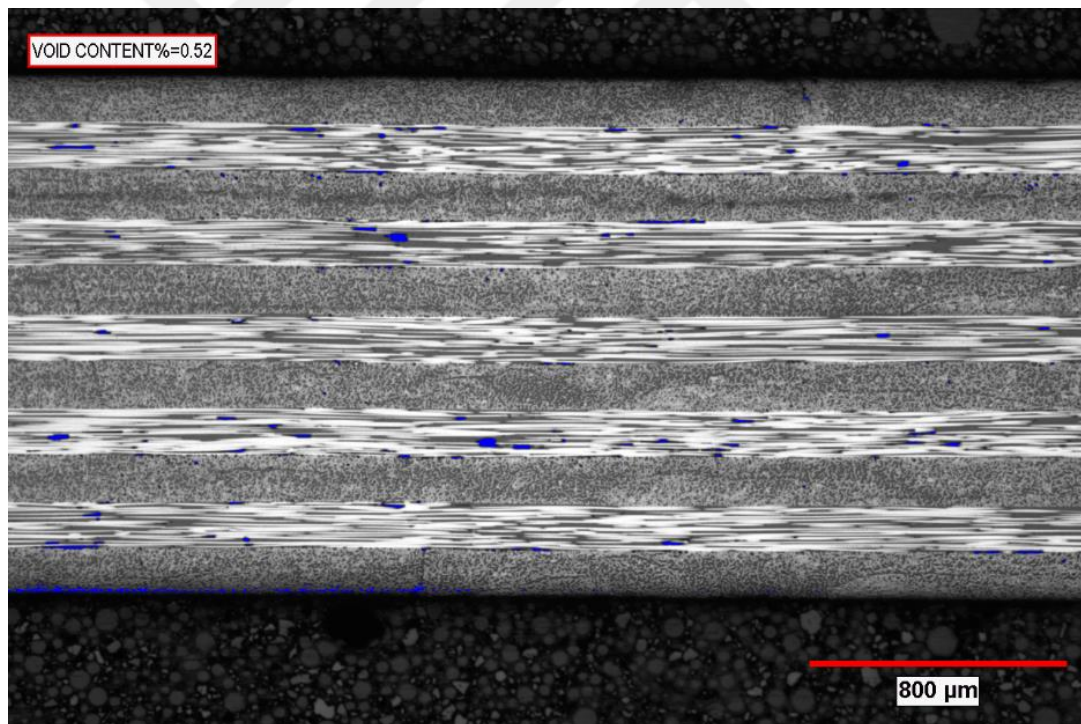
Low Speed-In Situ (a)



High Speed-In Situ (b)



Low Speed- Autoclave (c)

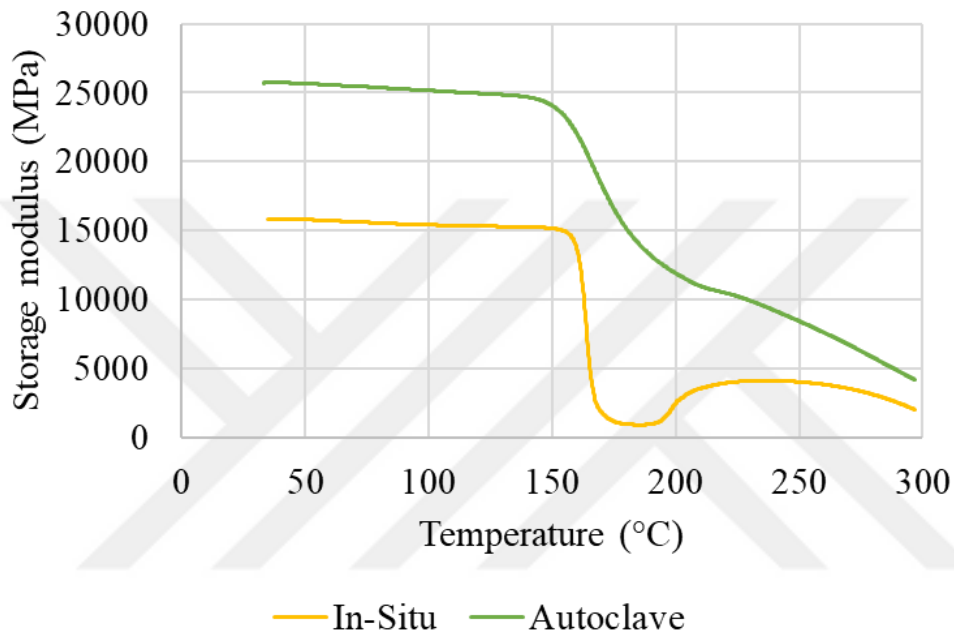


High Speed-Autoclave (d)

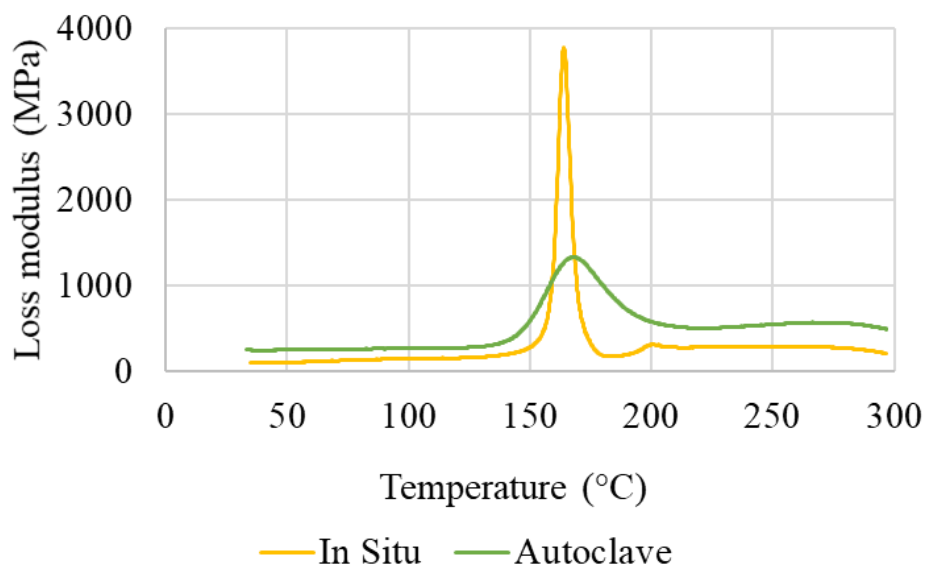
Figure 4.4 : Microcut analysis for in situ (a,b) and autoclave (c,d) treated panels.

For the thermo-mechanical performance investigation samples are taken for DMA test. Results are given in Figure 4.5. It can be concluded that mechanical properties are improved over 65%. This can be explained by the decrease of internal void content, improving crystallinity. Storage modulus analysis is related with the measure of the

elasticity and the ability of a material to store the energy, loss modulus on the other hand is the ability of the material to lose the energy, it can be explained as lost energy in the form of heat. Storage modulus value increased significantly after autoclave treatment which is a desired property increase for the materials having higher strength. Tan delta value is calculated by dividing loss modulus to storage modulus. It is a measure of vibration and damping property, which is not desired to see in the structural parts.



Storage Modulus (a)



Loss Modulus (b)

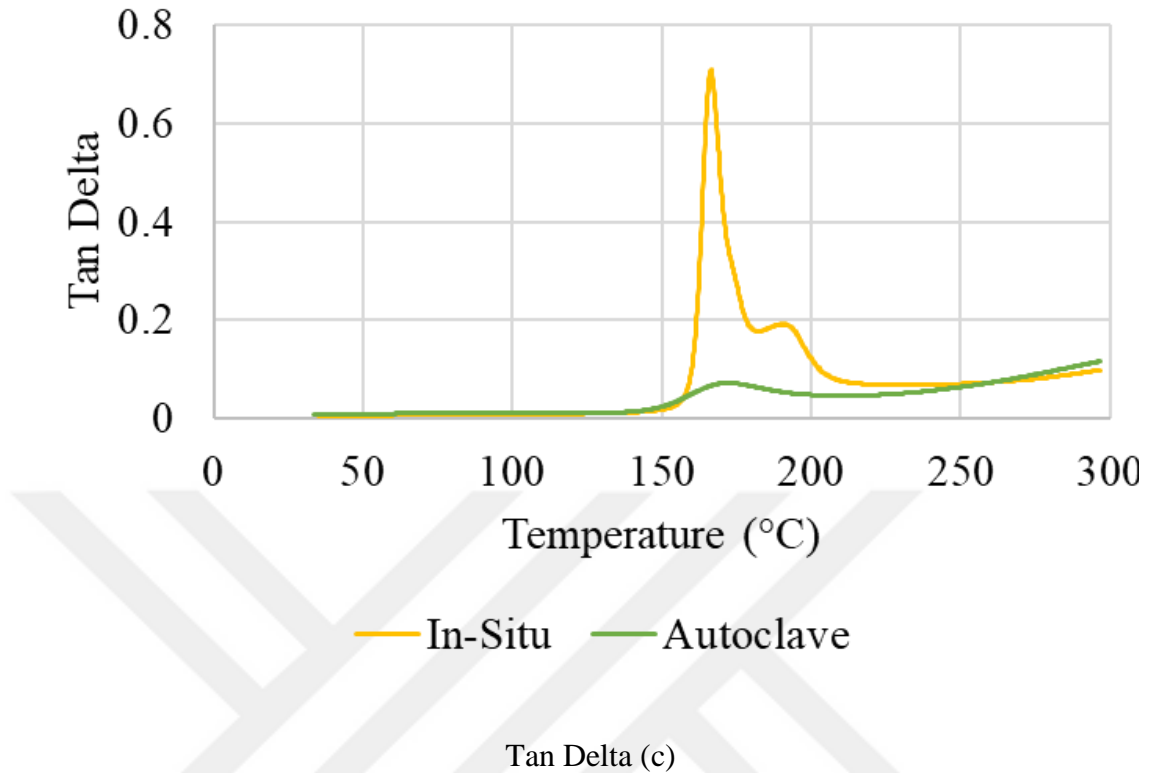


Figure 4.5 : DMA results; storage and loss modulus (a,b), tan delta (c) for autoclave post consolidated and in situ panels.

In the second part of the study, a different material for the matrix is used, in this composite material melting temperature of the matrix is relatively lower than the other. In the Figure 4.6 and Figure 4.7 first and second heating cycle analysis of the high and low melt composite material is given.

It has been concluded that lower melting temperature increases the crystallinity but not as much for the autoclave level final product, regardless of process lay-up rate. It is expected to see the outcomes of higher crystallinity in the thermo-mechanical properties. Since, crystallinity increases the stiffness which is desired to some extent, in such composites 25-30 % crystallinity is expected to achieve to see the desired thermo-mechanical properties and to guarantee the full consolidation.

In the case of relatively low melt matrix composite, nearly 5 % higher crystallinity is achieved. To be able to see the effect of crystallinity increase dynamic mechanical analysis is also conducted.

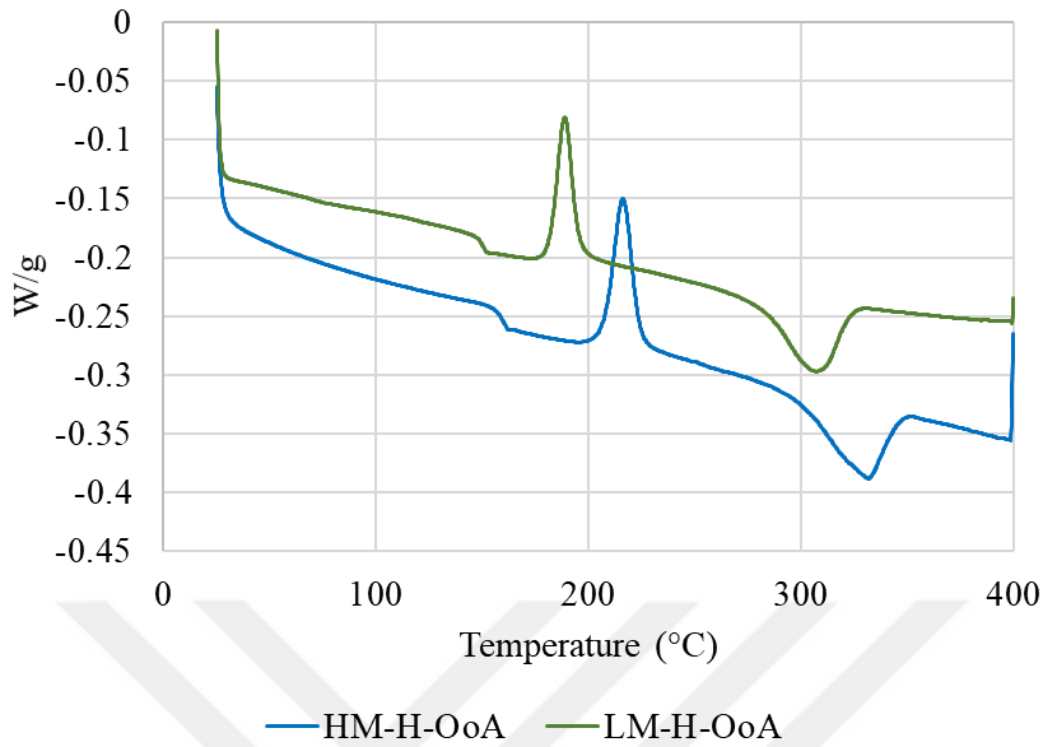


Figure 4.6 : First heating cycle for high/low melt panels.

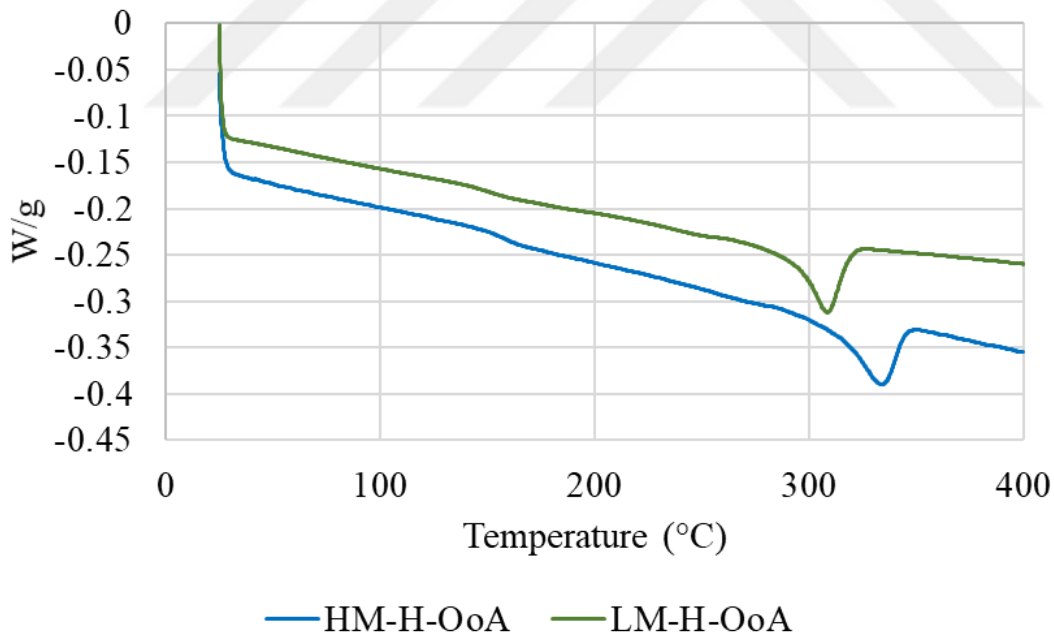


Figure 4.7 : Second heating cycle for high/low melt panels.

In the Table 4.3, crystallinity percentage for high and low melt composite are given. In this comparison all the panels are left as in situ without any further post consolidation. Results has revealed that lay-up speed is not a significant parameter for crystallinity, panels with higher lay-up rates results in lower crystallibity percentage

as expected. Low melt material has slightly more crystalline structure than high melt material. Results are not close to autoclave level crystallinity percentages.

Table 4.3 : Crystallinity (%) for in situ panels.

Effect of Material Change on Crystallinity (%) for In situ

Low Speed-High Melt	6.23	High Speed- High Melt	5.79
Low Speed-Low Melt	11.58	High Speed- Low Melt	9.95

Identical panels with the previously described panels are post consolidated in the autoclave. In the Table 4.4 effect of post consolidation is investigated in terms of crystallinity. Low melt material has slightly higher crystallinity degree than the low melt material but the main difference is in the post consolidation. Autoclave post consolidated panels crystallinity has remarkable increased with compare to in situ panels.

Table 4.4 : Crystallinity (%) for autoclave consolidated panels.

Effect of Material Change on Crystallinity (%) for Autoclave Consolidated

Low Speed-High Melt	28.79	High Speed- High Melt	29.31
Low Speed-Low Melt	31.15	High Speed- Low Melt	33.82

To be able to investigate the effect of polymer melting temperature on the density under the same process conditions gas pycnometer analysis is conducted to the samples collected from the panels. Density calculation results are tabulated in Table 4.5 for in situ panels. Results have shown that low melt matrix composite's density is relatively higher than the high melt, this can be explained by the decreasing void content with the application of heat above melting temperature.

Table 4.5 : Density (g/cc) for in situ panels.

Effect of Material Change on Density (g/cc) for In situ

Low Speed-High Melt	1.52±0.0008	High Speed- High Melt	1.53±0.0010
Low Speed-Low Melt	1.54±0.0013	High Speed- Low Melt	1.56±0.008

A similar comparison of density for the high and low melt matrix composite material is done with post consolidated panels. Results are given in Table 4.6. It has been seen that as in the case of in situ panels, low melt materials density is nearly same but a little bit higher than the high melt material.

Table 4.6 : Density (g/cc) for autoclave consolidated panels.

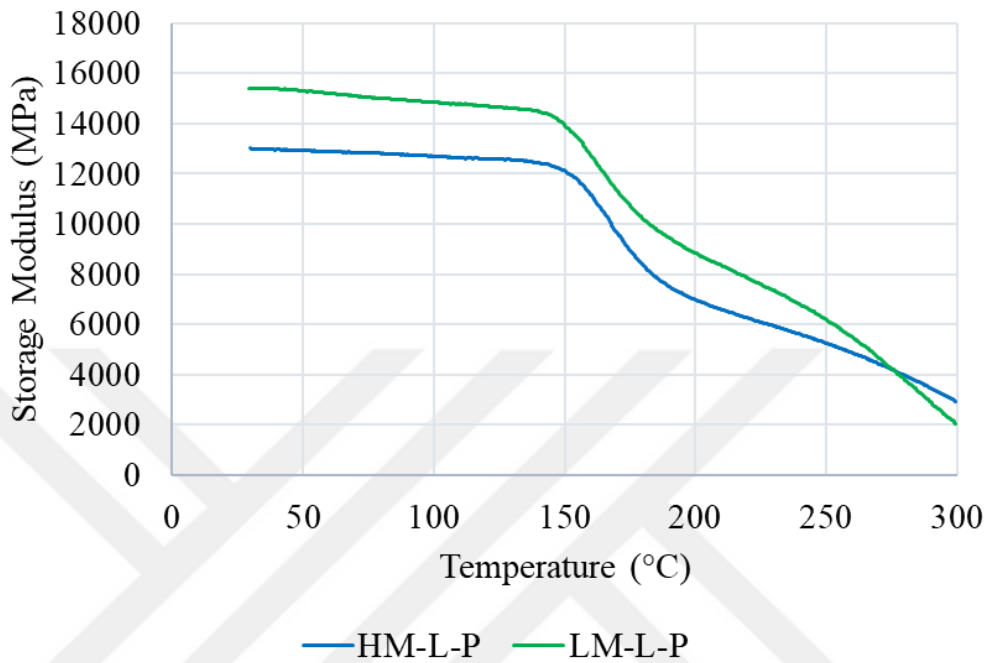
Effect of Material Change on Density (g/cc) for Autoclave Consolidated

Low Speed-High Melt	1.54±0.0014	High Speed- High Melt	1.56±0.0017
Low Speed-Low Melt	1.57±0.0014	High Speed- Low Melt	1.57±0.0015

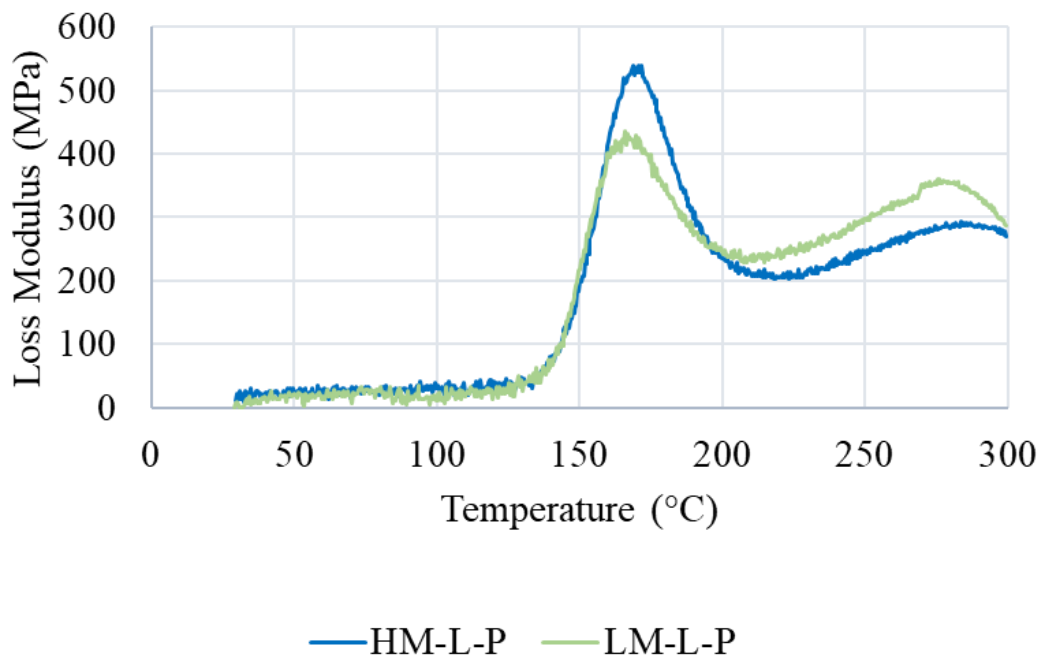
For the investigation of thermo-mechanical properties dynamic mechanical analysis is conducted. Results are given in the Figure 4.8. It has been seen as in compliance with the crystallinity values, low melt matrix composite material shows nearly same mechanical performance with the high melt matrix composite parts.

Relatively higher storage modulus value in case of low melt material can be explained with the higher crystallinity results get from the differential scanning calorimeter test. Higher the crystallinity result better the amount of consolidation and thermo-

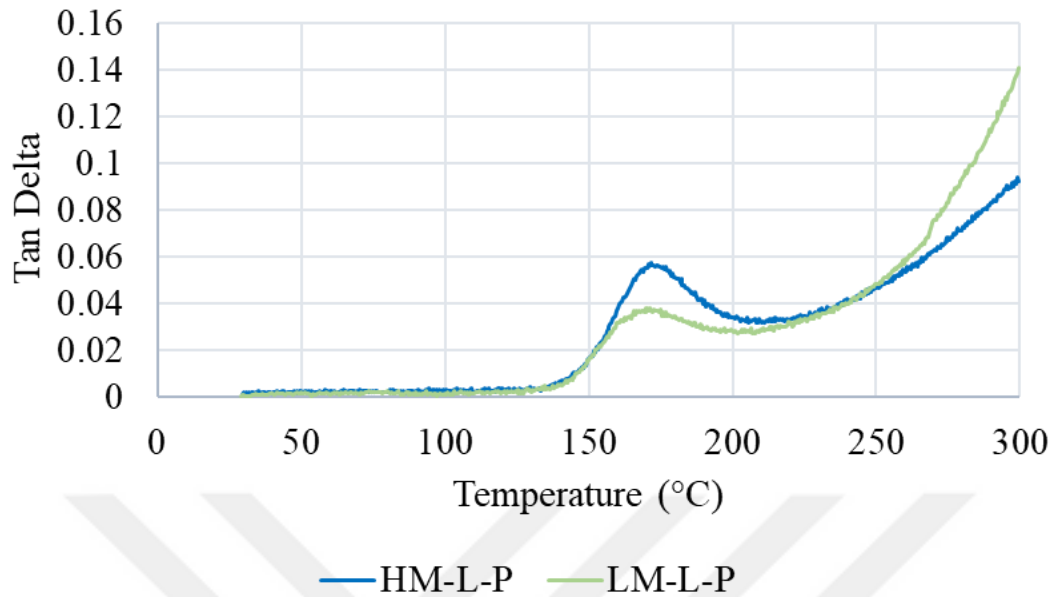
mechanical properties. Storage modulus and tan delta results are also complies with each other. Loss modulus value dropped a little in case of low melt which is the amount of energy lost as heat. Damping property is relatively increased in case of low melt as expected.



Storage Modulus (a)



Loss Modulus (b)



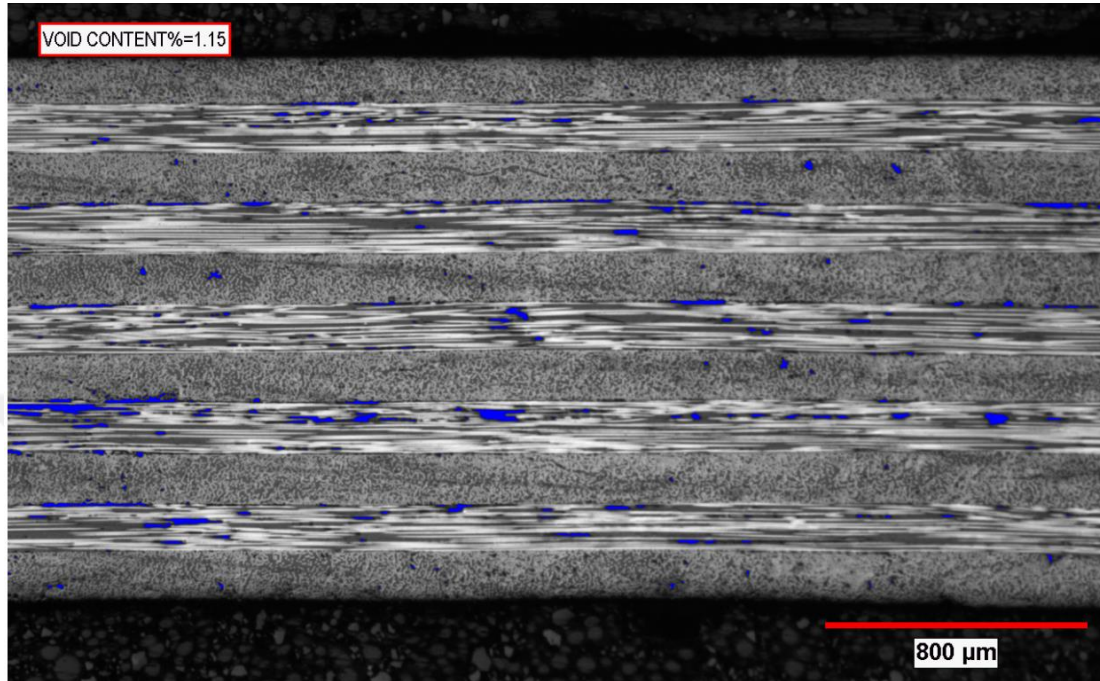
Tan Delta (c)

Figure 4.8 : DMA Results; storage and loss modulus (a,b), tan delta (c) for LM-in situ panels.

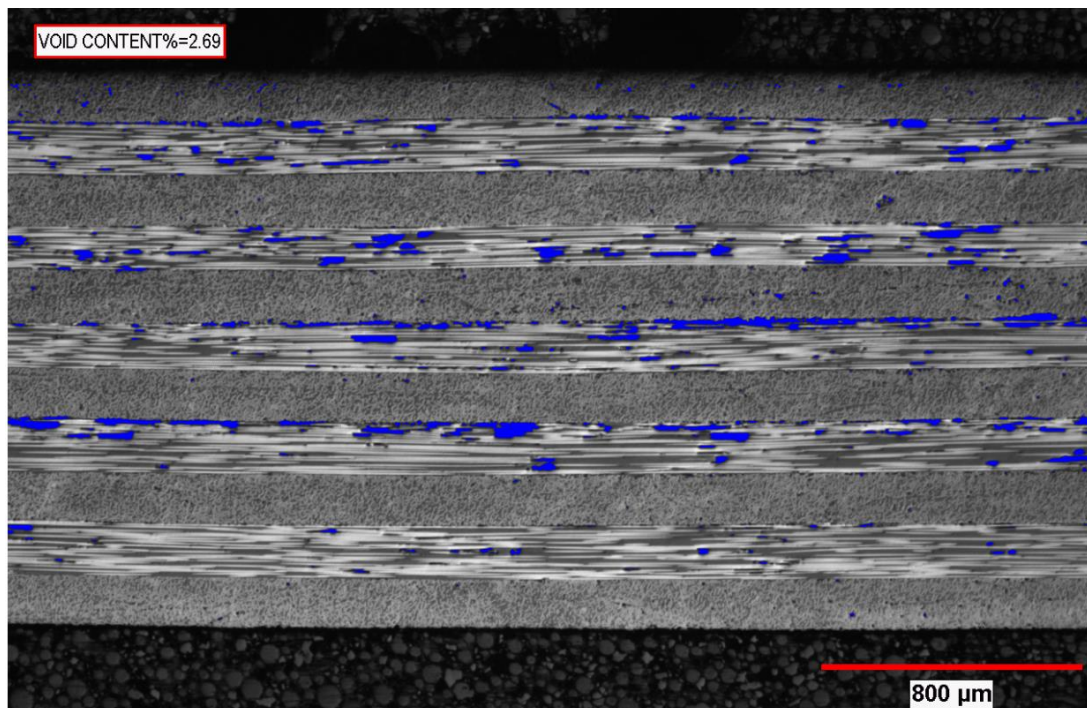
In order to see the internal void content microscope samples are taken from the parts. In the Figure 4.9 microscope views of in situ panels for high and low melt matrix composite material is given. It has been seen that there is no remarkable effect of melting temperature on the void content.

Regardless of melting temperature all the panels have shown high void content. However, it has been seen that when the lay-up speed increases than the void content increases in most of the cases. Since the microscope analysis is done with a small sample taken from the original part, it is not expected to conclude much from the result because the sample could be taken from a micro region in which an internal defect taking place. Those defects can be delamination, wrinkle or a resin rich area. That is the reason why of making gas pycnometer test in addition to the void content analysis. Detailed microscope analysis results are given in the appendix to be able to address more accurate conclusion. microscope test analysis is also highly depended on operator of test. Two different operator might get different results with the same sample, that is why objected judgement is only achievable with additional testing.

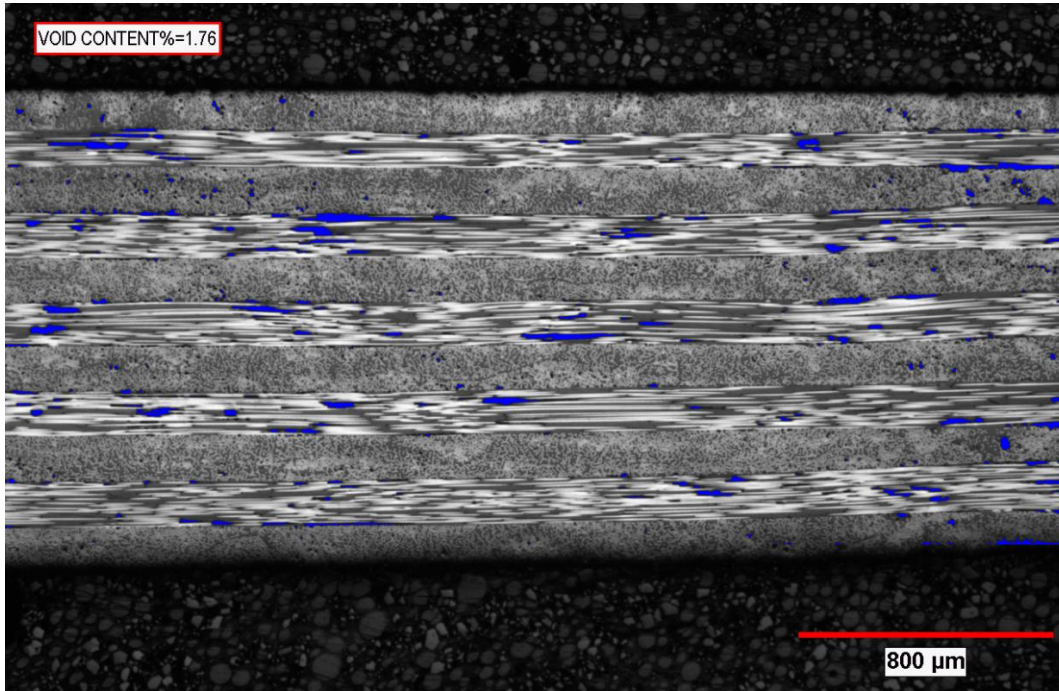
It has been concluded that regardless of the lay up speed, for the high melt material without autoclave treatment void content value is higher than 1%. Which is not desired for the high quality part. Results shows similar pattern in case of relatively low melt material having more than 1% of void content.



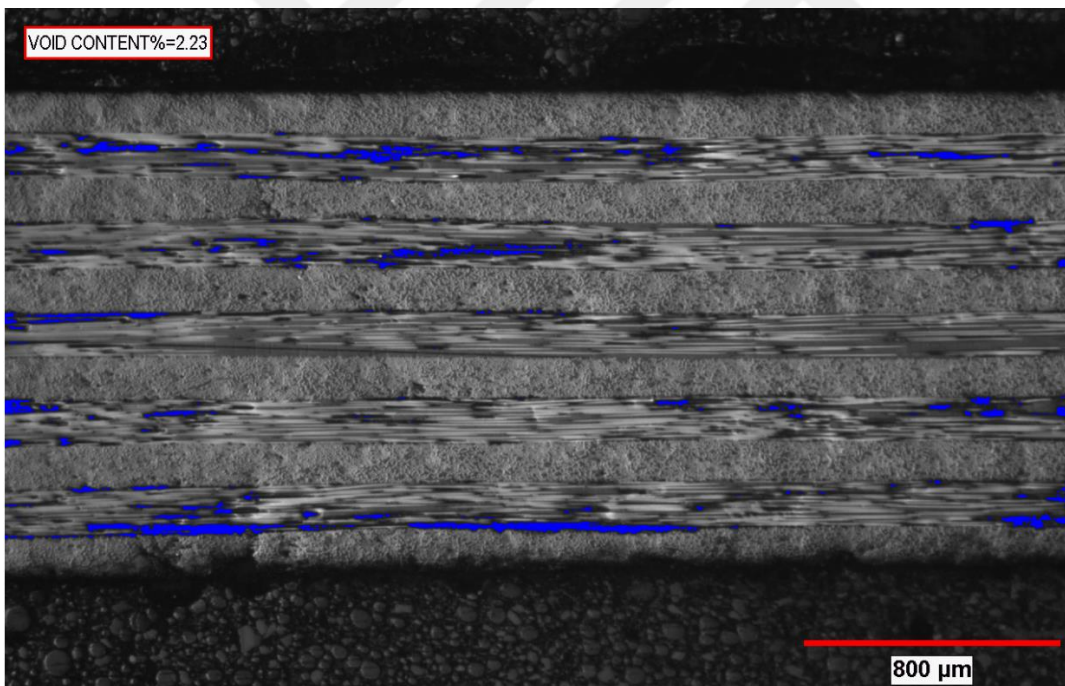
Low Speed- High Melt (a)



High Speed- High Melt (b)



Low Speed-Low Melt (c)

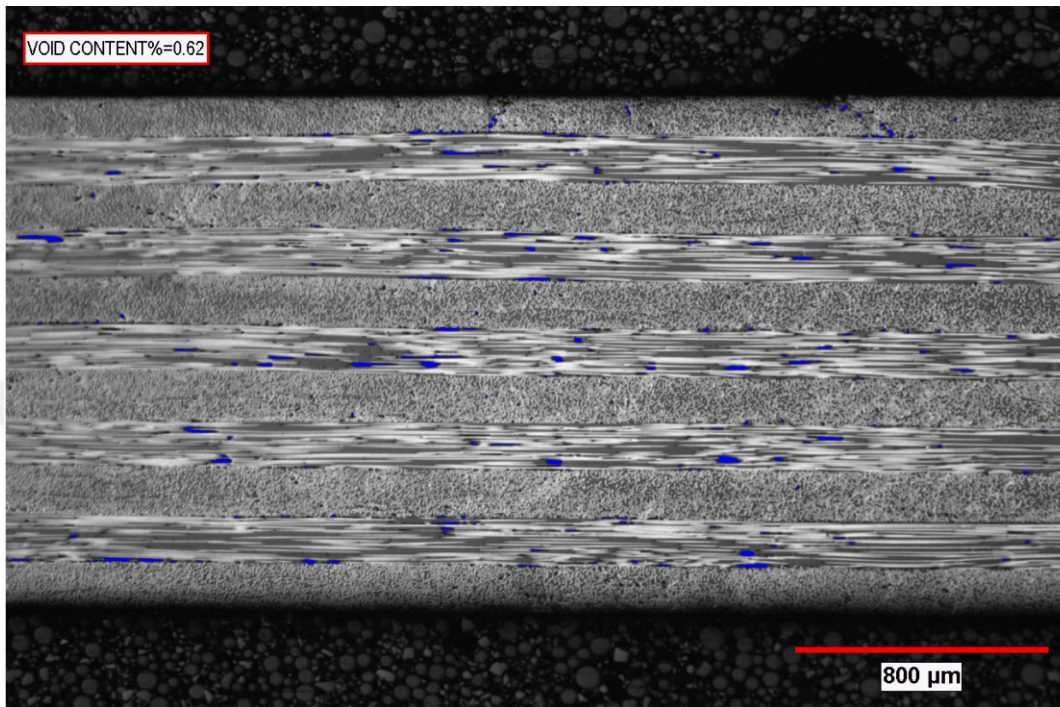


High Speed- Low Melt (d)

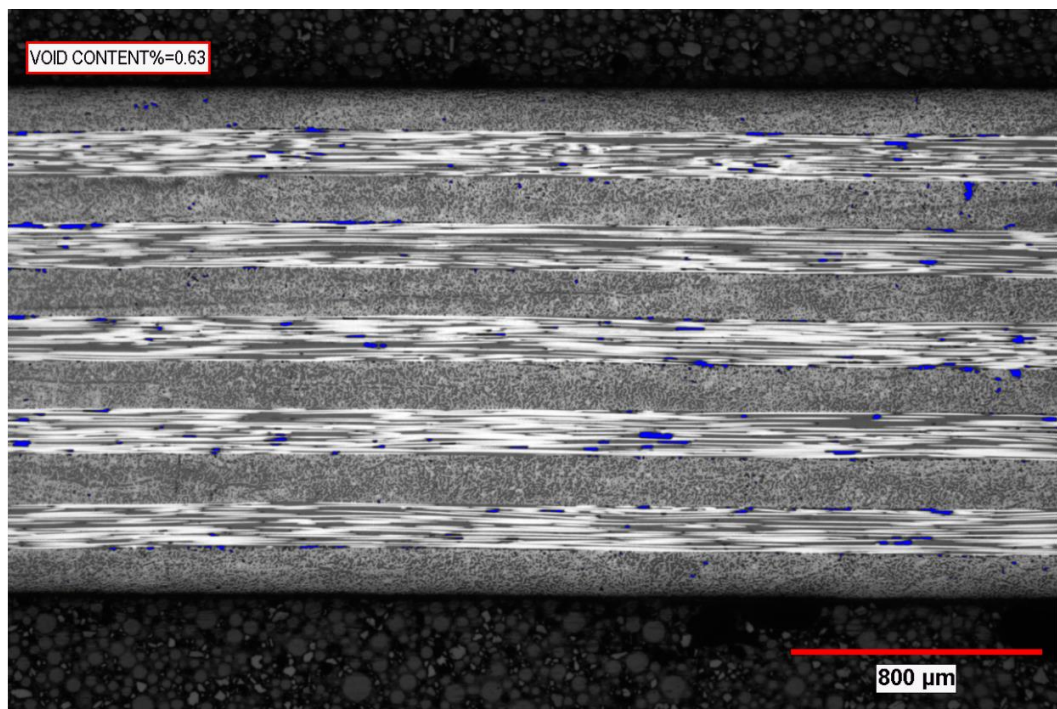
Figure 4.9 : Microcut analysis for high (a,b)/low (c,d) melt in situ panels.

A similar analysis is done for the autoclave post consolidated panels. Microcut analysis for the autoclave post consolidated panels are given in Figure 4.10. Results have shown that post consolidation has decreased the void content significantly with

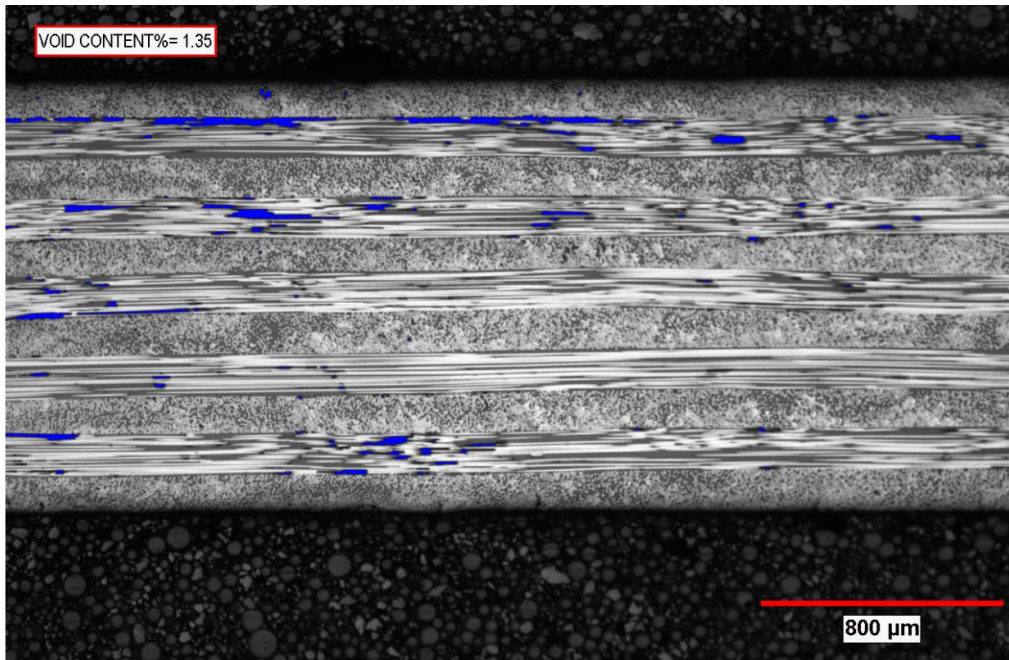
compare to in situ panels. However, microcut analysis might not be a reliable way of determining the internal void content. Since, microcut is taken in a small part of the whole part that is why there can be a spot in which porosity is cumulated which may not be the case for the entire structure.



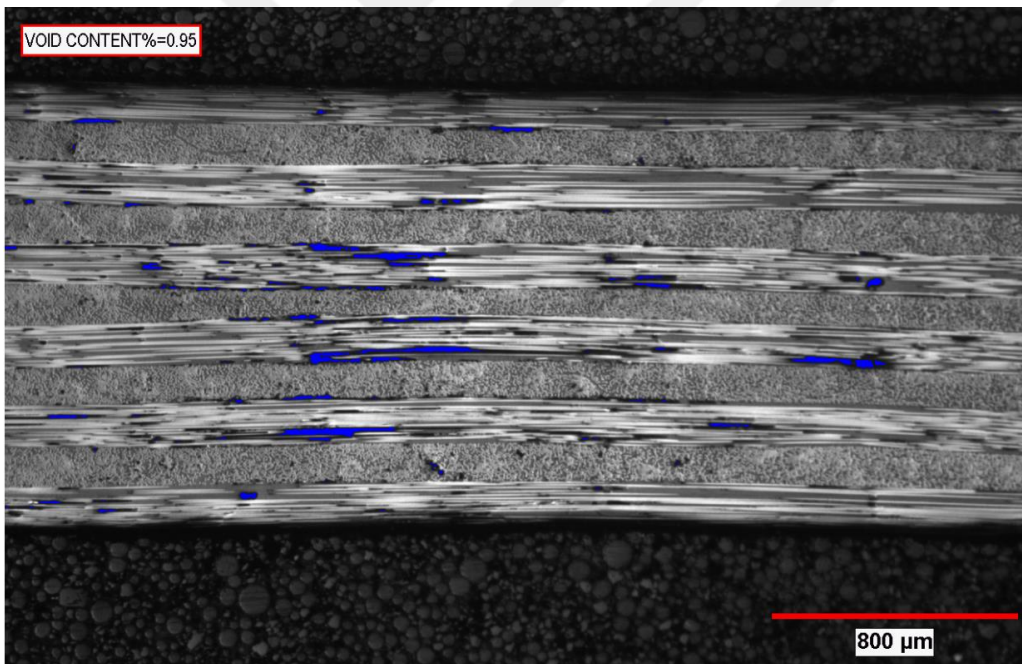
Low Speed- High Melt (a)



High Speed- High Melt (b)



Low Speed-Low Melt (c)



High Speed- Low Melt (d)

Figure 4.10 : Microcut analysis for high (a,b) /low (c,d) melt autoclave treated panels.

5. CONCLUSION

The recent trends in composite manufacturing for aerospace grade materials requires fast and reliable processes. The rate enabler process, AFP with the availability of digital manufacturing has been growing in the market. However, the literature still lacks an integrated process design to minimize the manufacturing cost with maximum part performance. This process design also needs to consider the new material developments to satisfy the market needs. In this study, a parametric study is presented to observe the effect of i. process parameters of AFP lay-up, ii. Post-consolidation type and iii. polymeric matrix type with different melting temperature towards an optimum process design.

This study reveals that there is no significant effect of lay-up speed on crystallinity. When the placement rate is increased to 400 mm/sec from 100 mm/sec, crystallinity percentage is decreased from 6.23 % to 5.79 %. This increase is even dropped to less than 1 % when the parts are autoclave post consolidated. This can be explained by the starting over with the thermal history of the parts when the parts are objected to autoclave treatment. Results have shown that lay-up speeds can be elevated for the mass production to increase efficiency.

Post consolidation has a strong influence on both degrees of crystallinity and thermo-mechanical properties, which can be explained by the applied energy and pressure in the autoclave which reduces internal void content and improves effective consolidation. Autoclave post consolidated panels have shown increase on degree of crystallinity nearly five times; crystallinity of the panels with low placement rate has increased to 28.79 % from 6.23 % and for the high placement rate panels crystallinity has increased to 29.31% from 5.79 %. Post consolidation of panels have a remarkable decrease on internal void content under 1% with microcut analysis. In addition, autoclave post consolidated panels have 25000 MPa of storage modulus while in situ panels have 15000 GPa which means nearly 65% improvement.

In terms of the polymer matrix type change with different melting temperature, there has been no significant improvement on both degree of crystallinity and the thermos-

mechanical properties. In the case of low melting temperature polymer matrix composite without further post consolidation crystallinity has increased to 9.95 % from 5.79 % and 11.58 % from 6.23 % for placement rate of 400 mm/sec and 100 mm/sec, respectively. When the parts are autoclave post consolidated, crystallinity has increased to 33.82 % from 28.31 % and 31.15 % from 28.79 % for placement rate of 400 mm/sec and 100 mm/sec, respectively. Changing the matrix polymer with low melt material has not shown a noticeable change on the thermo-mechanical analysis, that storage modulus value of low melt is about 15000 MPa while high melt is 13000 MPa.

In order to conclude, further research and analysis is needed to achieve autoclave level performance part manufacturing without any post consolidation. This study has shown that changing placement rate and polymer matrix does not have significant effect on the performance of the product, but the post consolidation significantly increases the performance of manufactured part.

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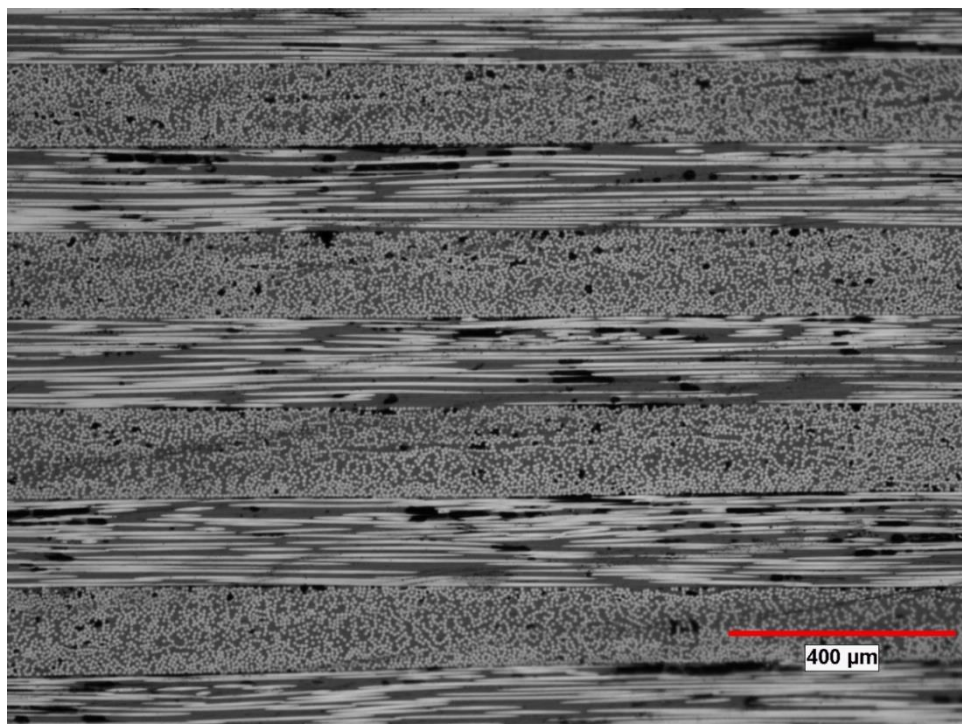
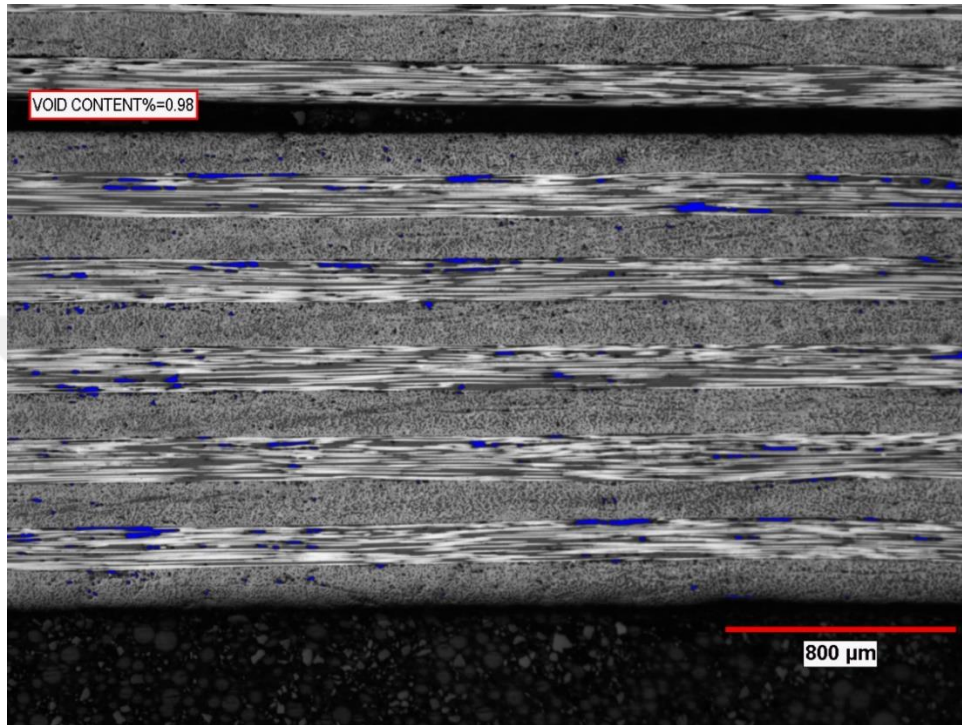
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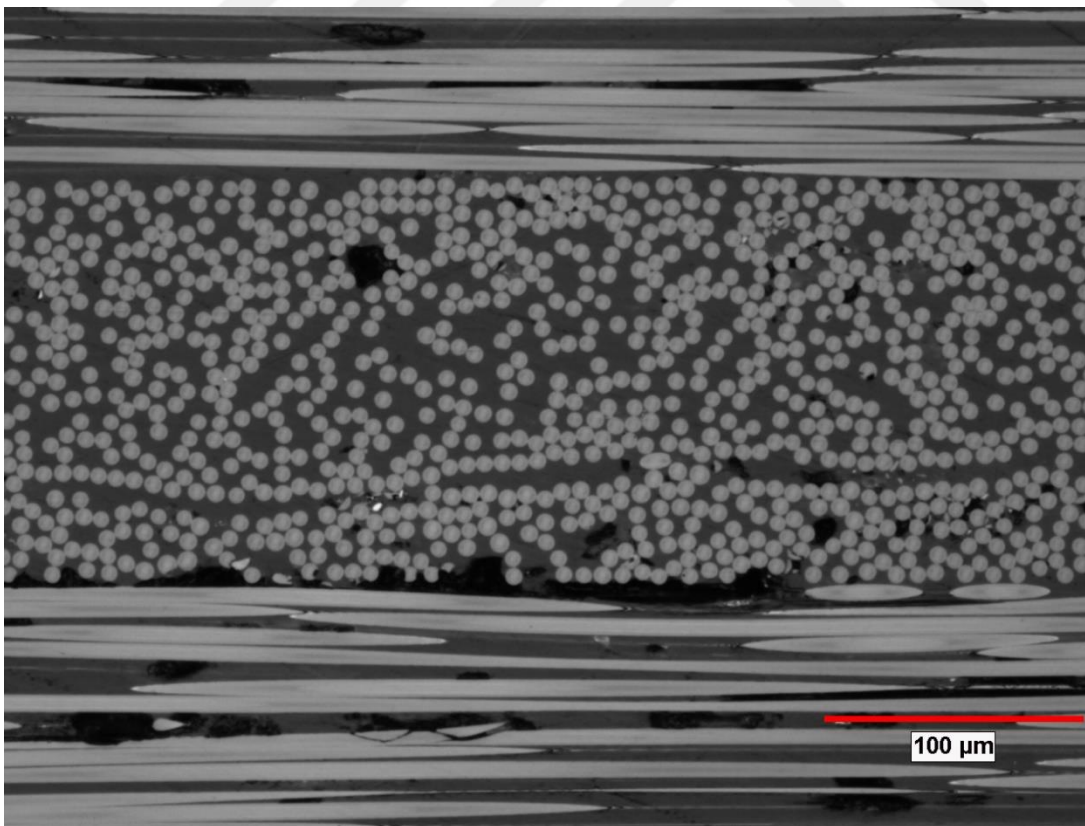
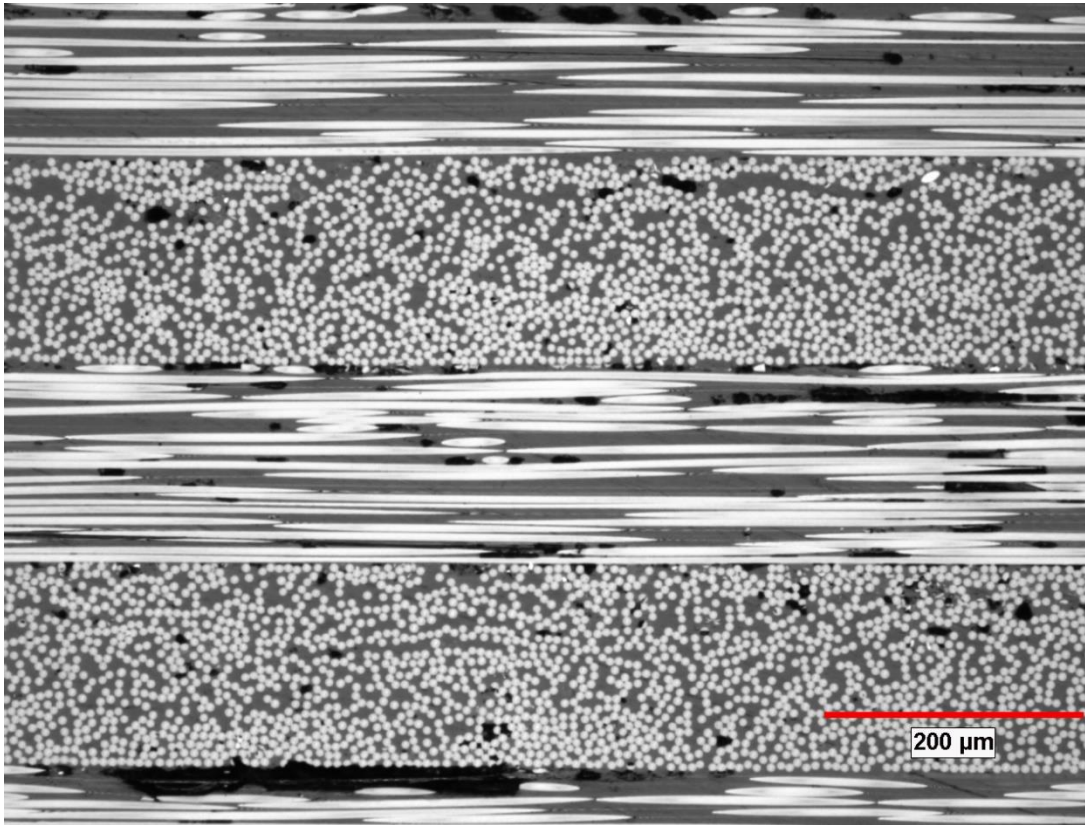
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APPENDIX





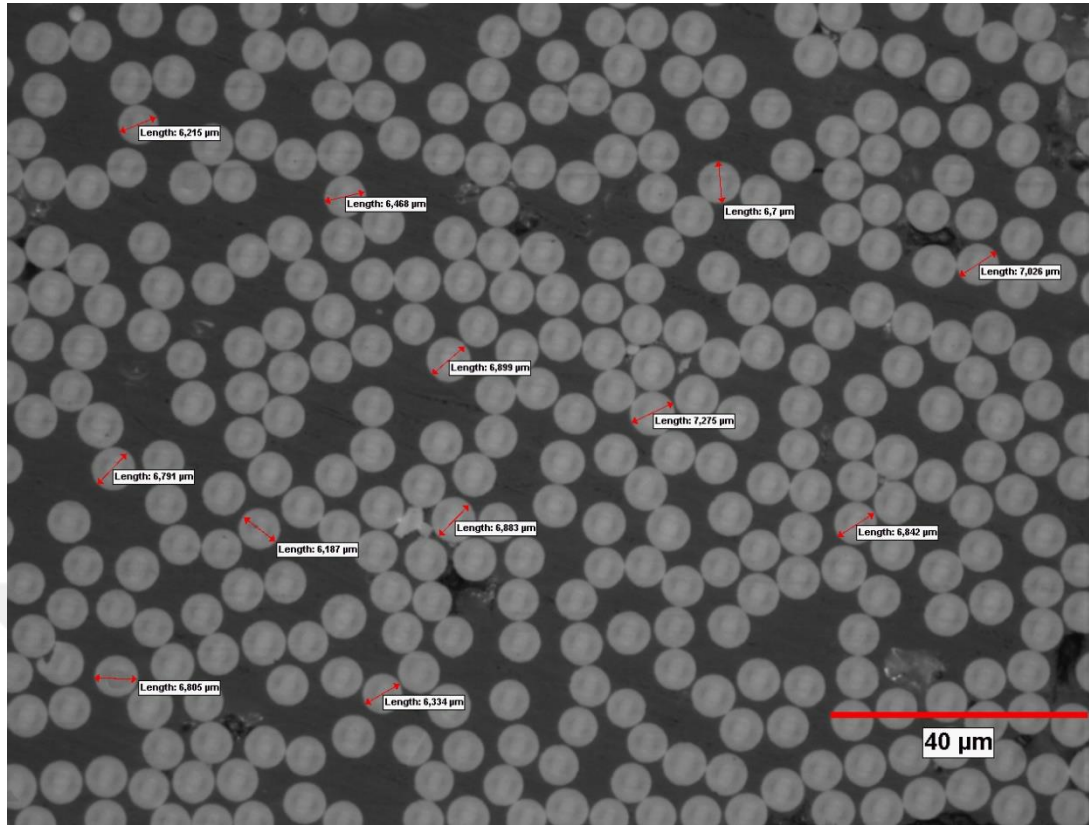
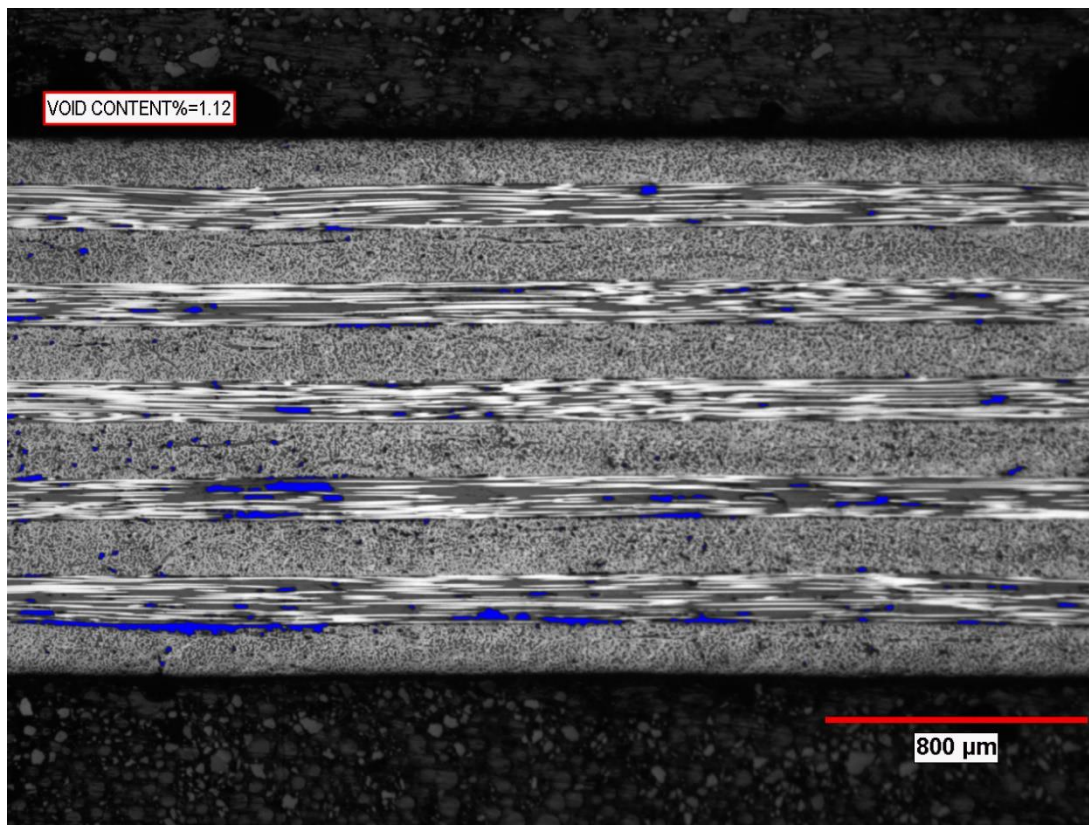
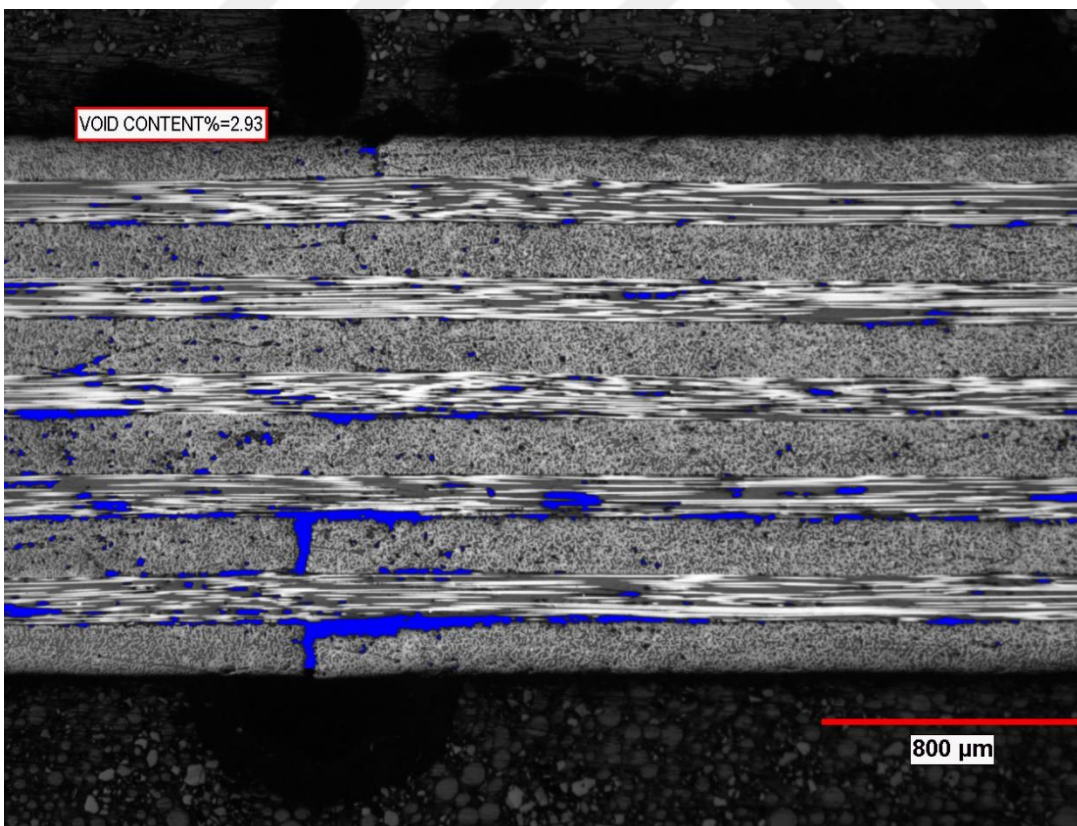
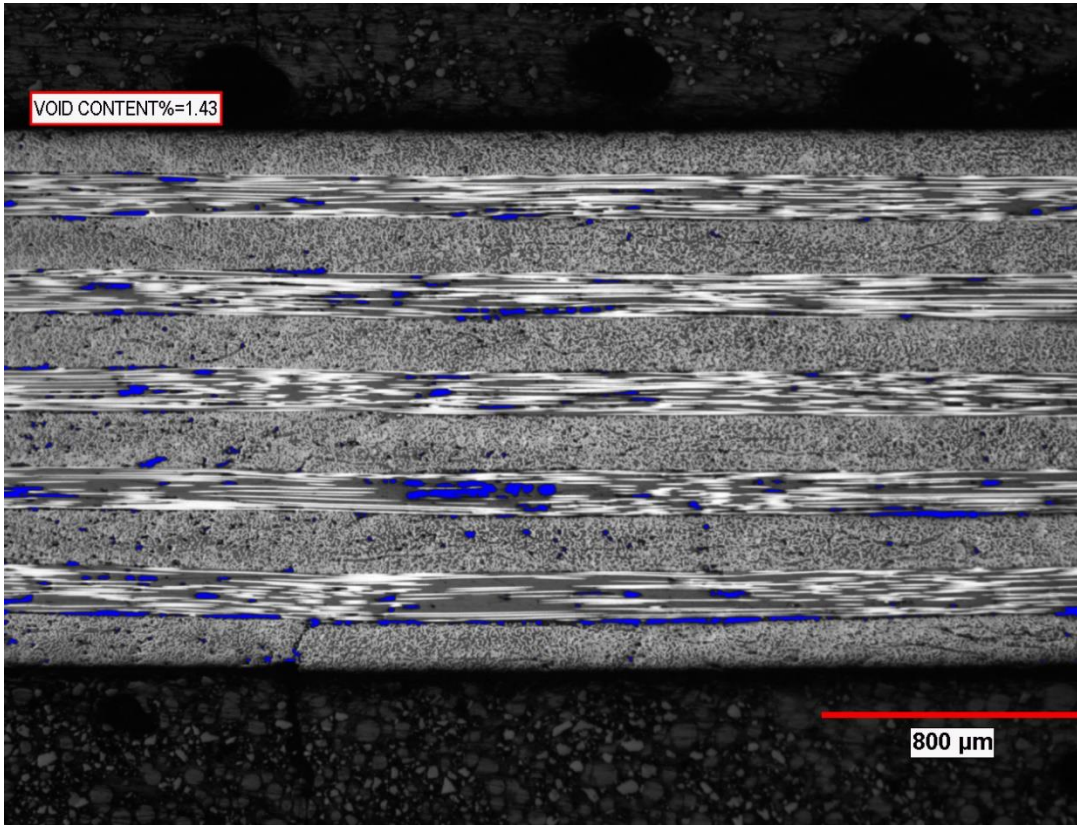
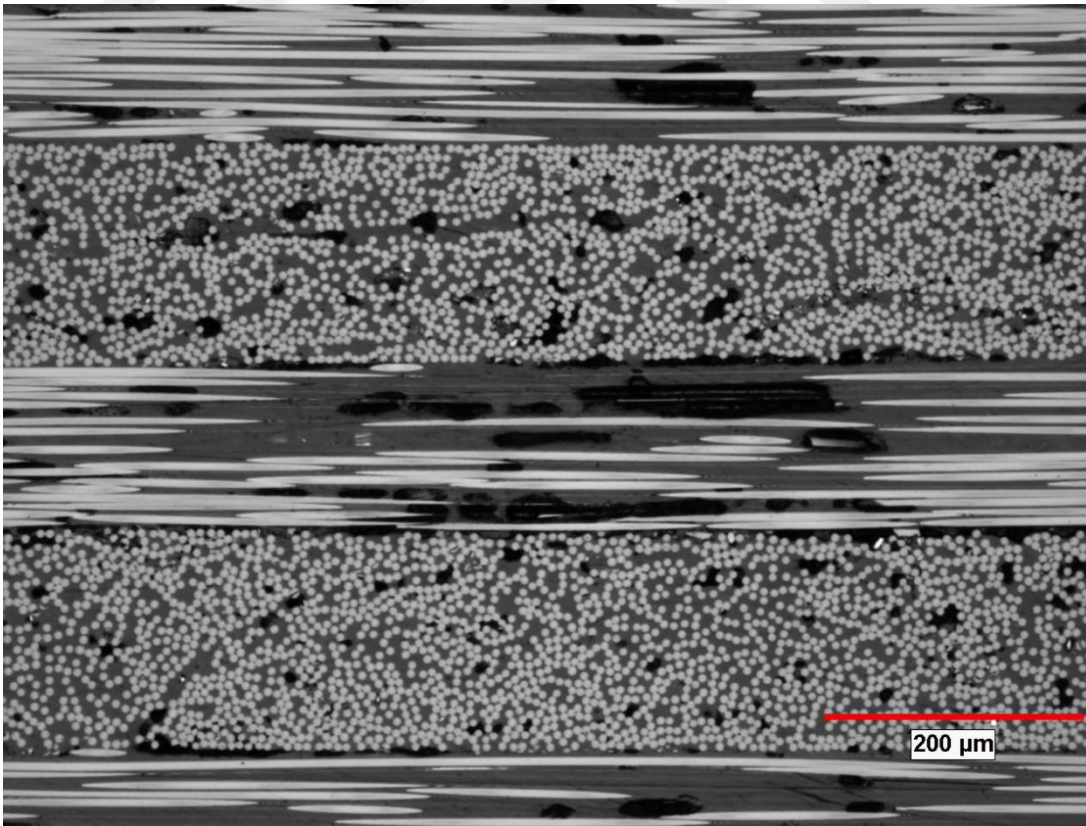
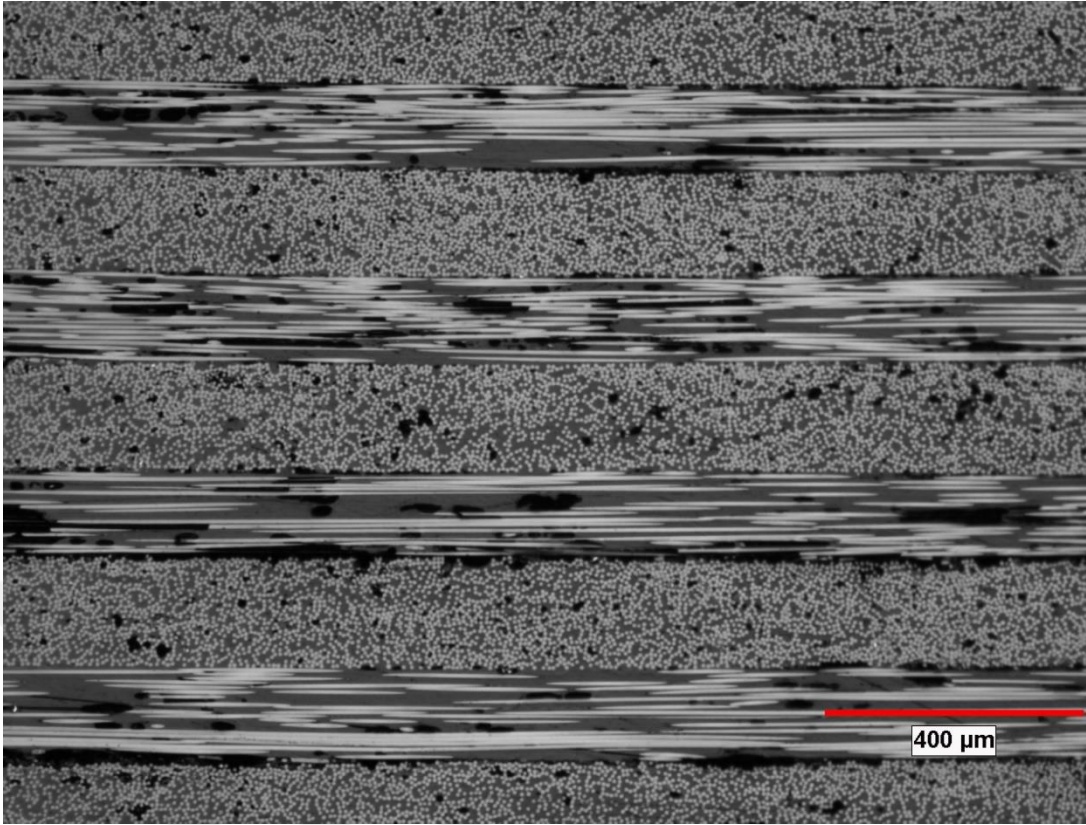


Figure A.1 : High melt -low speed in situ panels microcut results (1-a)







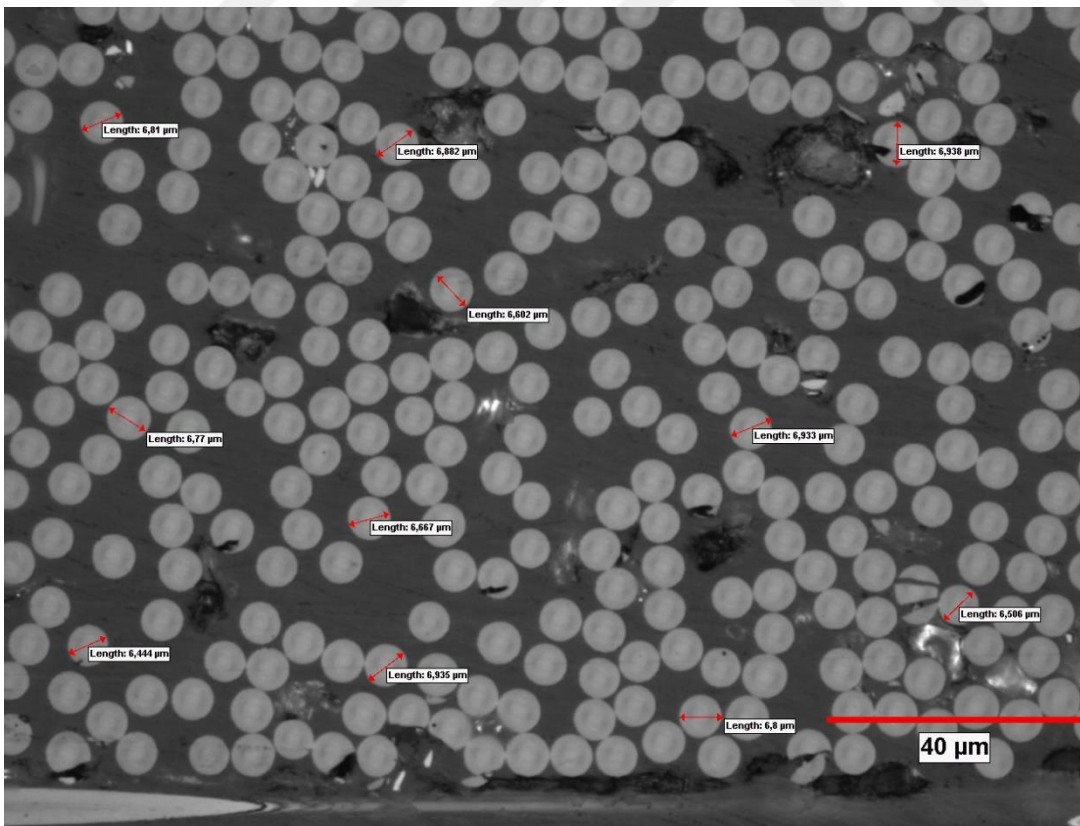
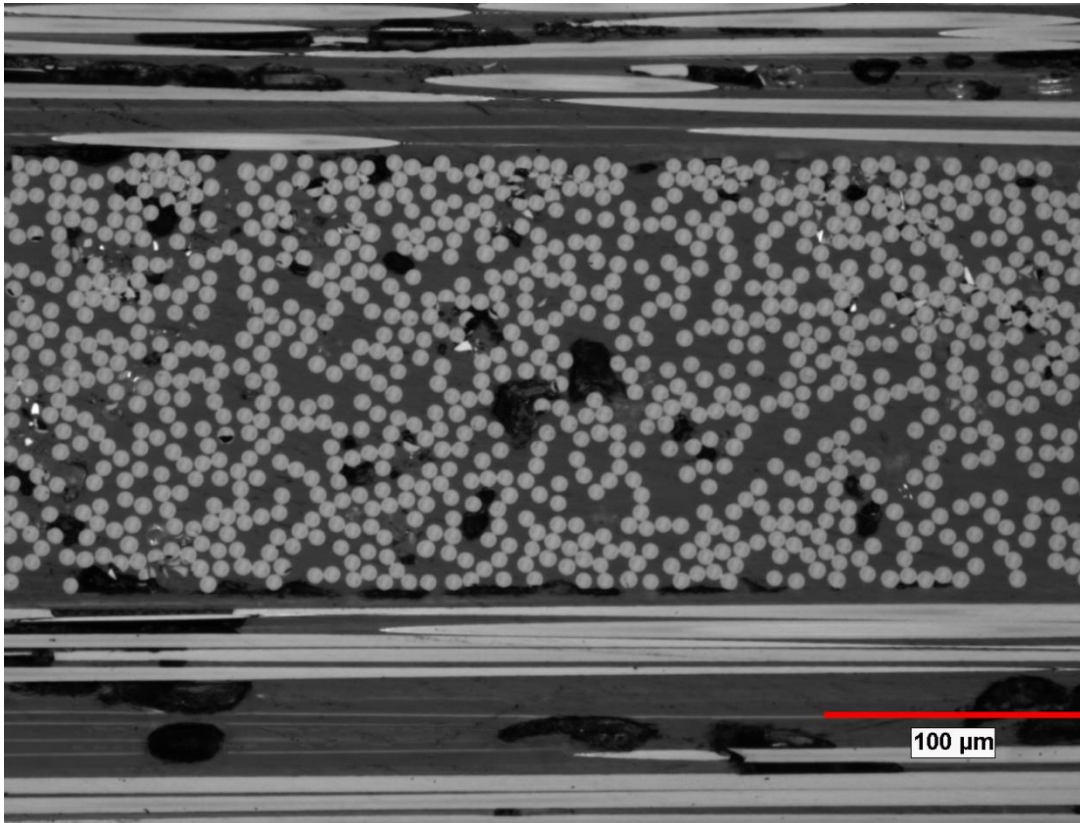
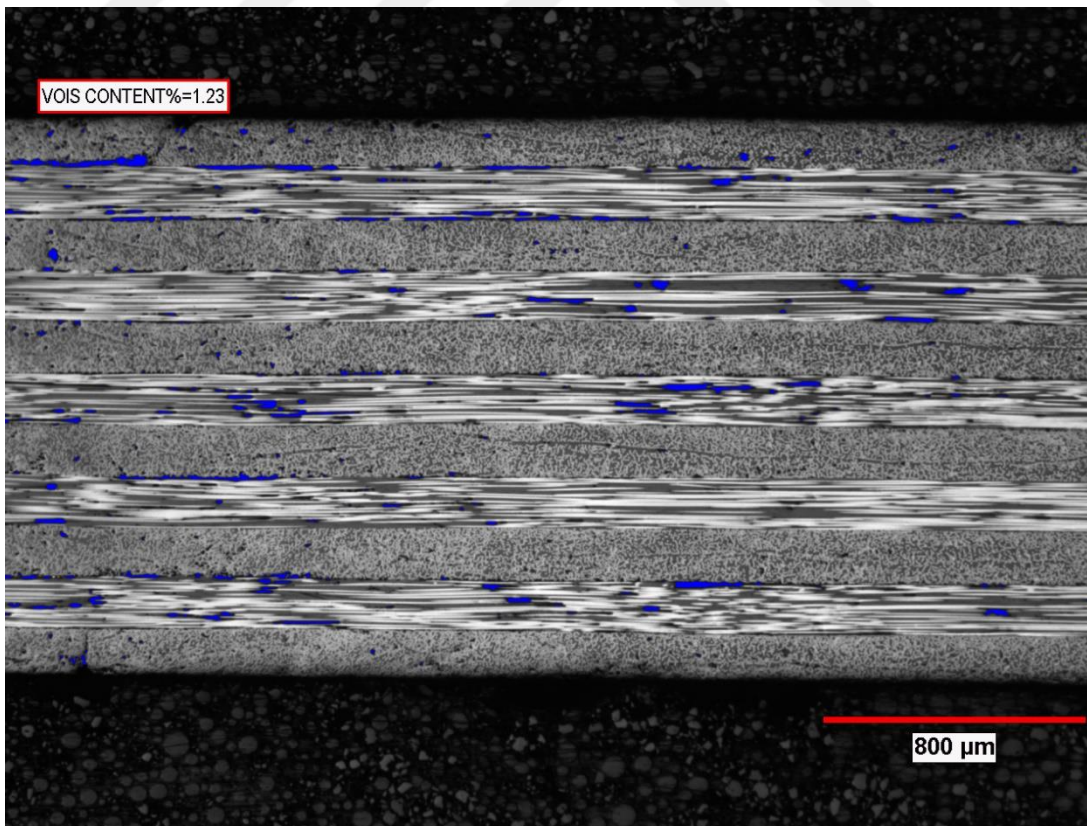
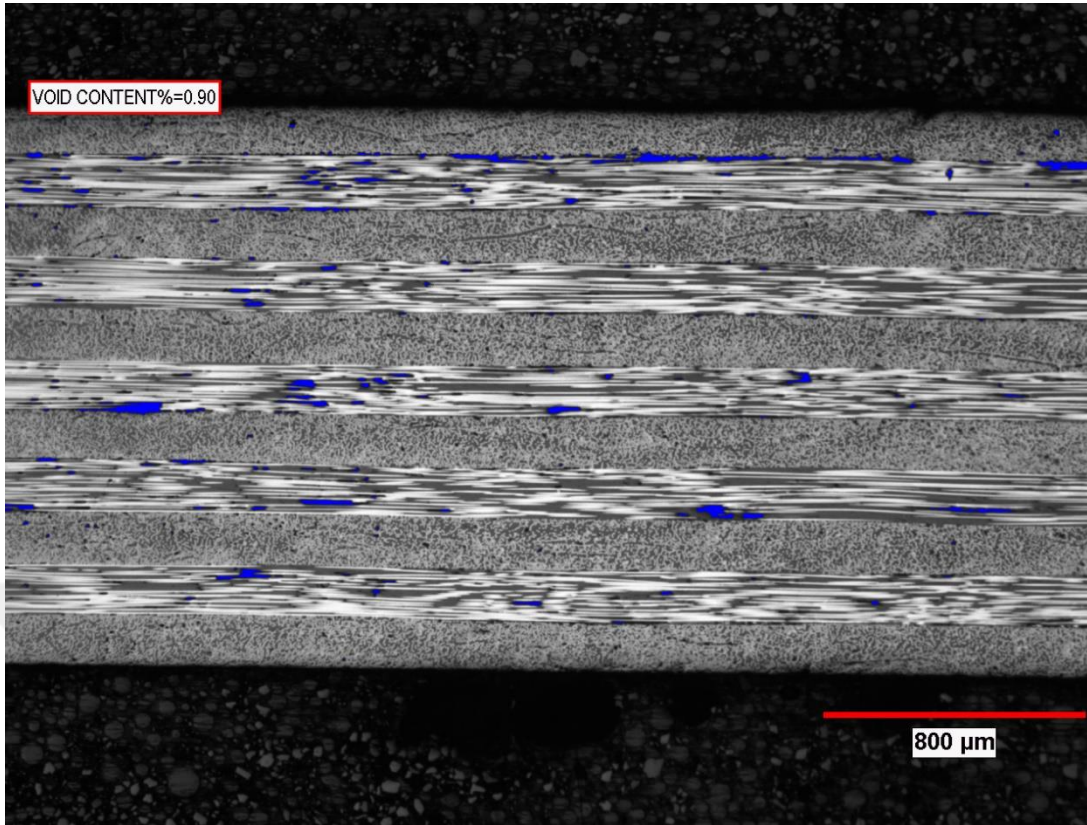
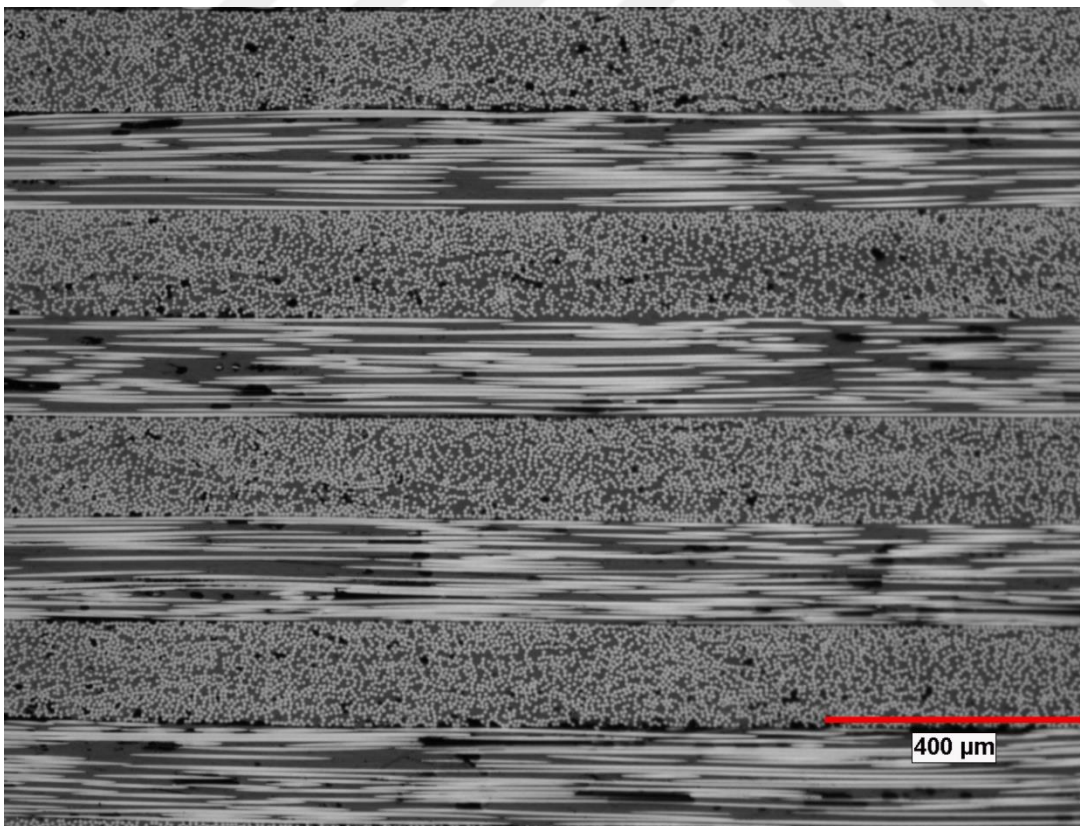
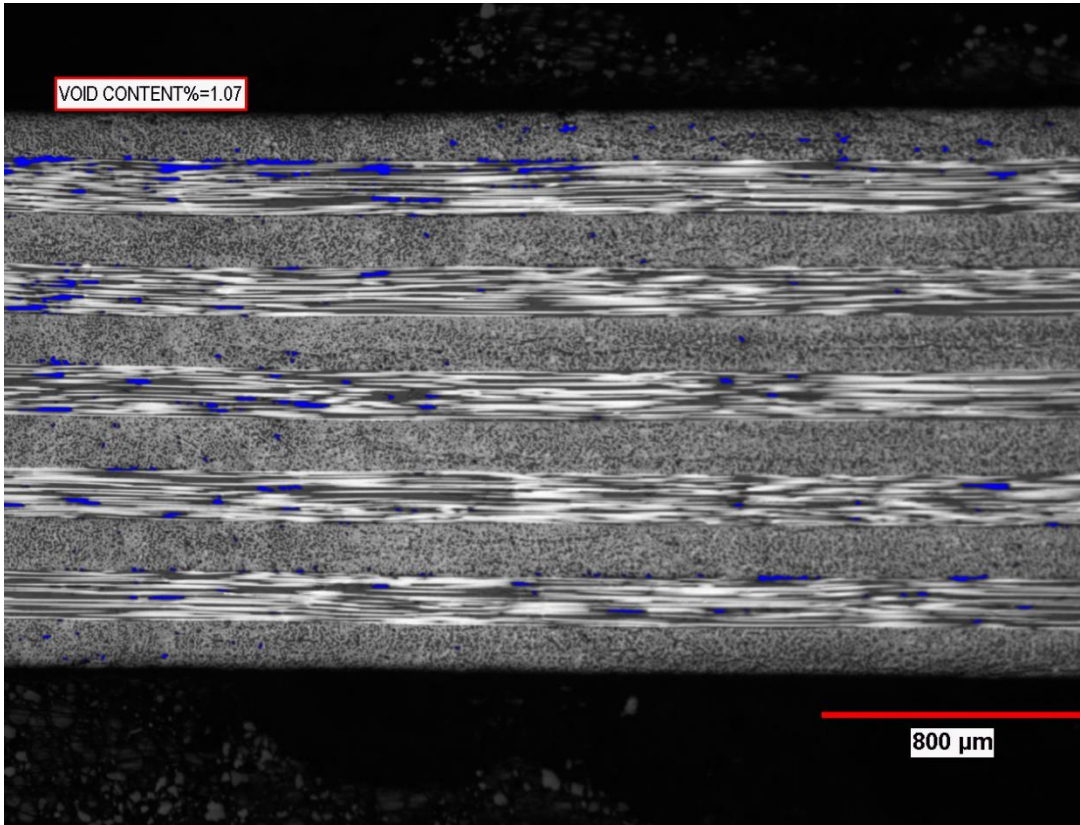
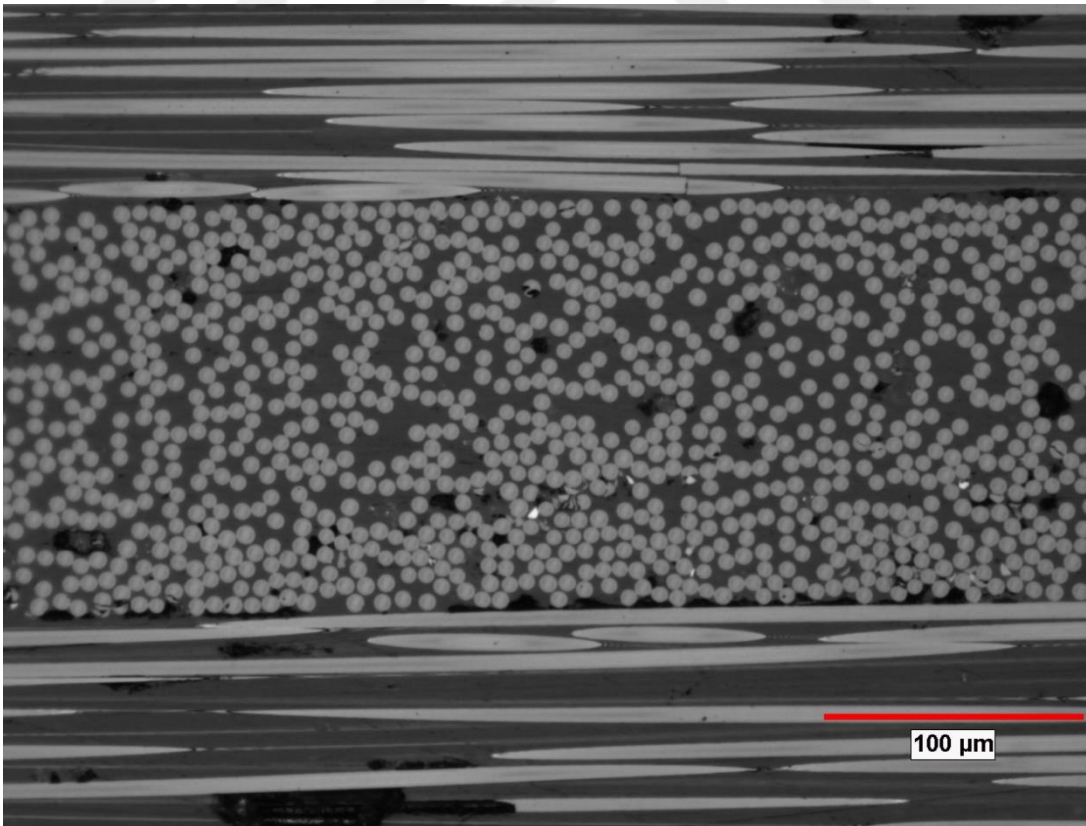
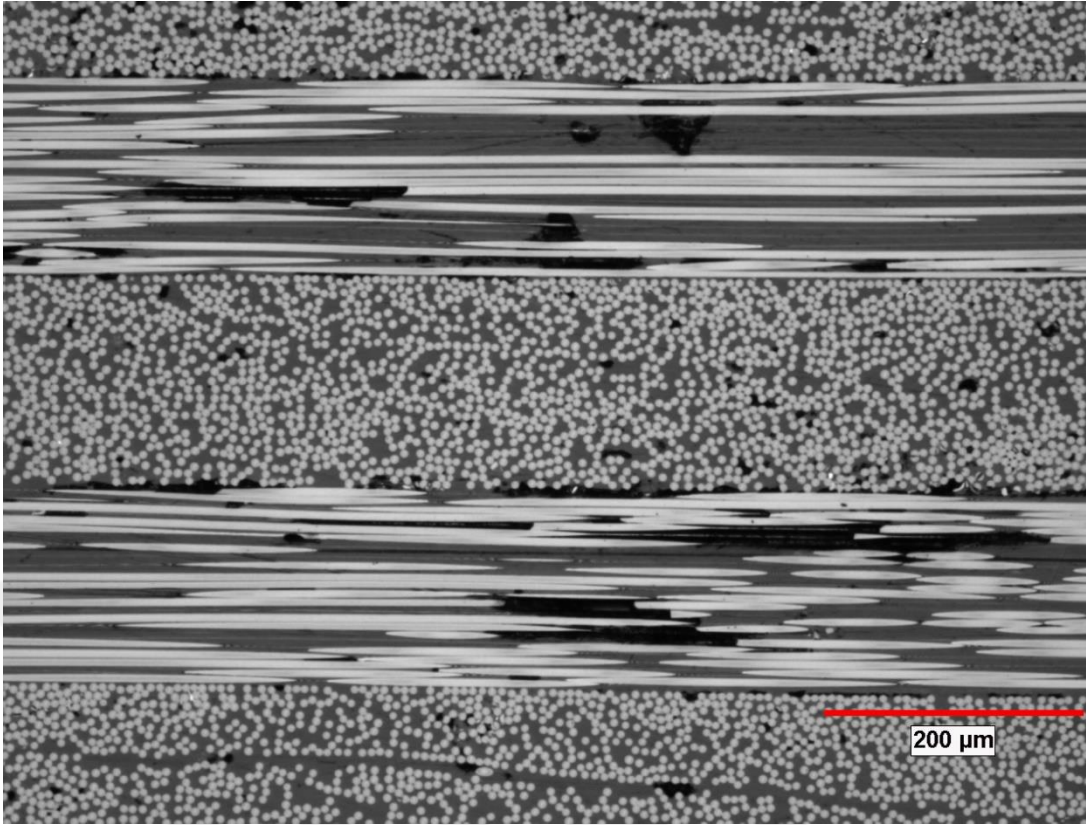


Figure A.2 : High melt -low speed in situ panels microcut results (2-a)







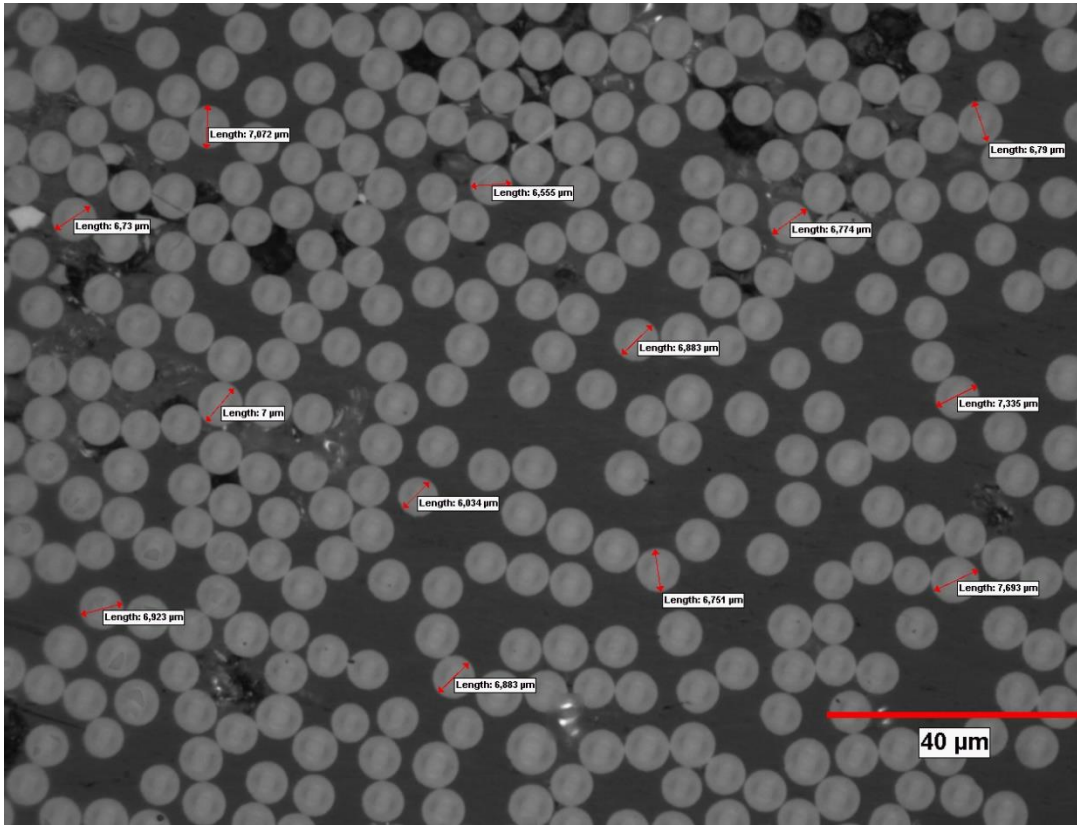
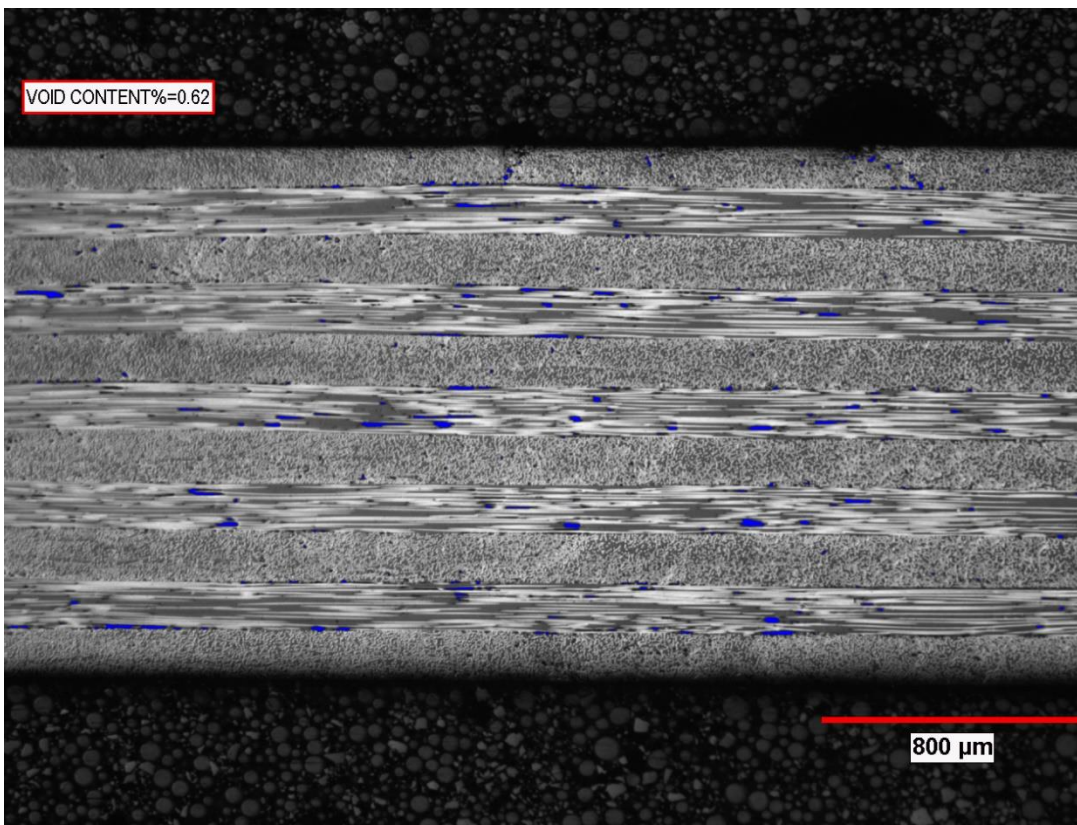
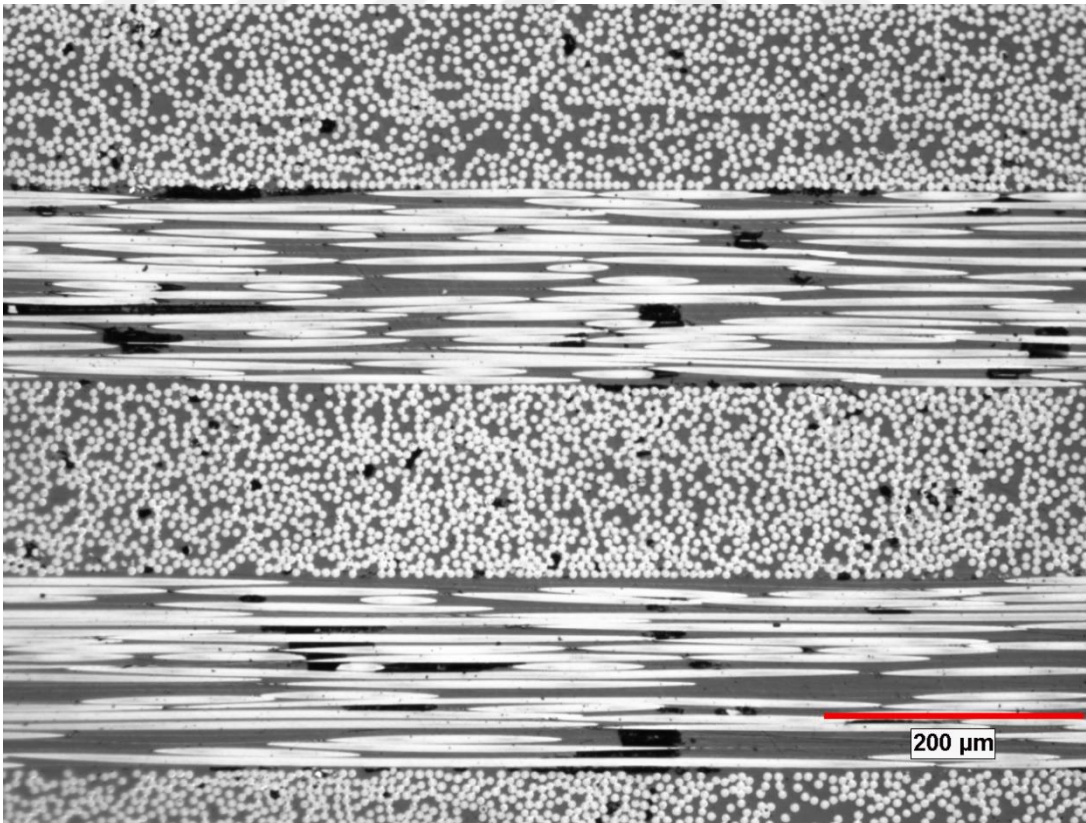
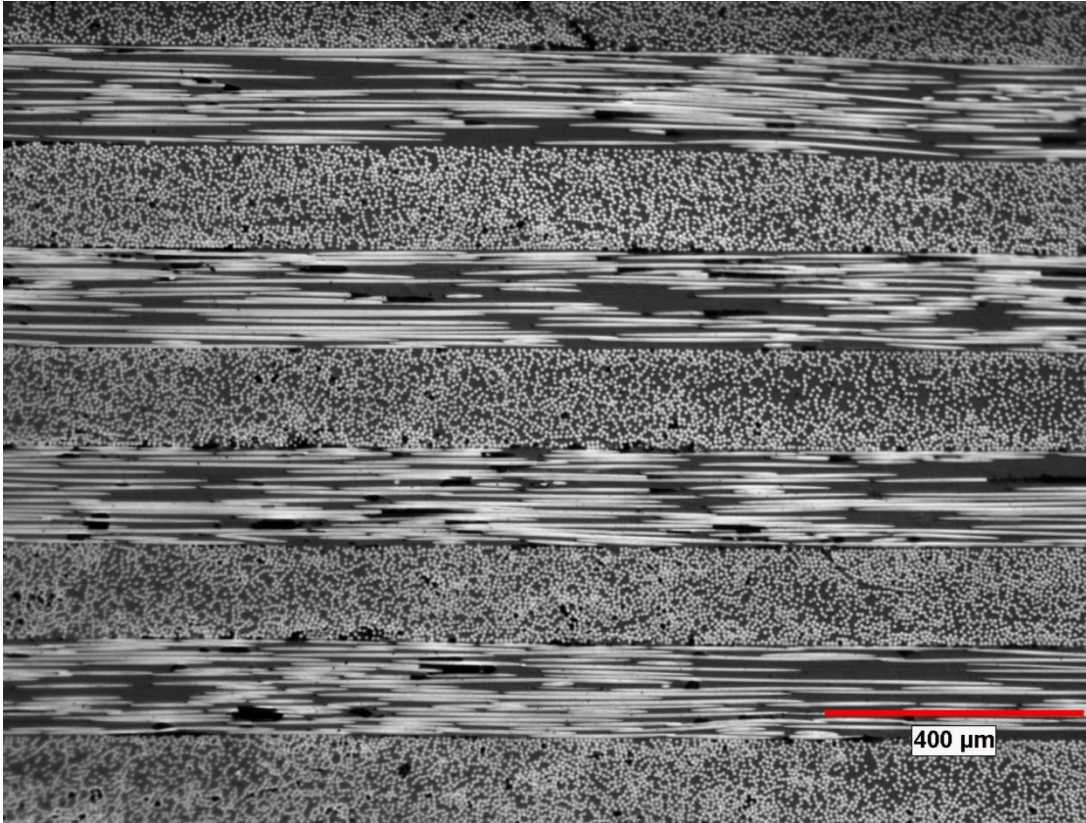


Figure A.3 : High melt -low speed in situ panels microcut results (3-a)





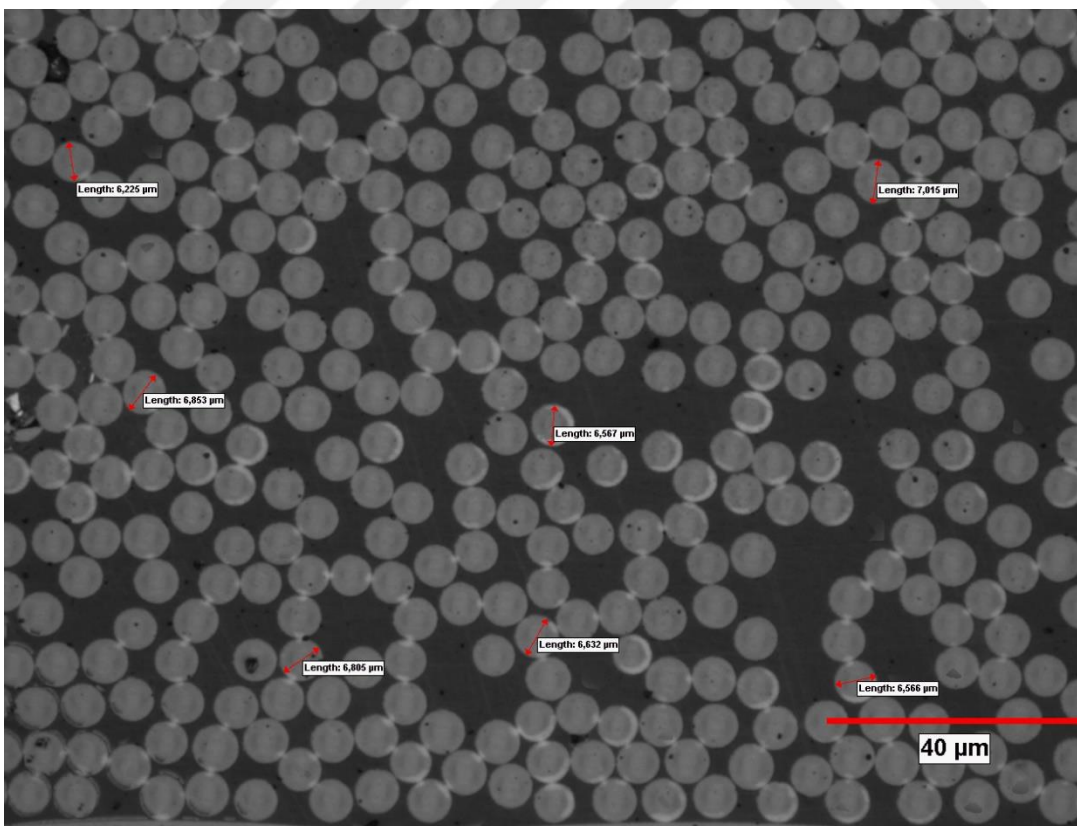
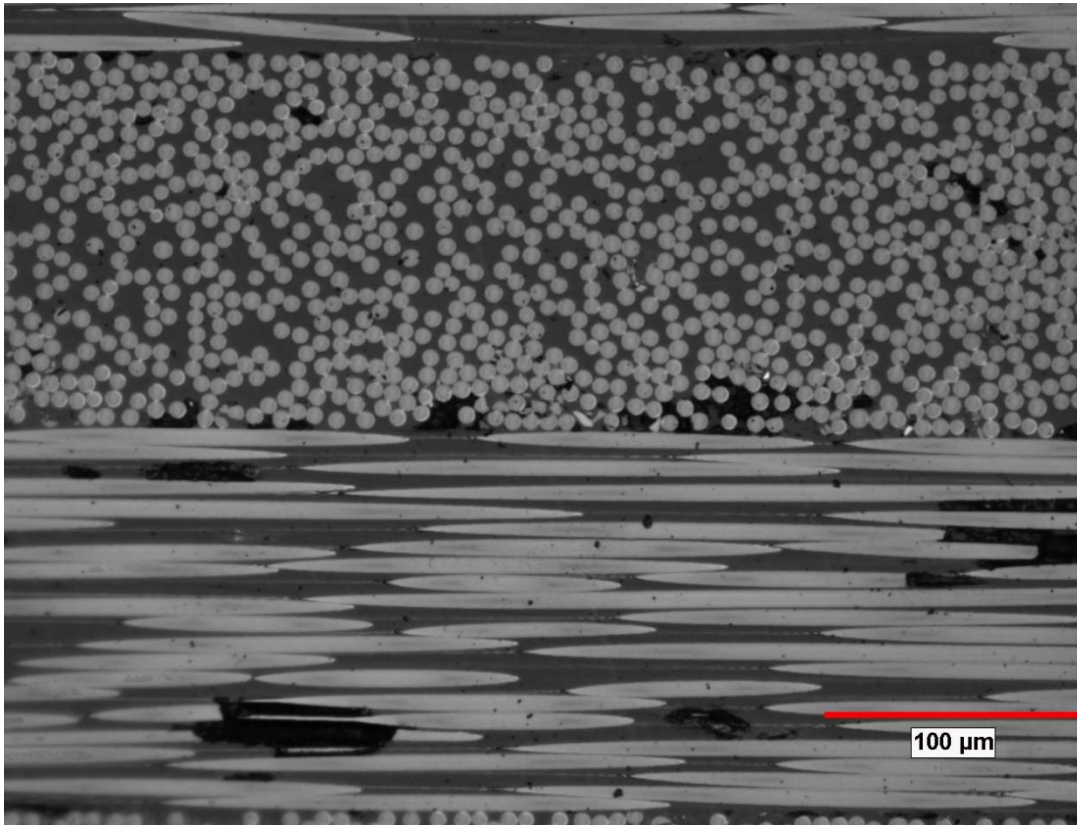
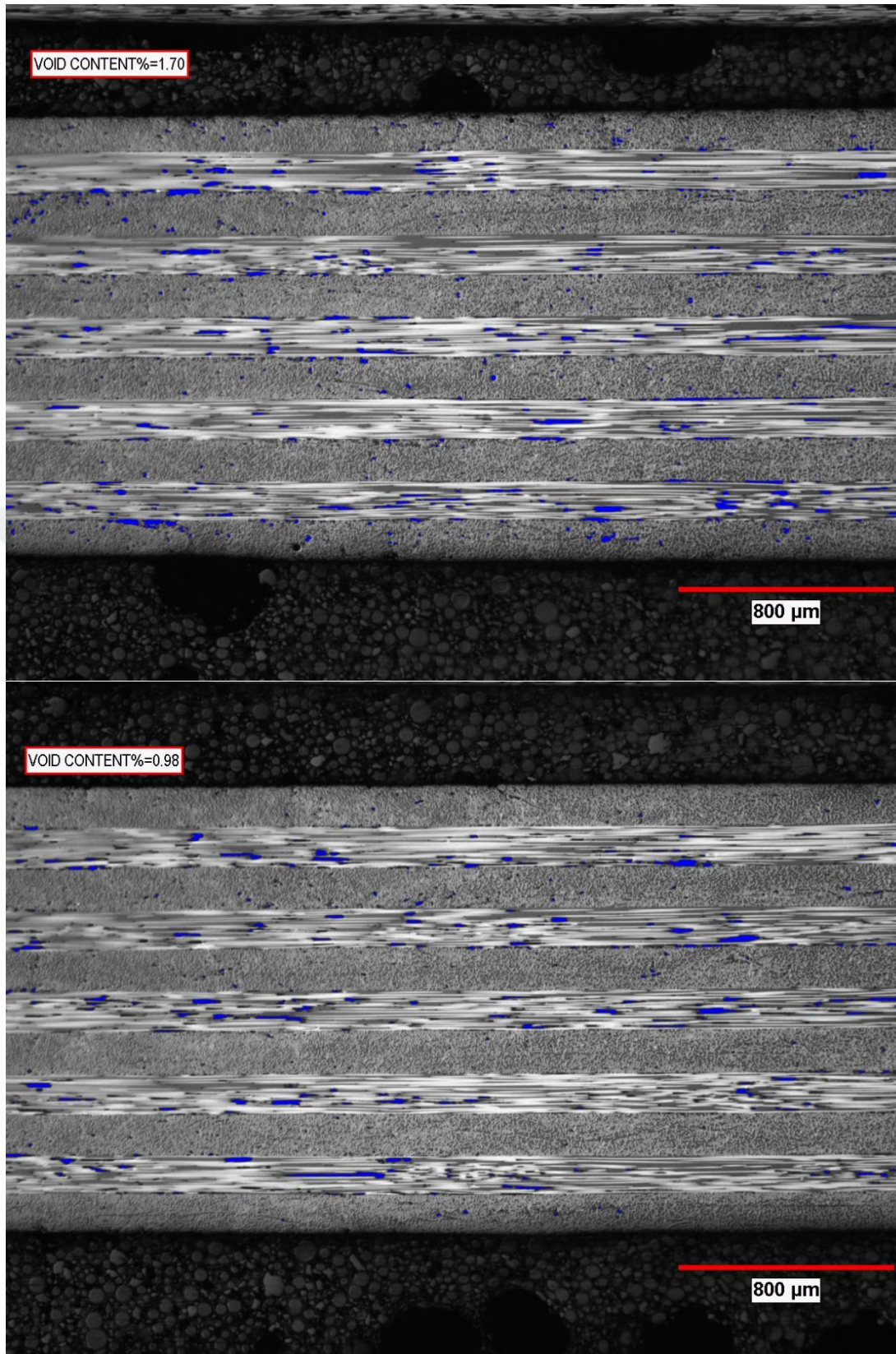
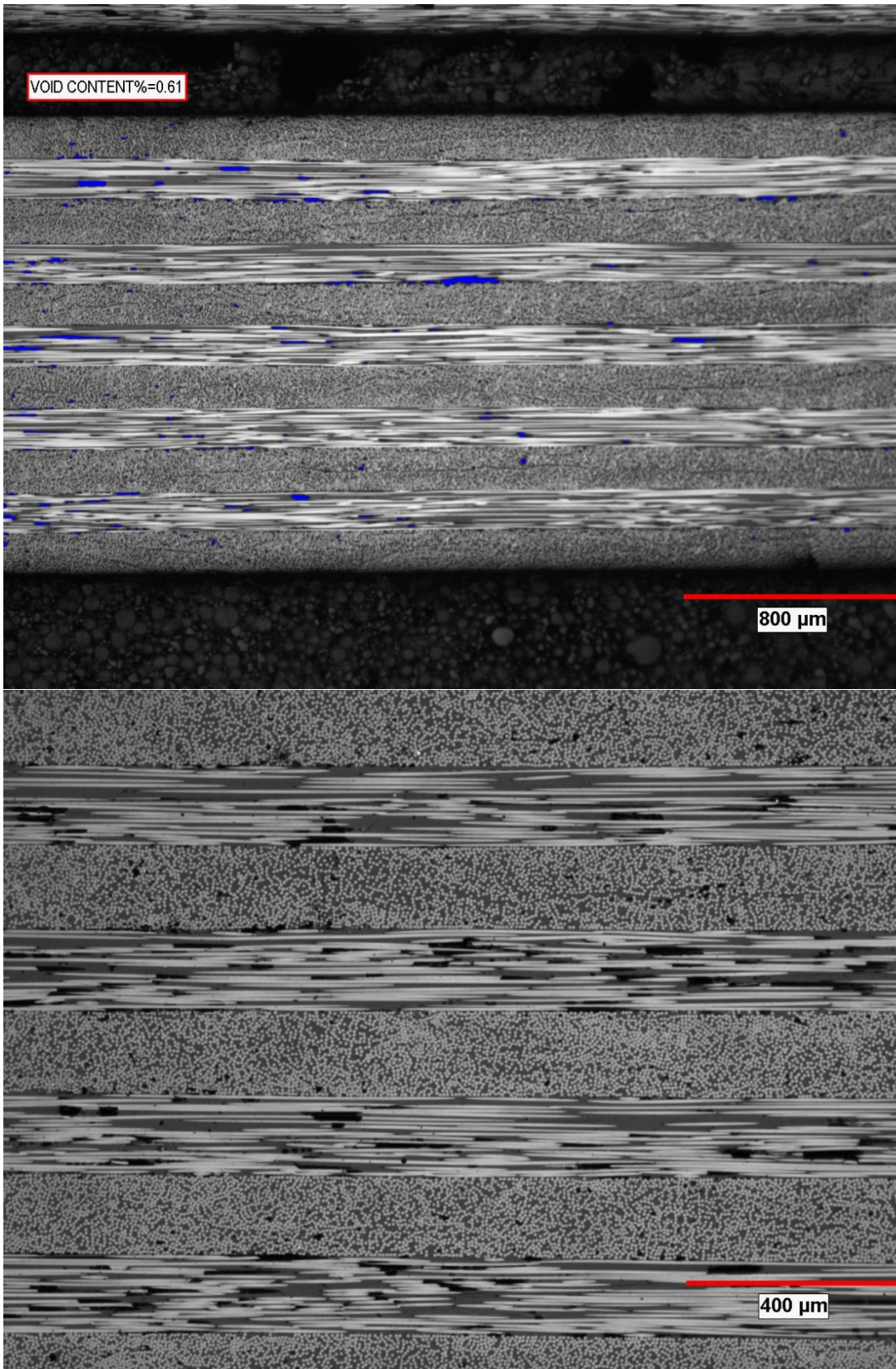
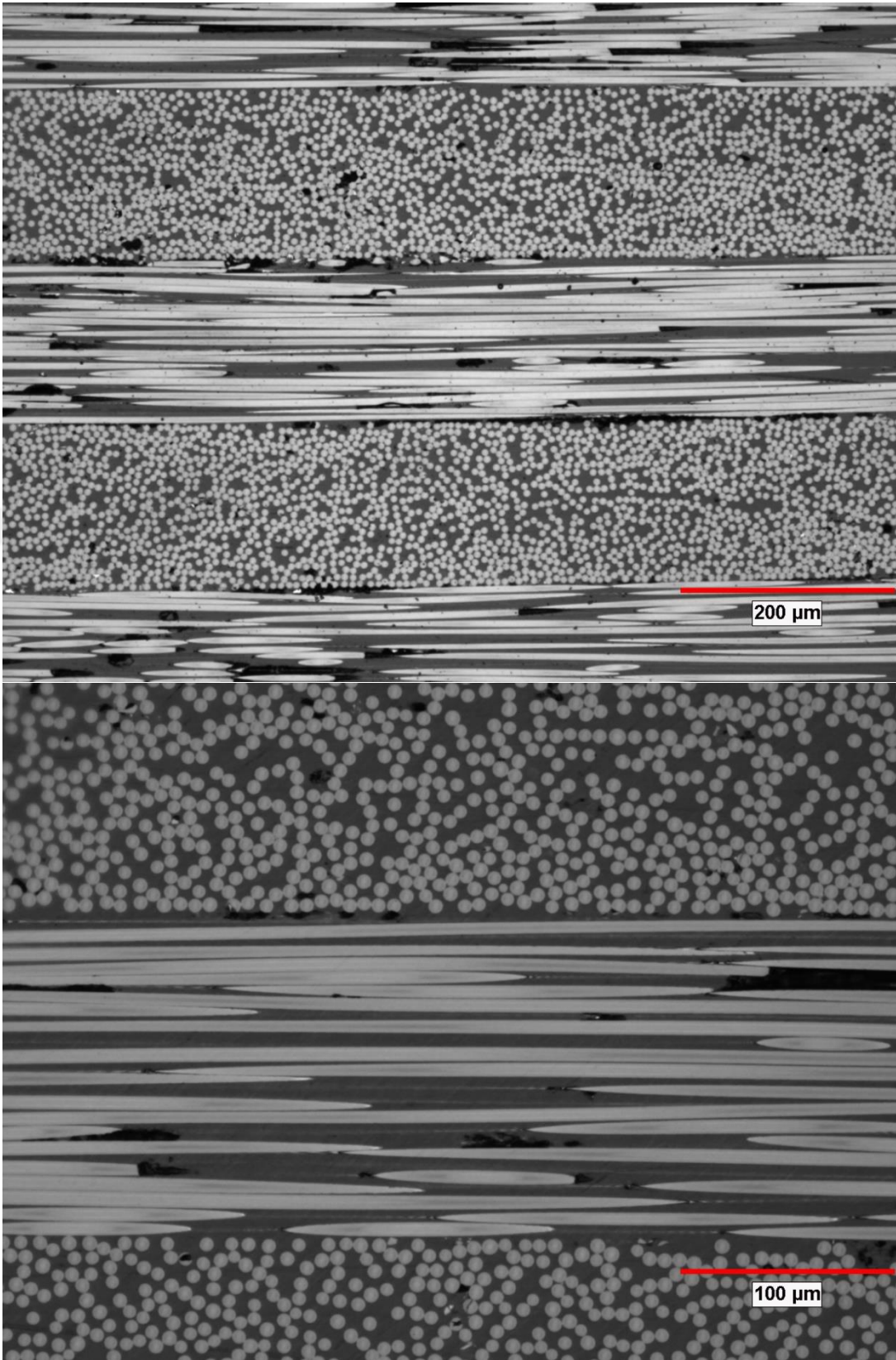


Figure A.4 : High melt-low speed autoclave panels microcut results (1-a)







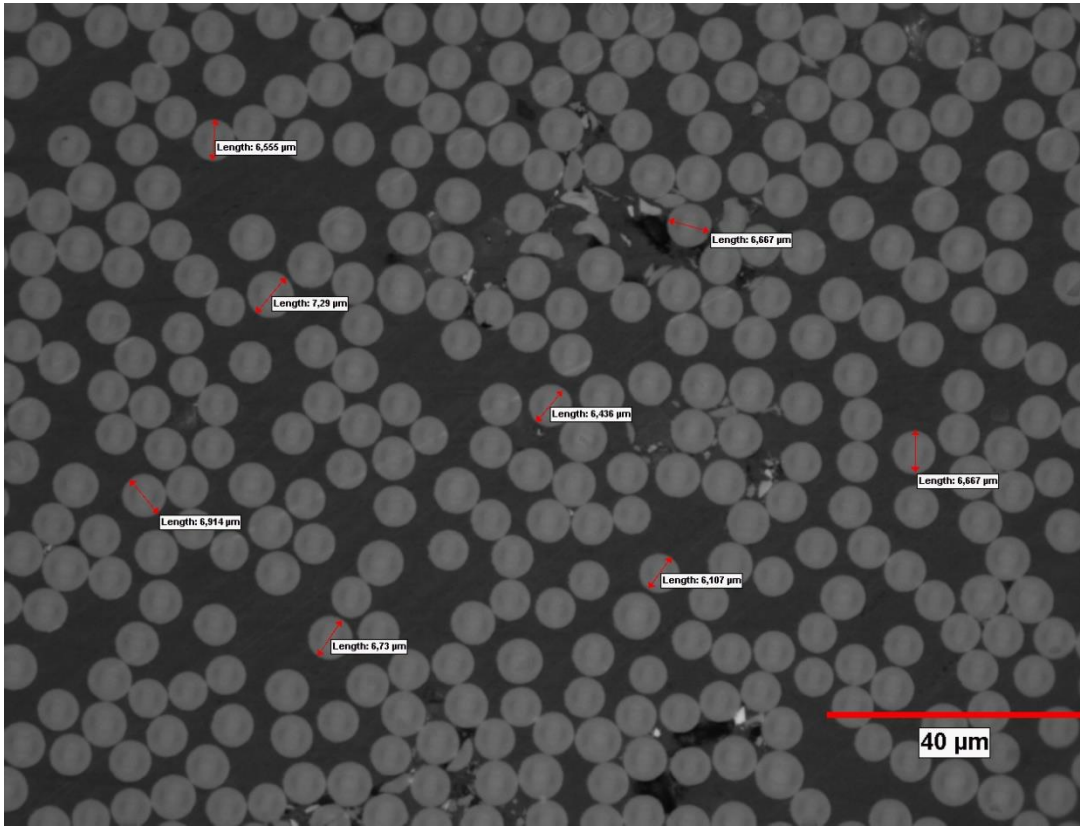
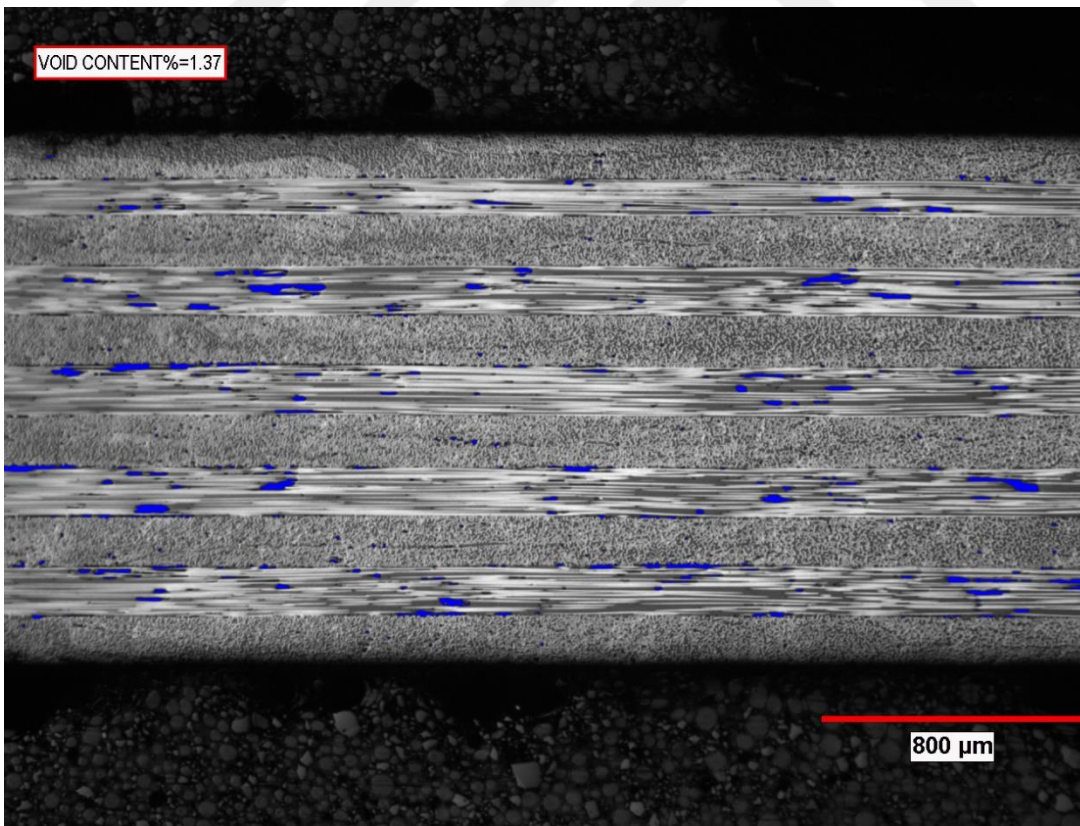
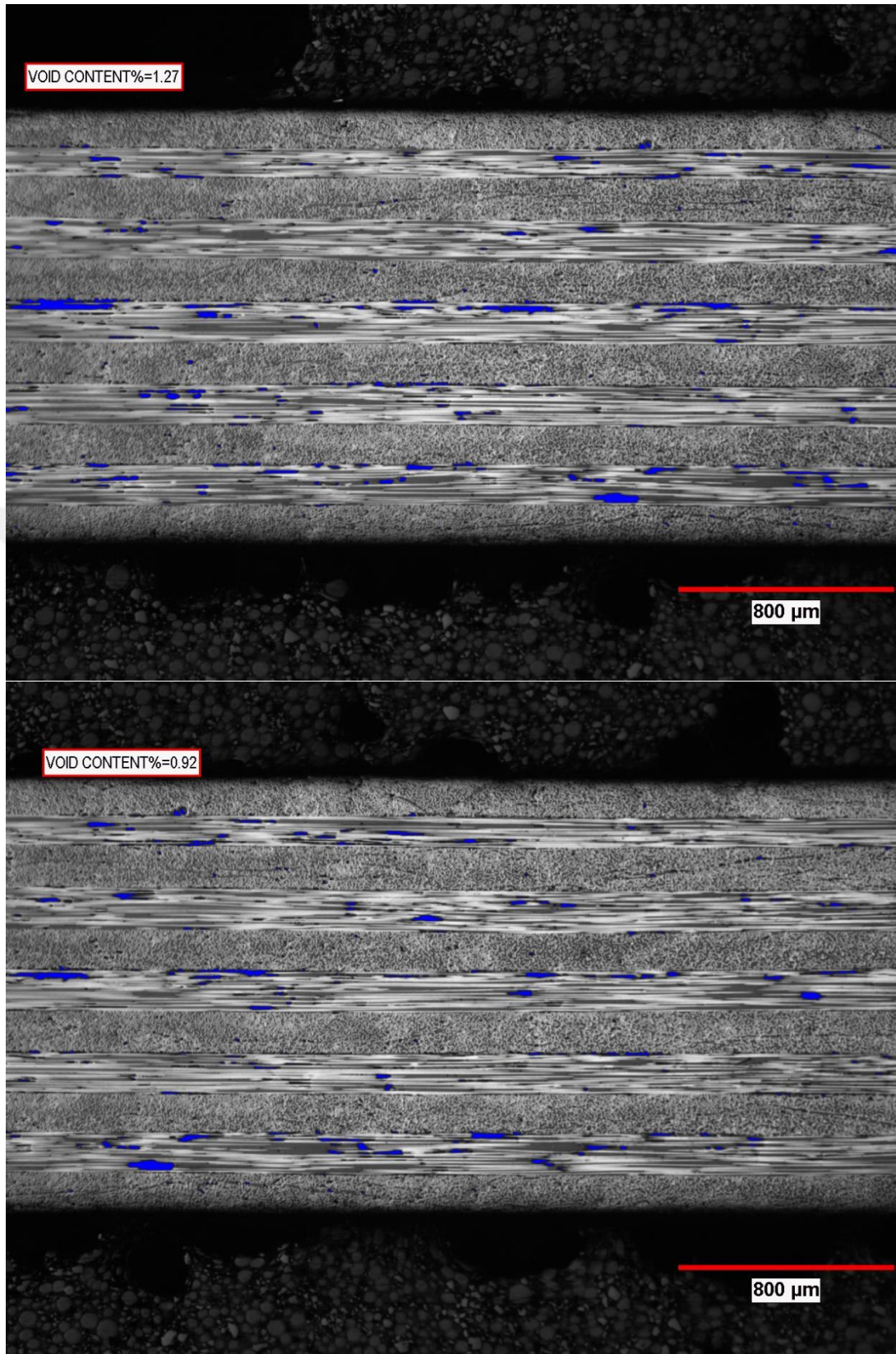
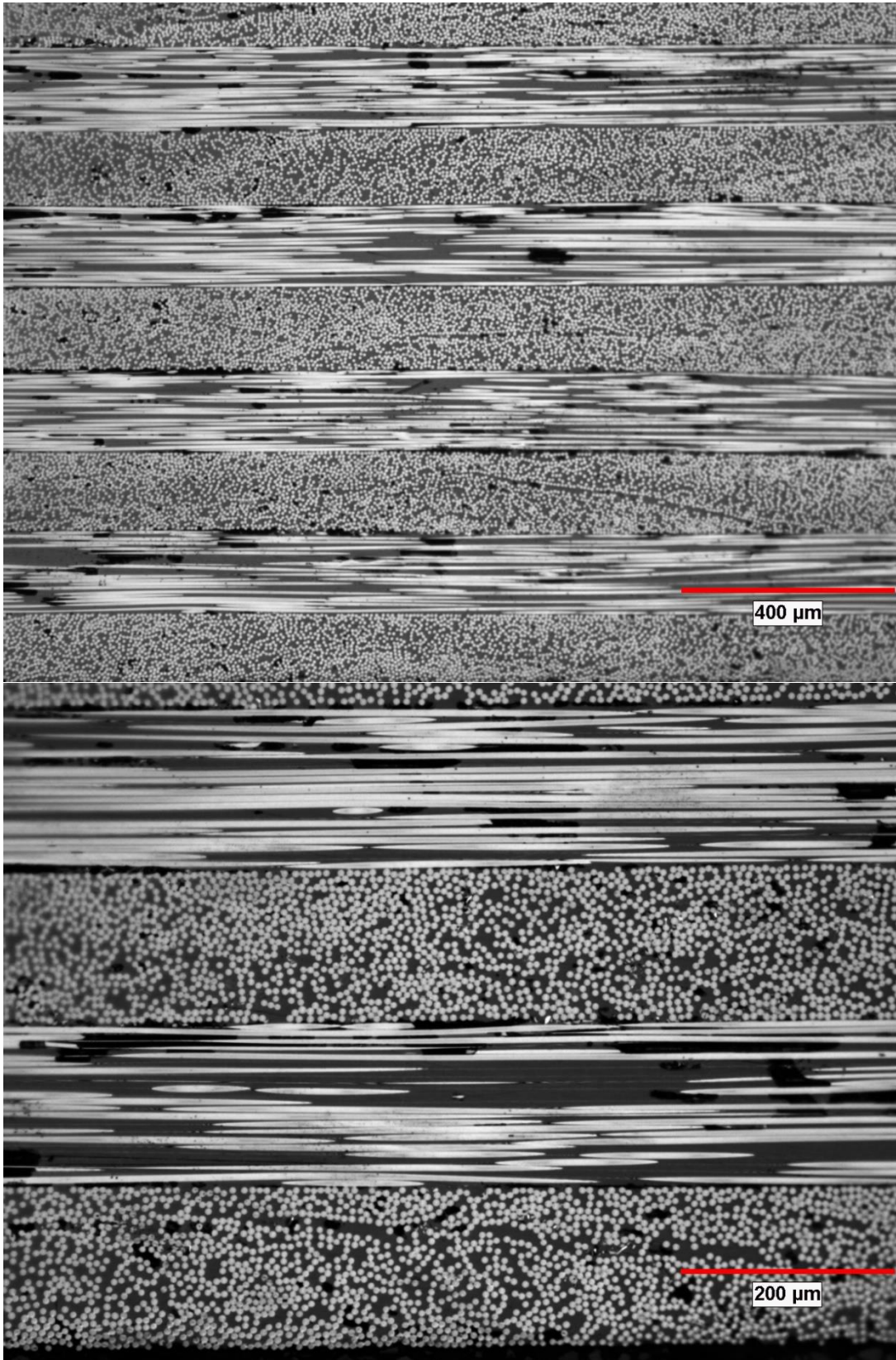


Figure A.5 : High melt-low speed autoclave panels microcut results (2-a)







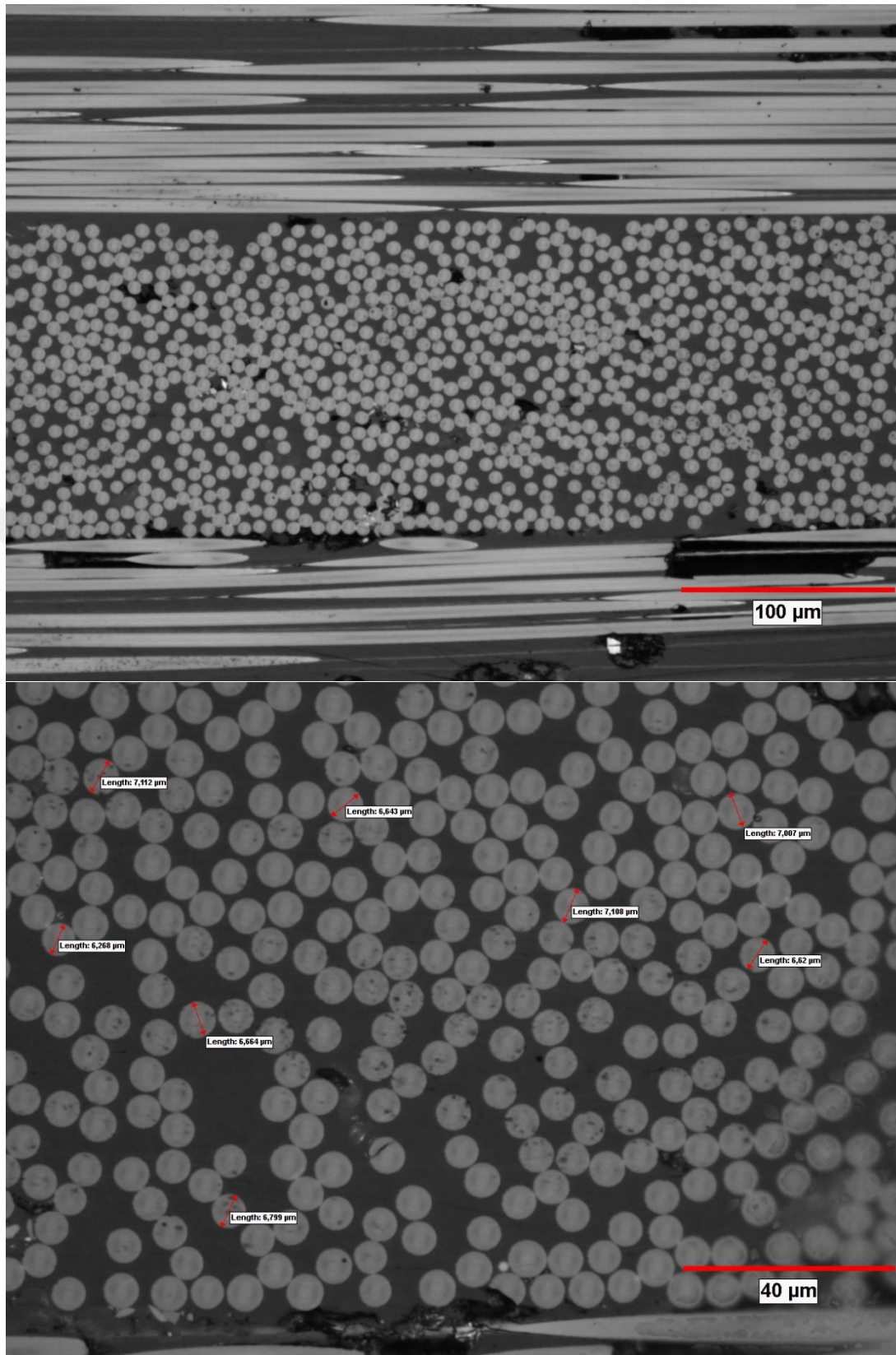


Figure A.6 : High melt-low speed autoclave panels microcut results (3-a)



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