

EXPERIMENTAL INVESTIGATION OF THE FAILURE OF AIR
PLASMA SPRAYED THERMAL BARRIER COATINGS

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ABSTRACT

EXPERIMENTAL INVESTIGATION OF THE FAILURE OF AIR PLASMA SPRAYED THERMAL BARRIER COATINGS

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In this thesis, an experimental method is developed to determine the interfacial fracture toughness of air plasma sprayed thermal barrier coatings. The thesis is composed of three major parts. In the first part, nanoindentation is used to measure the elastic modulus and hardness of the APS TBC specimens composed of three major layers: Inconel 718 substrate, NiCrAlY based metallic bond coat (BC) and YSZ based ceramic top coat (TC). Microscopy, XRD and EDS are used for metallurgical characterization. In the second part, a model material system of bonded Al/PMMA composite beam is used to develop a 4-pt bending test to measure the critical interfacial energy release rate as proposed by Charalambides et al. in conjunction with digital image correlation (DIC) method. In the third part, the properties measured in the first part and the experimental method developed in the second part are used to carry out 4-pt micro-bending tests on an APS TBC system to determine interface toughness for three different coating thicknesses. The failure progress is recorded in-situ using a microscope in conjunction with a DIC system. Failure mechanism is comprised of nucleation and growth of vertical cracks in the TC followed by interfacial crack growth. The critical interfacial energy release rate is measured and is found to increase with thickness. The developed experimental method can be used by jet turbine engine companies to develop standardized quality assessment procedures for coatings.

KEYWORDS: Thermal barrier coating, energy release rate, digital image correlation, nanoindentation, interfacial toughness

ÖZ

HAVA PLAZMA SPREY ISIL BARIYER KAPLAMALARIN KIRILMASININ DENEYSEL İNCELENMESİ

Kütükoğlu, Başar

Yüksek Lisans, Havacılık ve Uzay Mühendisliği Bölümü

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Bu teze konu olan çalışmada hava plazma sprej ısı bariyer kaplamalarında (HSP IBK) arayüz kırılma tokluğunun ölçülebilmesi için deneysel bir metot geliştirilmiştir. Tez üç ana başlıktan oluşmaktadır. İlk kısımda nanoindentasyon ile üç ana katmandan oluşan HSP IBK numunelerinde elastik modül ve sertlik ölçümleri yapılmıştır. Bu üç ana tabaka: Inconel 718, metal temelli NiCrAlY astar kaplama (AK), seramik temelli YSZ üst kaplamadır (ÜK). Ayrıca, bu üç ana katmandaki metalürjik özellikler mikroskop, XRD ve EDS yöntemleri kullanılarak karakterize edilmiştir. İkinci kısımda, Charalambides ve arkadaşlarının önerdiği şekilde birbirine yapıştırılmış Al/PMMA kompozit bar ve dijital görüntü korelasyonu (DIC) yöntemi kullanılarak arayüzde kritik enerji bırakma nispeti ölçümü yapılabilecek bir yöntem geliştirilmiştir. Üçüncü kısımda, ilk kısımda ölçülen özellikler ile ikinci kısımda geliştirilen yöntem kullanılarak üç farklı kalınlıkta üretilen HSP IBK numunelerinde mikro 4 nokta bükme testleri gerçekleştirilmiştir. Kırılma mekanizması mikroskop ve DIC sistemi yardımıyla yerinde kaydedilmiştir. Kırılmanın önce Ükda dikine ilerlemesi ve ardında da arayüzde ilerlemesi gözlemlenmiştir. Son olarak, arayüz için kritik enerji bırakma nispeti değerleri hesaplanmış ve artan ÜK kalınlığı ile arttığı gözlemlenmiştir. Bu tezde geliştirilen deneysel yöntem IBK numunelerinde mukavemetinin ölçülebilmesi için gerekli standart yöntemlerin oluşması açısından iyi bir adaydır. Motor şirketleri bu yöntemi AR-GE ve kalite kontrol sistemlerinin geliştirilmesinde kullanabilir.

ANAHTAR KELİMELER: Isıl bariyer kaplama, enerji bırakma nispeti, dijital görüntü korelasyonu, nanoindentasyon, arayüz tokluğu

To my beloved family,

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LIST OF SYMBOLS

SYMBOLS

$A(h)$	Area function
a	Crack length
C	Total compliance
C_f	Compliance of testing frame
C_i	Area function constants ($i=1,2,3,\dots,8$)
C_s	Compliance of specimen
E	Modulus of elasticity
E_i	Modulus of elasticity of an indenter tip
E_r	Reduced modulus
E_s	Modulus of elasticity of substrate
EIT	Indentation modulus
G_i	Shear modulus of elasticity corresponding to materials ($i=1,2$), respectively
G, G_{ss}	Energy release rate, steady-state energy release rate
G_c	Critical energy release rate
H	Hardness
HIT	Indentation hardness
h	Penetration depth
h_c	Contact depth
h_f	Depth of residual indentation impression
h_{max}	Maximum penetration depth
h_s	Surface displacement in the vicinity of indentation
h_1, h_2, h_c	Layer thickness
K, K_i	Complex stress intensity factor in mode ($i=I, II$), respectively
K_c	Critical stress intensity factor
$ K $	Scalar of complex stress intensity factor
$\bar{K}$	Complex conjugate of stress intensity factor
m	Indenter shape constant

M	Weibull constant
M	Moment
N	Number of observations
i	Observation index
P	Load
$P_{\max}$	Maximum applied load
p	Survival probability
r	Radial distance from the crack tip
S	Contact stiffness
U	Strain energy
x	Measured parameter
x_0	Characteristic value of measured parameters
α	Indenter shape constant
ϵ	Bi-material constant
Δu_x	Crack opening displacement in x-axis
Δu_y	Crack opening displacement in y-axis
$\partial()/\partial()$	Partial differential
ν	Poisson's ratio
ν_i	Poisson's ratio of an indenter tip
ν_s	Poisson's ratio of substrate
ϕ	Phase angle of crack opening displacements
ψ	Phase angle of complex stress intensity factor

ABBREVIATION

APS	Air Plasma Spray
BC	Bond Coat
BF	Bright Field Microscopy
COD	Crack opening displacement
DF	Dark Field Microscopy
EB-PVD	Electron Beam Physical Vapor Deposition
EDS	Energy Dispersive X-Ray Spectroscopy
EDX	Energy Dispersive X-Ray Spectroscopy
NiCrAlY	Nickel-Chromium-Aluminum-Yttrium
S	Substrate
SEM	Scanning Electron Microscopy
TBC	Thermal Barrier Coating
TC	Top Coat
TEI	Turkish Engine Industry
TGO	Thermally Grown Oxide
YSZ	Yttrium Stabilized Zirconia

CHAPTER 1

INTRODUCTION

In the first part of this chapter, the sequence of events that encourage the industry focus on *thermal barrier coatings* are analyzed in parallel with a brief history of aviation. Next, cooling methods used in propulsion systems are depicted and as a part of these, the anatomy of thermal barrier coatings are examined. Later, failure modes of a thermal barrier coating are discussed. Then, the fundamentals of failure are correlated with the underlying problem: “*Indefinite Durability and Lack of Quantifiable Evaluation of Thermal Barrier Coatings*”. At the end of this chapter, literature review of the related to experimental studies are presented in a logical order. Some of the literature related of experiments and methodology are presented under their related chapters.

1.1. Modern Challenge: *Green Aircraft*

Mankind has dreamt of flying since the beginning of time. Unfortunately, they had to wait until the end of 19th century to see this lucid dream became real. In the days of the first flight of man, flying was nothing but a balance between the weight and lift. The first 50 years were spent to set the fundamentals of aeronautics and to invent new ways and vehicles for flying. The invention of propulsion systems altered the way of thinking, it overhauled the materials that aircraft made of. Replacing wood and textile with sheet metal changed the concepts and limits of flying. Flying was no longer a rich men hobby, but it was a new industry: *aviation*. Each innovation in aviation claimed better engineering and thereby better conditions of flight. After 50 years of research and the booming decade of aviation in the 1930s, flying formed something that we are very familiar with today. The sound barrier was penetrated and heavy lift airplanes were built. In the pursuit of best aircraft, countless experimental researches were

conducted on the aerodynamics, propulsion, and material science. Mankind increased its experience as a practitioner and engineer and built better aircraft.

In the first 100 years, the best meant nothing more than a show of power with speed, dimension and so on. As a result of the oil crisis in the 1970s and 1990, fuel consumption of aircraft become a significant factor in both military and commercial aviation [44, 79]. Engineers had spent more than 30 years to develop powerful aircraft, but they were in danger to become non-efficient and non-economical [55]. The idea of overdosing aircraft by fuel consumption just to gain more power, space, range or speed ended dramatically. The second century of flight will be dominated by whom thinks more of performance, efficiency and green together. Indeed, today, the best flight will be the cheapest flight while claiming the best both on conditions and time [51]. However, the ferocious demand on power remains. Thus, engineering needs more on aerodynamics, material and propulsion not just to build more powerful and greener aircraft but to keep going innovative. Today, development of a green aircraft that consumes less fuel while generates more power every day is the ultimate challenge in engineering.

1.2. Propulsion and *Green Aircraft*

In the concern of propulsion science, conceptually, it is very simple to harvest more power and more efficiency from an engine: *increase the inlet air, increase the burning quality and exploit the exhaust energy* [16]. Practically, increasing the burning temperature of fuel would increase the burning quality and create an extra exhaust energy for turbine. Therefore, increasing the fuel burning temperature would both give more power per unit fuel and decrease the fuel consumption of an aircraft for a specified power. However, exposing combustion chamber and turbine to elevated temperatures arouse an additional cooling need in these stages if parts start working above their melting temperatures [68]. Today, a typical combustion chamber is exposed to 1400 °C during the operation that is far more than the melting temperature of structural parts.

The idea of *thermal barrier coating* is basically coating structural parts by a ceramic powder to insulate the structural parts from hot air and ensure them not to melt during operation. Thermal barrier coatings impact the propulsion science and shifted the limits on allowable gas temperatures in the combustion chamber [Fig. 1.2.1]. At that

point, thermal barrier coatings occupy a significant space in the field of aviation to accomplish the historical motivation: *green aircraft*. Ceramic coatings are unpredictable in service because of their brittleness. Furthermore, applications are completely based on trial and error experience and after-service part investigations. Eventually, the mechanical durability of a thermal barrier coating become a primary issue in design. Today, there is still a lack of evaluation of thermal barrier coatings via testing before flight. As a result, the desire of cost-efficient, powerful and green flight leads us to develop new methods to evaluate TBC systems.

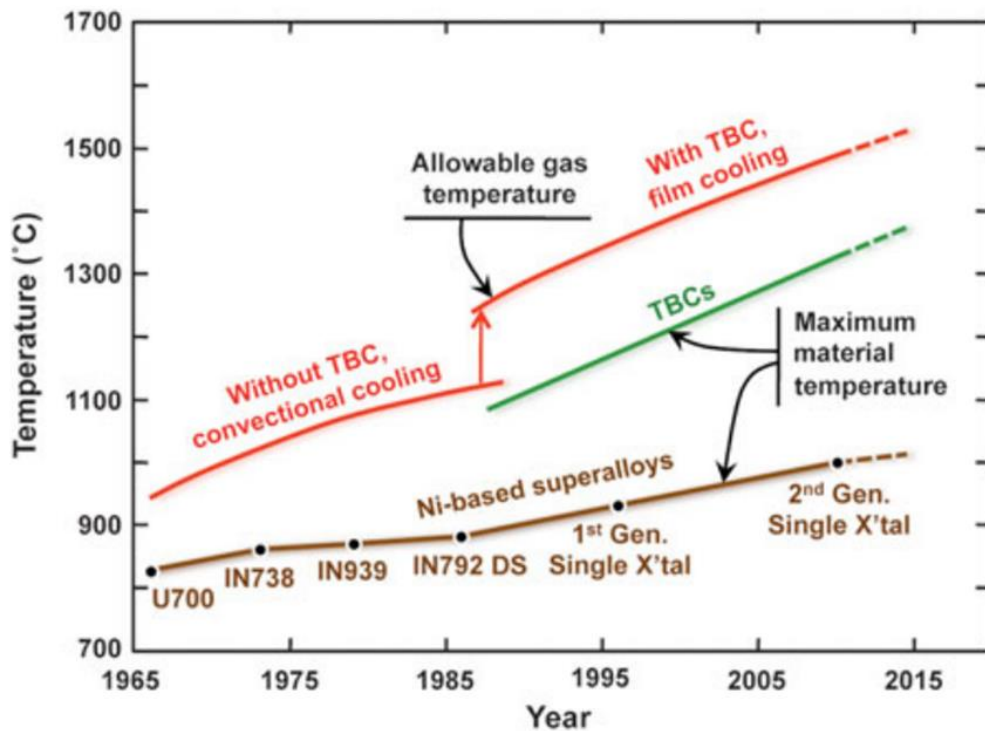


Figure 1.2.1 Effect of cooling methods in an aero-engine parts in the sense of allowable temperature [48].

1.3. Cooling Methods

The primary goal of a green aircraft is to generate more power with less fuel. Increasing the burning quality of the fuel-air mixture is the key factor for both power and efficiency concerns. Therefore, fuel air mixture has to be burned at maximum temperature possible in the combustion chamber.

Today, a typical combustion chamber is exposed to 1400 °C during the operation that is far more than the melting temperature of structural parts. Any metal or alloy

available in the world cannot resist such temperatures without any additional cooling system. Thus, integrated cooling systems are essential to sustain the structural integrity of an engine. There are three fundamental cooling methods for hot stages of an engine: *internal cooling holes*, *film cooling* and *thermal barrier coatings*.

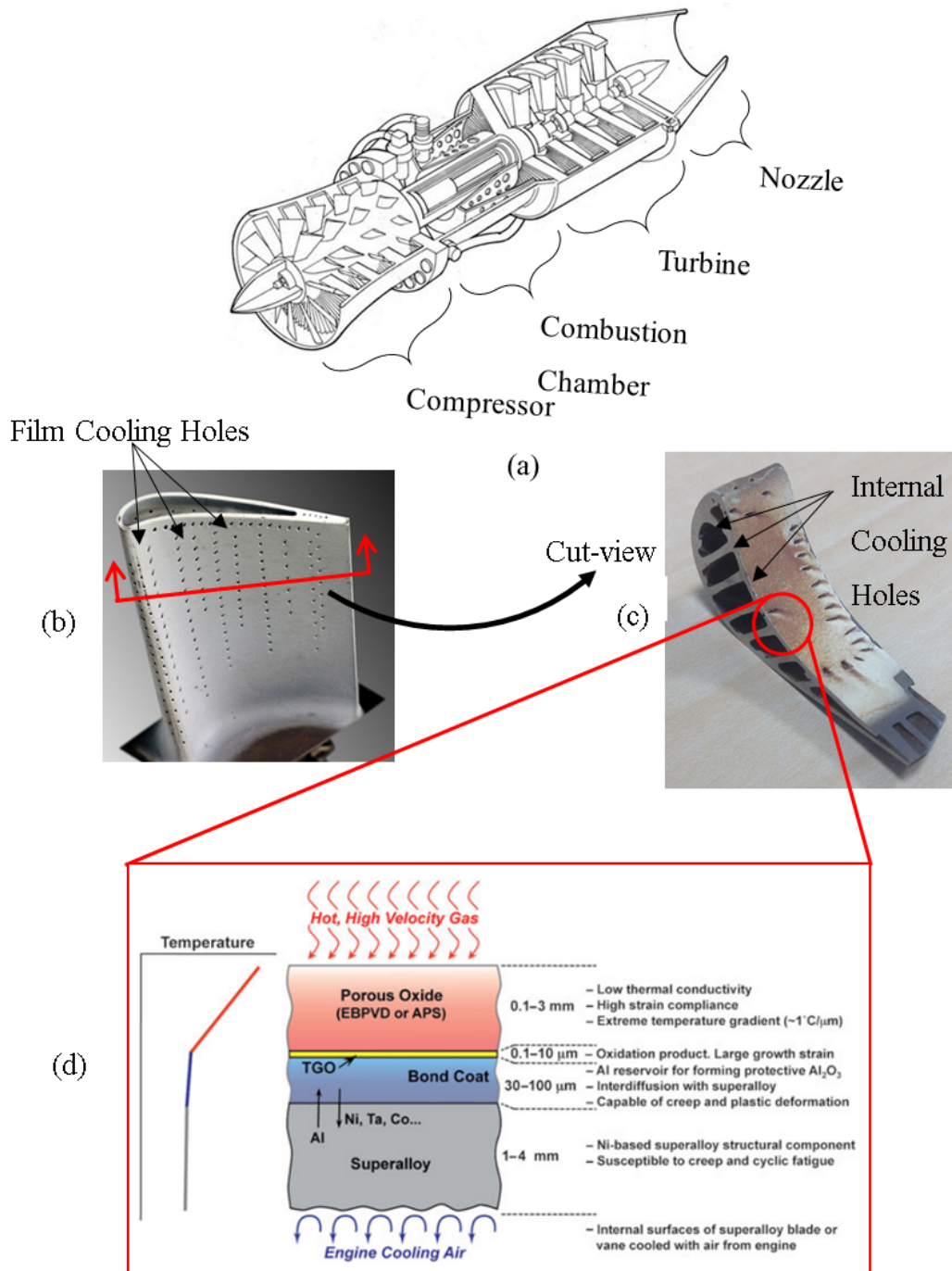


Figure 1.3.1 (a) A typical turbojet engine structure, (b) A TBC coated turbine blade before operation, (c) Cut-view of a TBC coated turbine blade after operation, (d) Cooling methods on turbine blade in operation.

1.3.1. Internal Cooling Holes

Internal cooling holes cool down the part from inside by small cooling holes embedded inside which is also illustrated in Fig. 1.3.1(c). There is an internally circulating air which is relatively cooler than the ambient temperature of the combustion chamber. This air internally lowers the overall component temperature but could not affect the surface temperature. Internal cooling holes are very efficient and sustainable but limited to overall temperature of parts.

1.3.2. Film Cooling

Film cooling is an innovative cooling method which is mostly used in aerodynamic parts. A set of micro holes in Fig. 1.3.1 (b) are drilled on parts at a certain angle by using a precision laser beam drilling machine. These holes provide an air leakage sourced from the internal cooling holes. The leaked air jets through the surface of the part and creates a cool air stream on the part. This phenomena provides both extra cooling on the surface where internal cooling is not sufficient and boost the aerodynamic efficiency of the aerodynamic part.

1.3.3. Thermal Barrier Coatings

Thermal barrier coatings (TBCs) are passive cooling systems which work as an insulation layer between hot air and structural part. TBCs contain four major layers and illustrated in Fig. 1.3.1 (d): a metallic substrate, a metallic bond coat (BC), a ceramic top coat (TC) and a thermally grown oxide (TGO) layer lying at the TC/BC interface.

Most of the engine parts are made of a nickel-based superalloy since they have perfect creep properties, excellent high temperature resistance and quite good elastic properties. First step is to coat the nickel-based superalloy parts by a nickel-based powder, NiCrAlY bond coat, using air-plasma spraying (APS) technique. The application of NiCrAlY undercoat both increases the adhesion of ceramic top coat and protects the nickel-based superalloy substrate from extreme environmental factors. Second, an insulating ceramic top coat can be manufactured by two different technique: air plasma spraying (APS) and electron beam physical vapor deposition (EB-PVD). Yttrium stabilized zirconia (YSZ) ceramic is used in both manufacturing technique but both manufacturing techniques end up different appearance and thermal properties. Hot ambient temperature in operation and manufacturing process increase

the diffusion of aluminum in the BC and permeability of oxygen through ceramic TC. As a result of this, the ambient oxygen penetrates through the TC and oxidizes the diffused aluminum in the BC and form an alumina (Al_2O_3) layer between top coat and bond coat.

1.3.3.1. Air Plasma Sprayed TBCs

Thermal spraying processes contain four subsystems [82]: composition of the enveloping atmosphere, flame or plasma stream, substrate material and powder material. Air plasma spraying or atmospheric plasma spraying (APS) is a thermal spraying technique which processed under atmospheric conditions using a plasma stream. A typical APS application is illustrated in Fig.1.3.2 (a). A plasma gun structured by a cathode bar and an anode shell covering it. A stream of plasma gas is pushed between the two opposite charges to form the plasma stream. Then, the powder is injected inside the plasma stream and pushed through the surface. The powder particles get melt in the plasma stream and accelerate through the surface. They splash the surface and cool down instantaneously. Powder particles piled up one on another and form a thickness of coating. Each pile of cooled powder particle is called as splat. APS technique is a simple application that used in many industries for thermal, corrosion and wear protection of the substrate.

A typical structure has shown in Fig.1.3.2 (b). APS TBCs have two major features. First, APS TBCs are randomly structured coatings that result flaws appeared inside of the coating layer (porosity, micro-cracks). Second, APS TBCs has wrinkled TC/BC interface which also increases the adhesion of TC on BC.

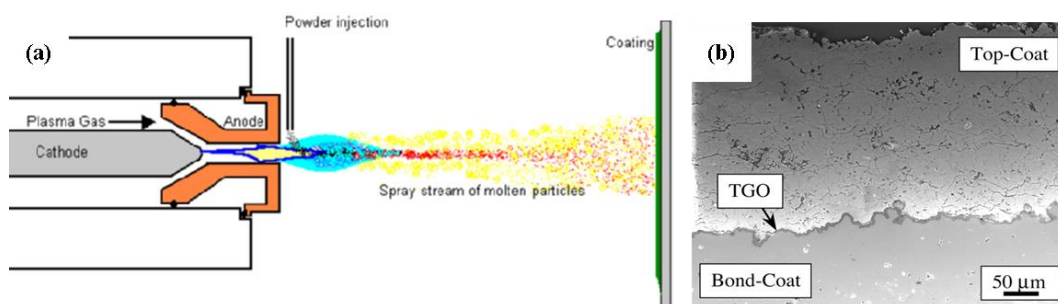


Figure 1.3.2 (a) Components of a typical air plasma spraying (APS) application showing (b) a typical resultant coating structure from its cross-section.

1.3.3.2. Electron Beam Physical Vapor Deposition TBCs

Electron beam physical vapor deposition is based on physical vapor deposition technique which is illustrated in Fig. 1.3.3(a). There is an anode electrode to generate an electron beam under high vacuum targeted to the ingot supplement of the ceramic. The high energy is transmitted through electron beam and vaporizes the ingot supplement of the coating. The specimen is showered by the vapor of the coating material to form a thickness. This method is an energy consuming and a very slow technique to form a coating. However, different from any other coating methods, a feather-like structure can be achieved by EB-PVD process.

A typical EB-PVD TBC structure is presented in Fig. 1.3.3 (b). The feather-like structure has a more compact and free of large air gaps structure. The thermal insulation performance is way better than any coating because of its microporous structure. However the resultant interfaces are very smooth when it is compared to APS TBCs and as a result of this, the interfacial crack propagation is a significant problem in the industry. The formed ceramic layer by EB-PVD has a smooth interface and the bond coat metallurgy had to innovate to satisfy the superior needs of EB-PVD processes top coat.

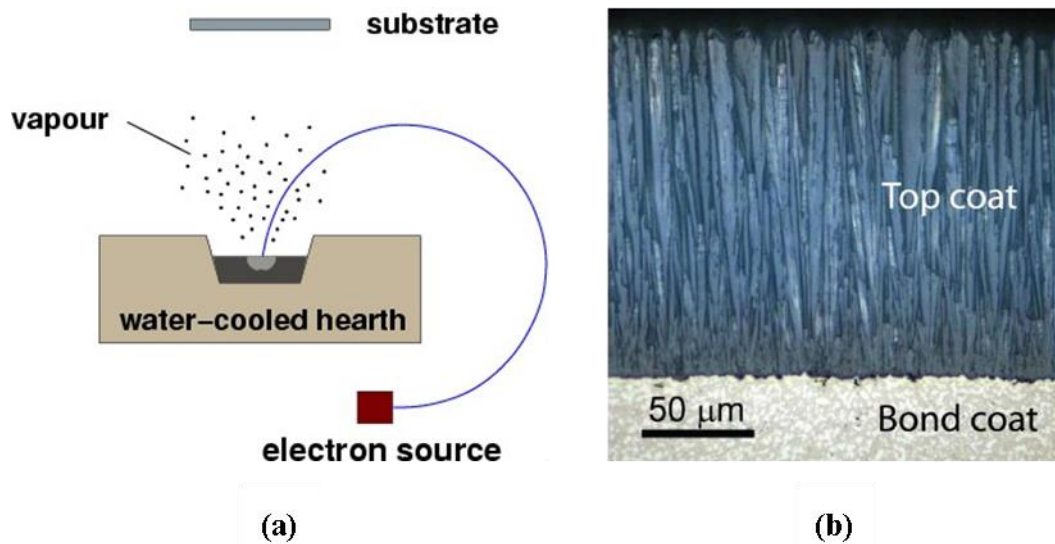


Figure 1.3.3 (a) Components of a typical electron beam physical vapor deposition (EB-PVD) application showing (b) a typical resultant coating structure from its cross-section.

1.4. Failure of Thermal Barrier Coatings

Thermal barrier coatings are brittle ceramic coatings deposited on ductile substrates. They operate under extreme conditions where creep, thermal fatigue and sintering may become critical. Failure of TBCs is a major problem for the insulated parts and the system. Mechanical failure of TBCs are categorized under 5 fundamental mechanisms [25]; aluminum depletion, nickel diffusion through TGO, foreign object damage, planar interface with imperfections and undulating/wrinkled interface.

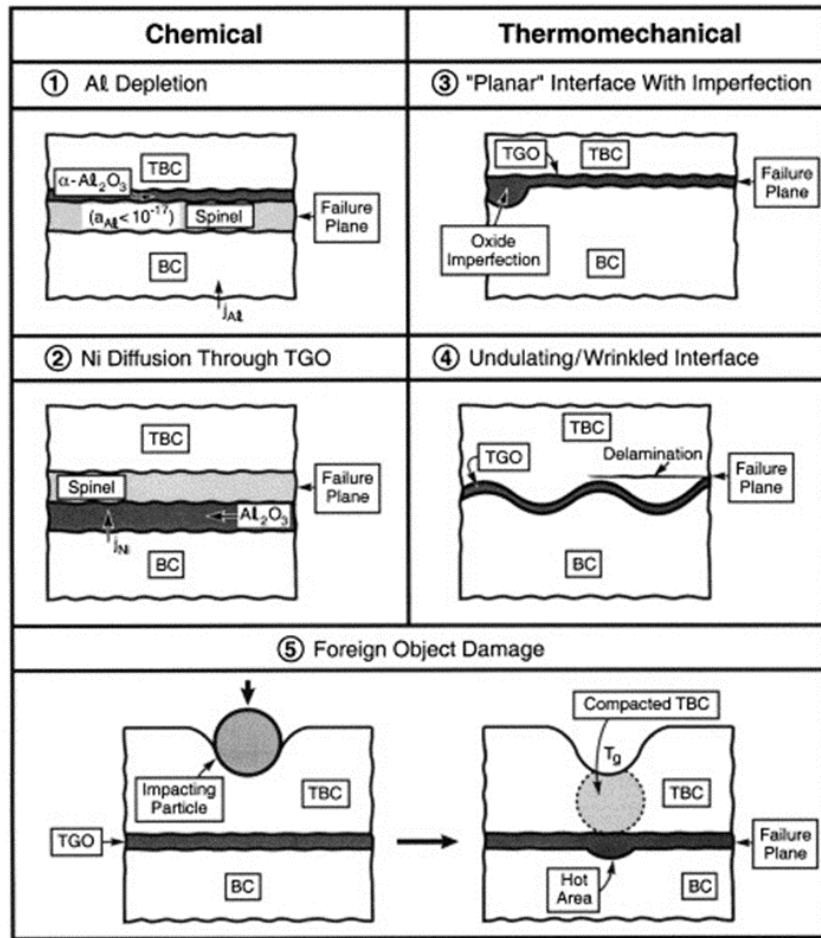


Figure 1.4.1 Categories of mechanical failure of TBCs [25].

- Aluminum depletion and nickel diffusion based failures are chemically driven failure mechanisms. Both chemical mechanisms are fundamentally weakening the TC/BC interface by causing interface cracking. The elevated temperature in the combustion chamber increases diffusibility and reactivity of atoms and molecules. This cause a continuous modification of the material content and

property of the interface. Formation of more brittle films at the interface by the chemical reactions, failure of the interface is inevitable.

- TBCs can be damaged from particle or objects traveling through the engine. These type of damages are evaluated under foreign object damage (FOD). For instance, engine sucks the dust in the ambient and burn the dust in the combustion chamber while working. The melted dust particles stick on the turbine blades or combustion chamber parts and chip off small pieces from the ceramic coating and bare metallic components. Corrosive effects cause a substantial amount of coating loss during operation by corrosion.
- Air plasma sprayed TBCs have undulating/wrinkled interfaces. Typical failure mechanisms in APS TBCs are illustrated in Fig. 1.4.2(a). There are four fundamental sub-mechanisms depicted in the following figure. As a result of the manufacturing technique, there are peaks and valleys at the TC/BC interface. Because of the thermal mismatches between the materials of TGO, BC and TC, peaks and valley are under pre-tension and pre-compression stresses, respectively. The pre-tension at peaks cause an opening stress at BC/TGO (I) and TGO/TC (II) interfaces. As a matter of fact, most of the interface failure occur at the pre-tensioned (peaks) parts of the interface. The interface cracks can propagate and pass through the BC or TC and lead to ultimate failure of the coating (III). Moreover, porosities in TC and BC may act like a pre-crack and propagate through the coating (IV).
- Electron beam physical vapor deposition TBCs have planar interfaces. A typical failure of EB-PVD TBC interface is illustrated in Fig. 1.4.2. Imperfections, such as porosities, local wrinkles, pre-cracks at the interfaces mostly drive the failure. The smooth interface of EB-PVD TBCs is vulnerable to any opening stresses. Thermal mismatches between TC and BC during the cyclic heating and cooling may initiate an opening at the interface (I). TC/BC interface of EB-PVD TBCs cannot even limited imperfections cannot be tolerated. A tiny interface porosity can initiate a crack (II, III) and cause spallation of TBC. Different from APS TBCs, EB-PVD TBCs fail because of a perfect interface failure at the TC/BC interface.

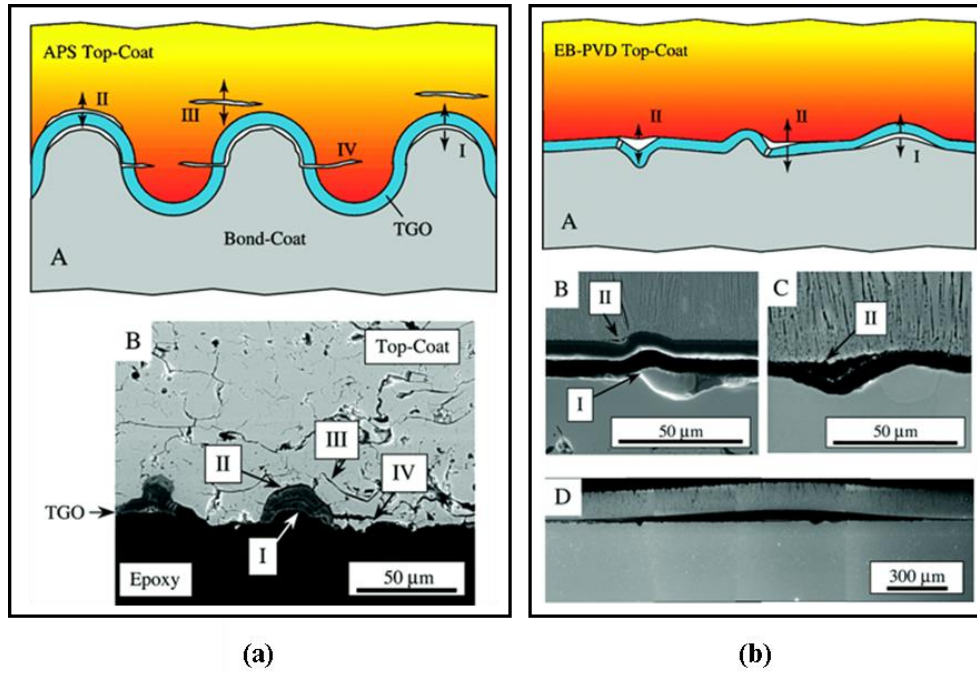


Figure 1.4.2 (a) Failure of an APS TBCs by (I) BC/TGO interface opening crack (II) TC/TGO interface opening crack (III) TC opening crack (IV) Crack propagation through TC (b) Failure of EB-PVD TBCs by (I) BC/TGO interface opening crack (II) TC/TGO interface cracking (III) Interface porosity opening crack [47].

1.5. Studies on Experimental Evaluation of Thermal Barrier Coatings

1.5.1. Historical Background

South African Airlines had first used thermal barrier coatings to extend the service of their engine turbines in late 1970s [34]. This attempt made a huge difference in the service life of engine parts and the engine industry had to adapt this technology to their engines. After fuel consumption had become a major problem for airliners, TBCs became more popular, the unit fuel consumption for a specified thrust should be reduced. This means that aero engine manufacturers should increase the burning quality and as a natural result increase the turbine inlet temperature. The extensive use of TBCs on engine parts arise new problems to qualify TBCs in the manner of fracture and failure. In more than 30 years of experience on TBCs, industry still suffer from the lack of evaluation of TBCs by quantifiable methods and lack of fracture mechanics based life prediction models [48, 51, 81]. There are numerous studies that focus on experimental methods and analytic models to solve ultimate problem of “*indefinite durability of thermal barrier coatings*”.

1.5.2. Experimental Studies on TBCs

In this part relevant experimental studies were presented from general to specific order.

In the paper “*Measurement of the adhesion of a brittle film on a ductile substrate by indentation*”, Drory and Hutchinson [21] followed a testing procedure to measure an interface toughness of a diamond-coated titanium alloy where diamond acts as the brittle coating and titanium acts as a ductile substrate. More important than their definite results of interface toughness under particular conditions, they illustrated and listed a series of adhesion measurement techniques in detail. They also discussed the characteristics of each test method. They also made clear and objective judgements on both advantages and disadvantages of each testing method.

In Fig. 1.5.1, there are 8 different possible testing methods to measure the adhesion quality of various combinations of coating and substrate by quantitative methods. These tests are examined according to their limitations on specimen geometry, dominant mechanisms leading to delamination etc.

First, the film adhesion can be measured by lifting the coating by a tape glued on the coating. This application can only be used for flexible coatings or very brittle interfaces. Second, a scratch test can be conducted as illustrated in the following figure. Moreover, an improved version of this test also developed Liu, Kagawa and Evans developed a “barb test” in [10, 43] which is an improved version of the scratch test. They measured the fracture toughness of TC/BC interface of EB-PVD TBCs. Third test is pulling the coating layer perpendicular to its surface with the help of a rod adhered on it. The adhesion quality can be measured by measuring the diameter of the ripped part of the coating and the applied load. Moreover, this test has already been using in the industry to qualify the APS productions. The first three tests are based on the impact size of the load and applied load on rod or the tape which drives the delamination. These methods are more suitable to measure the adhesion of very brittle and weak interfaces such as polymeric or dissimilar material interfaces. In addition to that, these methods can only supply consistent data if the testing conditions and material couple was set. Therefore, these methods mostly used by the industry to qualify their regular production. In terms of research studies, these testing methods require some technical improvements on measurements as well as the definition of the mechanism leads to the failure as Liu et al. [43] did in their study.

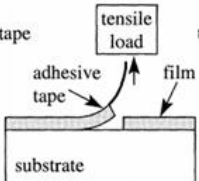
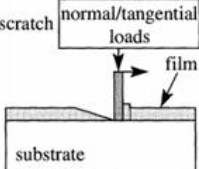
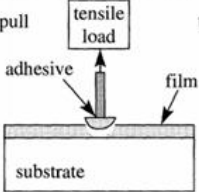
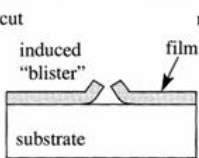
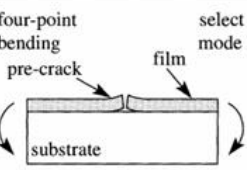
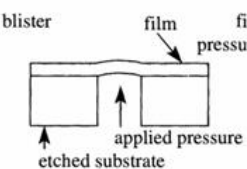
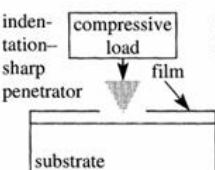
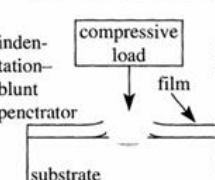
test technique	schematic	characteristic aspect for film delamination	limitations on specimen geometry	measurement of interface fracture toughness	sign of film stress	film	substrate	comments
1 tape		tensile force	none	no	T/C	ductile	ductile/ brittle	analysis is complex; not for brittle films
2 scratch		sliding penetrator	none	no	T/C	ductile	ductile/ brittle	analysis is complex; not for hardcoatings
3 pull		tensile force	none	no	T/C	ductile	ductile/ brittle	strength measurement
4 cut		residual film stress	none	yes	T	ductile/ brittle	ductile/ brittle	precracking difficult for hardcoatings; limited range of interface toughness
5 four-point bending		select mixed-mode loading	yes (special geometry)	yes	T/C	ductile	ductile/ brittle	pre-cracking difficult; involved specimen preparation
6 blister		film pressurization	yes (special geometry)	yes	T	ductile	ductile/ brittle	complex specimen preparation and pre-cracking
7 indentation-sharp		substrate plasticity (induced residual stress)	yes (requires smooth interface)	yes	T/C	ductile/ brittle	ductile/ brittle	requires smooth surface; for limited interface toughness
8 indentation-blunt		substrate plasticity (induced residual stress)	none	yes	C	brittle	ductile	for ductile substrates

Figure 1.5.1 A set of adhesion measurement techniques analyzing each test method by their characteristics, limitations, capabilities, material couples and loading conditions where C and T are compressive and tensile, respectively [21].

Measurement of interface fracture toughness is possible for the rest of the methods listed in Fig. 1.5.1 unlike methods 1-3. Therefore, rest of the methods were used and examined in academic studies on TBCs [22, 23, 43, 73, 78, 80-82]

One of the most comprehensive study on four-point bending was conducted by Zhou et al. [43] in 2002. Zhou et al. conducted tensile and four-point bending tests on APS TBCs to characterize and compare the fracture behavior of APS TBC systems with different properties (bond coat roughness, substrate thickness and material, etc.). In both tests, the fracture behavior of each specimen is found to be similar. When the substrate was plastically deformed, cracks initiated at the top of the coating and propagated through the thickness up to the bond coat/top coat interface where cracks either kinked and ran through or passed the TC/BC interface and kinked at the substrate/bond coat interface. Gross delamination occurred when multiple interfacial cracks coalesced. Zhou et al. [43] used two analytic fracture models to calculate interfacial fracture toughness: Hu & Evans's (1988) shear lag model (which is only used for uniaxial tensile testing) and Suo & Hutchinson's (1988) composite beam model. Zhou et al. [43] mainly aimed to calculate interfacial toughness from experimental data. They came to a clear conclusion that bond coat roughness positively affects the cohesion between top coat and bond coat.

Indentation is another testing method that carries huge potential. Vasinonta et al. [73] published a state-of-the-art article in 2002 which proposes a complete methodology to measure interface toughness in EB-PVD TBCs.

1.6. Scope of the Study

Thermal barrier coatings are insulating ceramic coatings that mechanically and thermally protects the base material from hot gasses. In an aero engine, the most required locations of TBCs are where the temperature and its effects on structural parts are severe. From this perspective, TBCs are directly correlated with the fuel efficiency, performance and service life of the engine. Lack of fracture models and standard testing methods on TBCs force the industry to use more experience based techniques in design. Engine industry in Turkey has no decisive criteria or testing procedure to develop new TBC systems. This study is focusing on development of a quantifiable method to measure fracture toughness of thermal barrier coatings. Gaining the ability to measure fracture properties of TBCs will improve the understanding of

thermal barrier coatings. This would be a significant step through the accomplishment of the ultimate aim: *green aircraft*. Hopefully, further TBC researches will lead more durable propulsion systems by ensuring their performance in maximum time possible in the future.

This study is organized under four major chapters. In chapter 1, first the fundamental motivation of this study is introduced which is the development of a better aircraft with better fuel efficiency: "*green aircraft*". Then, contribution of the propulsion science to the ultimate motivation is discussed. From the propulsion point of view, the fuel efficiency was directly correlated with the cooling systems and by means that with thermal barrier coatings. Then the available thermal barrier coating systems were introduced and failure of TBCs were discussed. Briefly, the experimental studies in the literature is surveyed. In chapter 2, the metallurgical and mechanical characterization of TBC specimens were done. Methods used during the characterization procedure was given in conjunction with their literature survey. First, the specimen set was introduced and metallurgical characterization were done by microscopy, EDS and XRD. Then, mechanical characterization was done by nanoindentation. At each layer, characteristic elastic modulus and hardness values were measured from large data sets using appropriate statistical approaches. In chapter 3, the method used in chapter 4 for the measurement of the fracture properties of TBCs was developed. A four point bending test procedure was introduced with the theory and formula underneath. Al-PMMA composite beam is used to model a similar material couple and used three different adhesives to demonstrate different interface properties. In chapter 4, the developed four point test in the previous chapter was applied on TBC specimens. From these tests, two major results were achieved. First, the fracture mechanism for three different coating thicknesses can be visualized by DIC. Second, the critical energy release rate of the TC/BC interface can be measured.

CHAPTER 2

METALLURGICAL AND MECHANICAL CHARACTERIZATION OF THERMAL BARRIER COATING SPECIMENS

In this chapter, TBC specimens were characterized by analyzing the microstructure and measuring the elastic modulus and hardness of each layer from the cross-section of mounted specimens before any coupon test. First of all, specimen preparation methodology and imaging techniques used in microstructural analysis were introduced. Then, benchmark tests: energy dispersive spectroscopy (EDS) and X-ray diffractometer (XRD) used to analyze powder forms of top coat and bond coat were interpreted for non-metallurgists. After the metallurgical methods had been covered, details of nanoindentation testing used to measure elastic modulus and hardness were introduced in conjunction with statistical data analysis methods. Finally, mechanical properties of the layers measured by nanoindentation were presented.

2.1. Methodology

2.1.1. Specimen Preparation and Microscopy

Failure mechanisms of TBCs are attached to the microstructure of TBC layers as discussed in Chapter 1. Microstructural characteristics of TBC specimens (Fig. 2.1.1(a)) are interpreted from cross-sectional microscopy of cut (Fig. 2.1.1(b)) and mounted TBC specimens (Fig. 2.1.1(c)).

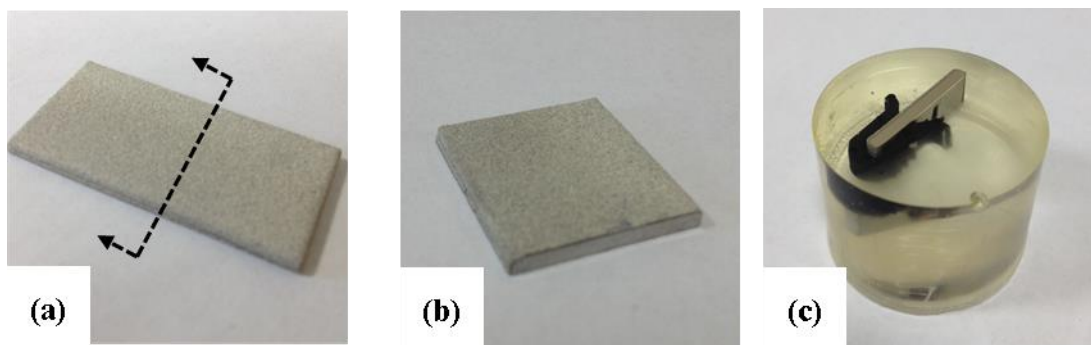


Figure 2.1.1 (a) As-sprayed TBC coupon (b) Cut specimen from the cross-section (c) Epoxy mounted TBC specimen

Surface quality of mounted specimens is very essential both for optical and electron microscopies. Visual improvement between un-polished surface (Fig. 2.1.2(a)) and polished surface (Fig. 2.1.2(b)) microscopy demonstrates the contribution of surface quality to the study. Surface defects, like in the Fig. 2.1.1(a), may cover valuable information and change the vital remarks for the study. Therefore, same in Fig. 2.1.2(b), flattened, mirror-like polished, non-scratch surfaces are postulated for microstructural analysis.

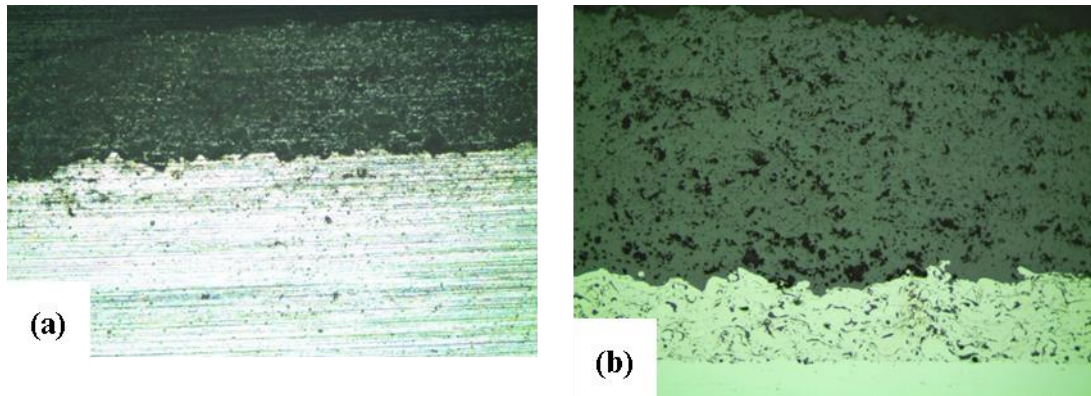


Figure 2.1.2 Typical optical microscopy (50X) from the cross-section of epoxy mounted TBC specimens to qualify surfaces after (a) cutting and (b) grinding and polishing.

There are certain rules and standards in specimen preparation [1, 3], and microstructural analysis of materials yet, the procedure is very dependent to series of subjective observations and deductions. Therefore, standardized specimen preparation takes an important place to achieve consistent results from microscopy.

In this study, surface preparation is essential for both microstructural analysis and nanoindentation tests to measure elastic modulus and hardness and for after failure analysis covered in Chapter 4. Cross-sectional surface preparation of dissimilar layered materials is quite different from surface preparation of monolithic materials. Therefore, at the very beginning of this study, the following standardized specimen preparation procedure was prepared to avoid inconsistency in the analysis of different specimen sets.

I. Cutting

TBC specimens were cut using Metkon Micracut 151 and Micracut 201 precision cutting machines. Diamond saw cutter was operated under water-based anti-rust bath to not damage the brittle coating layers while cutting. Specimens cut at 2000-2500 rpm saw speed in Micracut 201 machine and at 900 rpm saw speed in Micracut 151. Feed rate of the specimen in both machines is around 50-100 $\mu\text{m}/\text{min}$.

II. Mounting

Two component epoxy resin was used to mount the specimens because of its low shrinkage rate and short room temperature curing period [1]. Metkon Vacumet vacuum cabin was used to mount the specimens while a bubble removal procedure was followed to avoid air bubbles in the specimen [personal communication with I. Karaali, 42].

III. Grinding & Polishing

Grinding and polishing was done by Metkon Forcimat and Presi Minitec 233 polishing machines. Grinding and polishing procedure in Table 1 for TBC specimens was developed and standardized with the collaboration of Metkon R&D Laboratory [personal communication with I. Karaali, METKON, 42].

Table 2.1.1 Grinding and polishing steps of the standardized cross-sectional surface preparation procedure for TBC specimens followed after cutting and mounting.

Step	Sand Paper Mat	Application Coolant / Solution	Base Speed	Applied Force	Duration
1	400 Grid SiC	Water	250 rpm	30 N	2 min
2	800 Grid SiC	Water	200 rpm	25 N	2 min
3	1200 Grid SiC	Water	200 rpm	25 N	2 min
4	Metapo B	3 μ m Diamond	150 rpm	30 N	3 min
5	Metapo V	1 μ m Diamond	150 rpm	20 N	3 min
6	Collo	Colloidal Silica	150 rpm	15 N	1 min
7	Fedo 1N	0,25 μ m Diamond	150 rpm	15 N	2 min

Optical microscopy was used to qualify the surfaces after preparation, to measure TBC layer thicknesses and to analyze the microstructure of layers and interfaces. Optical microscopy was practiced on Huvitz HDS-5800 digital automated imaging system at the Mechanics of Materials Laboratory in METU Research Center for Wind Energy.

Scanning electron microscopy was used to measure particle sizes, to define particle shape and structure of BC and TC powders. SEM was practiced on 30 kV FEI 430 nano-scanning electron microscope in the Department of Metallurgical and Materials Science and QUANTA 400F field emission SEM in the Central Laboratory at Middle East Technical University. Also, energy dispersive X-ray spectroscopy feature of both machines were used to run an elemental analysis of BC and TC powders.

2.1.2. X-ray Diffraction Crystallography

X-ray diffraction (XRD) crystallography is a common technique to study the crystalline structure of materials. Many materials are made of crystals structured by a lattice of atoms. The crystalline structure cannot be seen by optical microscopy since visible light cannot penetrate through a material. X-rays are small enough to pass through surface of a material. In XRD crystallography, a beam of X-rays sends to the material at certain angles, as X-rays hit the material it diffracts from the crystal plane at a certain angle. The angles of diffraction are recorded, and the crystal planes can be calculated from the diffraction angles.

In this study, XRD crystallography is used to study the crystal structure of TC and BC powders. The crystal structure of TC powder would interpret the stabilization of the ceramic.

2.1.3. Energy Dispersive X-Ray Spectroscopy

Energy dispersive X-ray spectroscopy (EDX or EDS) is an elemental analysis method that relies on energy dissipated from the matter under X-ray excitation [27]. Material content in the specimen interacts with the X-ray beam and dissipates some of the energy as photons. A silicone-based sensor collects all dissipated photons and measures their energy in mV scale. The reliability of the collected data is dependent to the sensitivity of energy sensor. Acquired energy data is used to generate an energy spectrum. Each element has characteristic energy levels in the energy spectrum. Peaks at certain values in the energy spectrum indicate particular elements. Therefore, the elemental content and percentages of material can be obtained from the energy spectrum.

In this study, EDS is used to characterize and map elemental content of BC and TC powders.

2.1.4. Nanoindentation

Johan August Brinell developed first indentation technique in 1900 to measure the hardness of steel by correlating maximum applied load, penetration depth and resultant indent size. Since then, indentation gained acceptance as an easy to implement non-destructive in-situ material testing technique. As thin films and coatings were developed, in-situ non-destructive testing became a necessity for these materials and components. Therefore, researchers focused on finding new ways to measure valuable material properties by indentation.

Nanoindentation is a pioneering material testing concept that promises measurement of material properties by probing materials in the nano-micro scales. With this technique, elastic modulus and hardness of materials can be measured by monitoring the applied load at the loading scale of mN and depth of penetration at the length scale of nm. Moreover, with advanced sensing capabilities of nanoindenter, nanoindentation is used to generate 3D imaging of surface features and resultant indents which can be further used to determine fracture toughness and plastic properties of materials.

2.1.4.1. Studies on Instrumented Indentation

Instrumented indentation is a method based on continuous measurements of depth and load applied on a surface probed by an indenter tip. Instrumented indentation studies underlay the classical solutions of contact problems [9, 31]. Hertz's study [31] of the contact problem is particularly important in the manner of indentation. He defined a reduced modulus [15] or contact modulus between two elastic bodies given by

$$\frac{1}{E_r} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu_s^2}{E_s}, \quad (2.1.1)$$

where subscripts i and s denote the indenter and the substrate, respectively.

Tabor [65] in 1948 conducted instrumented indentation tests on metals by using conical indenters. This study stated four considerable observations on the mechanics of indentation beyond the load-displacement curve, illustrated in Fig.2.1.3:

1. The existence of plasticity during indentation was proven for both conical and spherical indenters from the shape difference of the resultant indent shape and the indenter shape.
2. The rebound of the indented surface during unloading was observed while keeping the contact impression the same.
3. Plasticity effects were diminished by loading and unloading the metal surface several times.
4. The relationship between applied load and penetration depth can be explained by Hertz's reduced modulus in Eq. 2.1.1.

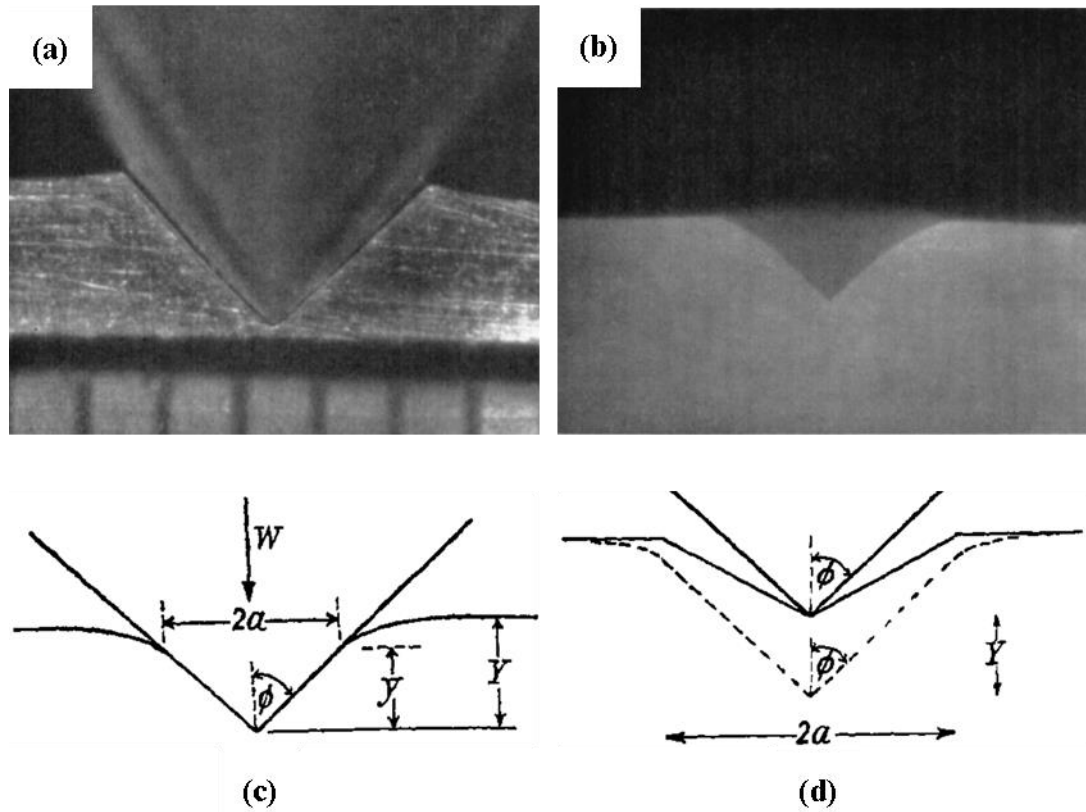


Figure 2.1.3 (a) Cross-sectional view of the indentation profile of a conical indenter lying in the indent formed in duralumin which is illustrated in (c) showing the contact depth y , measured depth Y and (b) Cross-sectional view of a resultant indentation impression of a conical indenter in a solid polymer which is illustrated in (d) showing the rebound of the indent [65].

As a result of these observations, Tabor [65] stated that elastic modulus and the area of the indent can be related to the unloading curve of the indentation and the total amount of the rebound of the indented surface. Stillwell [58] repeated the same study for spherical indenters in 1961.

In the 1970s, studies covered by Bulychev, Alekhin, Shorshorov and co-workers [6, 7, 57, 66] focused on measuring elastic modulus from a load-displacement curve by using an instrumented micro-hardness machine. The typical loading and unloading curve depicted in Fig. 2.1.4(a) was analyzed based on the elastic contact models. As a result, the very first unloading portion of data was correlated with the reduced modulus of two bodies (Eq. 2.1.1).

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A}. \quad (2.1.2)$$

The usability of Eq. 2.1.2 was verified by Buylchev et al. for spherical and cylindrical indents and verified by Pharr, Oliver and Brotzen [50] for any indenter that is a revolution of a smooth function. Moreover, Buylchev et al. argued that pyramidal indenters should demonstrate the behavior of Eq. 2.1.2 without any significant deviations. Therefore, the relationship between area of contact, reduced modulus and the slope of the initial unloading curve is not limited to any indenter geometry.

Instrumented indentation techniques became very popular in the 1980s after requirement for a practical technique capable of measuring mechanical properties of thin films and layered electronic components arose in the industry. There are particularly important studies conducted by Oliver, Hutchings and Pethica [49] and Doerner and Nix [20]. Oliver, Hutchings and Pethica [49] indicated a new method to predict the indent area of the indenter. The method was based on a simple assumption that the indent shapes are exactly the same shape as the indenter at a certain level of penetration. If this depth can be measured from load-displacement curves then the resultant area of the indenter can be calculated from the shape or area functions of the indenters using Eq. 2.1.2. They found that the very first assumption is wrong and instead the final depth of the contact area gives better results than the depth at peak load.

Doerner and Nix [20] collected many studies from the literature to produce a method of nanoindentation to determine hardness and elastic modulus from load–penetration curve. They proposed a comprehensive method to measure elastic modulus from load-penetration curve which are based on observations of steadiness of the contact area at the initial stages of unloading which is similar to the contact behavior of cylindrical flat punch. They also proposed an empirical method to determine the area. Their estimation of contact area is based on the extrapolation of the linear part of the unloading curve to the zero load and using the depth with the indenter shape function to calculate the contact area accurately. As a result, Doerner & Nix successfully calculated the elastic moduli of silicon and nickel.

Finally in 1992, Oliver and Pharr [45] published a landmark article with their remarkable conclusions on the behavior of contact proposing a complete new method of analysis to determine elastic modulus and hardness from load-penetration curve. Briefly, they took the Doerner & Nix method and derived an area function for Berkovich indenter and derived a model of test setup to calculate the compliance on depth measurements. Then, they justified the area function for six different materials by non-dimensionalizing the calculated area and imaged area. Now, “*Oliver & Pharr Method*” is the fundamental analysis method of nanoindentation tests to calculate elastic modulus and hardness in most materials.

2.1.4.2. Oliver & Pharr Method

In Fig. 2.1.4, a typical load-displacement curve of an instrumented indentation test and a sketch of the corresponding impression of the indentation is presented. In Fig. 2.1.4(a), the loading and unloading curves were indicated where “S” is corresponding to the slope of the tangent line of the unloading curve at maximum load (P_{max}). In Fig. 2.1.4(b), the surface profiles were indicated at three stages of indentation: the initial surface profile at the beginning of the test, surface profile under loading and the residual surface profile after the load removed. The penetration depth is indicated as h , the depth of the residual indentation impression is indicated as h_f , the depth of the contact of indenter and surface under the load is indicated as h_c and the displacement of the surface at the vicinity of indentation is indicated as h_s . Therefore, the rebound of the indented surface can be described by $h_{max}-h_f$ where h_{max} is the depth of the indenter at maximum load and the total measured depth is h_c+h_s .

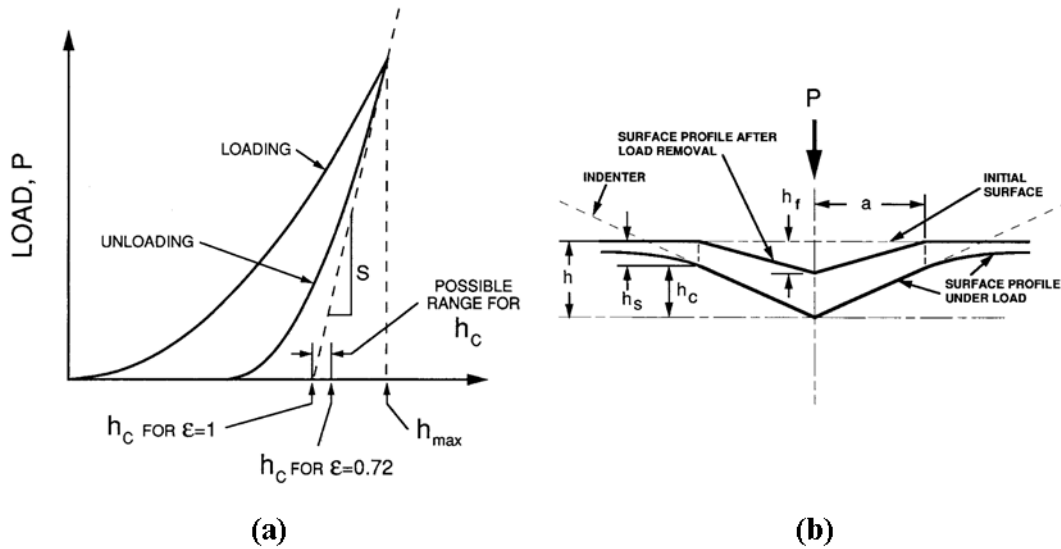


Figure 2.1.4 (a) A typical load displacement curve used to analyze the instrumented indentation, (b) the behavior of indent impression by the means of loading and unloading [45].

There are three important assumptions that Oliver & Pharr made before the analysis of the load-displacement curve:

1. The flat punch approximation is not sufficient to explain the contact behavior of unloading curve for Berkovich indenters.
2. Sneddon's analytical contact solution for punches, expressed by

$$P = \alpha h^m \quad (2.1.3)$$

gives a more accurate description of the elastic unloading of an indentation made with a Berkovich indenter where α and m are unique shape constants for an indenter tip.

3. The equations describing the elastic unloading of a flat semi-infinite half-space are the same as those for an indented surface that Sneddon's solutions apply equally well to a flat surface or a surface with hardness impression.

There are three fundamental equations used to calculate the elastic modulus of a material by using a nanoindentation: the reduced modulus of contact defined in Eq. 2.1.1, Sneddon's classical power law solution of contact of two elastic bodies and the relationship between stiffness of the unloading curve and contact stiffness defined in Eq. 2.1.2.

At the very beginning of the analysis of unloading curve, the stiffness of contact defined in Eq. 2.1.2 is rewritten as,

$$E_r = \frac{\left(\frac{dP}{dh}\right) \sqrt{\pi}}{2\sqrt{A}} = \frac{S \sqrt{\pi}}{2\sqrt{A}}, \quad (2.1.4)$$

is a solution for reduced modulus or the contact modulus where the area of contact is defined as a function of contact depth h_c ;

$$A = F(h_c). \quad (2.1.5)$$

Two important practical questions that arise in the measurement procedure of elastic modulus of a material: how the stiffness of the unloading curve will be determined and how the area function of Berkovich indenter tip is found. Oliver and Pharr proposed a power law approximation to determine the contact stiffness from an unloading curve of any kind of material and a determination method to find the area function of the indenter.

2.1.4.2.1. Determination of Contact Stiffness

Oliver and Pharr conducted series of nanoindentations on tungsten and noted two considerable features from the stiffness measurements by using linear fit of the unloading curve. First, they showed that the stiffness of the unloading curve changes as which fraction of the unloading curve is used to fit the unloading curve. Second, they showed that first and last unloading curves of successive indentations are completely different since the creep at indentations are more dominant at first indentation and then diminish after a few indentations. They proposed a power law fit to the unloading curve which describes the behavior of the unloading curve more precisely. Also, the power law approximation of the stiffness of the unloading curve is less-sensitive to creep. Approximation of the contact stiffness of unloading curve can be described as the derivative of Sneddon's power law of punches in Eq. 2.1.3 with respect to the depth of indentation:

$$S = \frac{dP}{dh} = \alpha \cdot m (h - h_f)^{m-1}, \quad (2.1.6)$$

where α , m and h_f are all determined by a least squares fitting. Finally, the stiffness of contact can be calculated at the very first fraction of the unloading curve by taking the derivative at maximum applied load.

2.1.4.2.2. Determination of Contact Area

To determine the area function in Eq 2.1.4 the contact depth should be calculated which can be defined from the sketch of the indentation in Fig. 2.1.4 as;

$$h_c = h_{\max} - h_s, \quad (2.1.7)$$

where the maximum depth, $h_{\max}$, is measured from the experiments but the displacement of contact surface h_s is still an unknown. As introduced before, Sneddon analytically solved the contact problem of two elastic bodies where the deflection of surface is dependent to the indenter geometry. As a result, for a conical indenter, the surface displacement, h_s can be expressed as,

$$h_s = \frac{\pi^{-2}}{\pi} (h - h_f). \quad (2.1.8)$$

Then, the relationship between the actual depth and the final depth can be expressed from Sneddon's force-displacement relationship for conical indenters as,

$$(h-h_f)=2\frac{P}{S}, \quad (2.1.9)$$

where P is the load at the depth of h and S is the stiffness at that load. The stiffness of the initial unloading curve was calculated by following the procedure introduced in the previous section. Putting Eq. 2.1.9 into Eq. 2.1.8 would give the displacement of the contact surface for conical indenters,

$$h_s=\left(\frac{2}{\pi}(\pi-2)\right)\frac{P_{\max}}{S} \quad \text{where } \epsilon=\frac{2}{\pi}(\pi-2) \text{ for conical indenter.} \quad (2.1.10)$$

“ ϵ ” is 0.72 for a conical indenter and 1 for a flat punch geometry. The estimated h_s data in the study of Doerner & Nix is the same as for an epsilon of 1 where they already made a flat punch estimation for the contact behavior. This shows that the method that Oliver & Pharr suggested to estimate displacement of the contact surface is also applicable for Doerner & Nix.

One of the ultimate measurement goal is to measure hardness without imaging the indent impression. The hardness of a material is described as the penetration of indenter at that load. Therefore, the hardness can be expressed as a proportion of a maximum load applied and the impression area at that load:

$$H=\frac{P_{\max}}{A}. \quad (2.1.11)$$

For high modulus materials, the compliance of machine may become important. The area of contact is still unknown. To estimate a good area, the compliance of the system is used. The total compliance is the summation of, the compliance of the specimen and the compliance of the testing frame, C_f , which is the inverse of the stiffness:

$$C=C_s+C_f \quad (2.1.12)$$

$$C-C_f=\frac{1}{S} \quad (2.1.13)$$

$$C-C_f=\frac{\sqrt{\pi}}{2E_r} \frac{1}{\sqrt{A}}. \quad (2.1.14)$$

By reorganizing the above equation, the area function of an indenter can be described in terms of compliance and reduced modulus, as,

$$A=\frac{\pi}{4} \frac{1}{E_r^2} \frac{1}{(C-C_f)^2}. \quad (2.1.15)$$

For a specified indenter-substrate couple, contact modulus should remain constant for any applied load where a plot of C and $A^{-1/2}$ should also be linear. To determine the area function of an indenter and the load frame compliance, it is assumed that for relatively large indentations the area function of the Berkovich indenter is converging to the following relation which is the area function of a perfect Berkovich indenter is given as,

$$A(h_c)=24.5h_c^2. \quad (2.1.16)$$

Two of the largest indentations on aluminum is used to get the slope of the linear plot of C vs $A^{-1/2}$ explained above. Then all other indentations were fit to the initial guess of perfect Berkovich indenter area function expressed in Eq. 2.1.16. Practically, there appear deviations at some levels of indentation, therefore ultimately, the area function will be fit to the power series of contact area, as,

$$A(h_c)=24.5h_c^2+C_1h_c^1+C_2h_c^{1/2}+C_3h_c^{1/4}+...+C_8h_c^{1/128}. \quad (2.1.17)$$

which is converging to the perfect indenter for large indentations. Then for other indentations the fit to new area function containing 8 different perturbation constants from the ideal condition. As, perturbation constants were found, the indenter tip area function is calibrated on aluminum which should be used for any substrate.

Oliver & Pharr showed the applicability of the area function of an indenter tip for 5 other materials. Each material yields the constant modulus assumption and also fits to the area function shown below as a line.

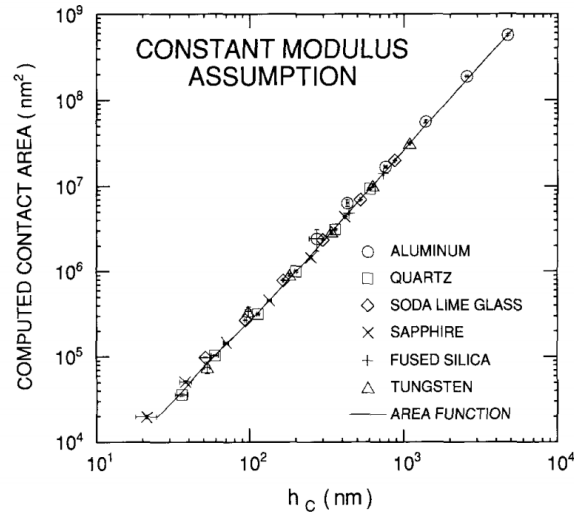


Figure 2.1.5 Oliver & Pharr resultant final figure [45].

After the state-of-the-art article in 1992, they refined and discussed the method according to the development of new methods and new materials in 2004 [46].

2.1.4.3. Nanoindentation on TBC Cross-section

In this study, nanoindentation is used to measure overall elastic moduli and hardnesses of three fundamental layers of a TBC system: metallic substrate and two overlaid plasma sprayed coatings on it. Elastic modulus results of TBC layers were used in the analysis of fracture toughness and energy release rates analysis in Chapters 3 and 4. The standard practice of an instrumented indentation application on metallic substrates was set by ASTM E2546-07 [2] and ISO 14577 series [37-40] standards. ASTM E2546-07 [39] standards was followed to calibrate two different nanoindenters used in this study which are CSM Nanoindenter in METU Central Laboratories and Hysitron Nanoindenter in Mustafa Kemal University Central Laboratories.

The entire measurements were done by Berkovich indenter tip at varying maximum loads applied by setting the loading and unloading time at 30 seconds and pause time 10 seconds to 20 seconds for coatings and metallic substrate respectively. The data analysis methods were determined after the preliminary measurements were taken for each layer from CSM nanoindenter in METU Central Laboratories.

The preliminary hardness and elastic modulus measurements on Inco718 substrate are very consistent. Therefore, the mean value of the series of measurements will be used as the characteristic modulus and hardness of Inco718 in further mechanical analysis.

The preliminary BC and TC measurements have a large scatter because of high porosity and heterogeneity. To come to a conclusive elastic modulus value on these layers, a Weibull distribution based statistical data analysis method was followed as the literature suggested [18, 19]. Series of measurement were done on the cross-section of the TC and BC layers of TBC specimens at 5 different maximum loads varying between 10mN to 500mN. The large scattered data were analyzed to come to a characteristic elastic moduli and hardness value for both TC and BC as introduced in the following section.

2.1.4.4. Scattered Data Analysis by Weibull Distribution

Weibull distribution is a widely used statistical method [76] to calculate mechanical properties of heterogeneous and porous materials which contain high scattered data sets, such as thermal barrier coatings [28, 30]. In this study, Weibull distribution function is used to determine conclusive values of E and H where a two parameter Weibull distribution function defines the probability, p, as,

$$p=1-\exp\left[-\left(\frac{x}{x_0}\right)^M\right], \quad (2.1.18)$$

where M, is Weibull constant, x is a given parameter or the measured data and x_0 is the characteristic value of the data set. The Weibull constant is a dimensionless number indicates the scatter and increases as the scatter decreases.

First of all, the survival probability of the i^{th} measurement of E and H in the data arranged in ascending order is expressed as,

$$p=(i-0.5)/N, \quad (2.1.19)$$

where N is the number of total observations. Then, a series of manipulations were done to linearize Eq. 2.1.18 with respect to the parameter, x, and probability defined in Eq. 2.1.19, as,

$$1-p=e^{-\left(\frac{x}{x_0}\right)^M}, \quad (2.1.20)$$

$$\ln\left(\frac{1}{1-p}\right)=\left(\frac{x}{x_0}\right)^M, \quad (2.1.21)$$

$$\ln\left[\ln\left(\frac{1}{1-(i-0.5)/N}\right)\right]=M[\ln(x)-\ln(x_0)], \quad (2.1.22)$$

$$f(i)=M (\ln(x)-\ln(x_0)). \quad (2.1.23)$$

The above equation is the linearized form of Eq. 2.1.18 which can be plotted on $f(i)$ vs. $\ln(x)$ plane where x indicates E or H for nanoindentation tests as in Fig 2.1.6. If the data series were linearly fitted by a least square method, the line with a slope of m represents the scatter of data and x_0 indicates the characteristic value of E and H which is the intersection of the fit line to x axis.

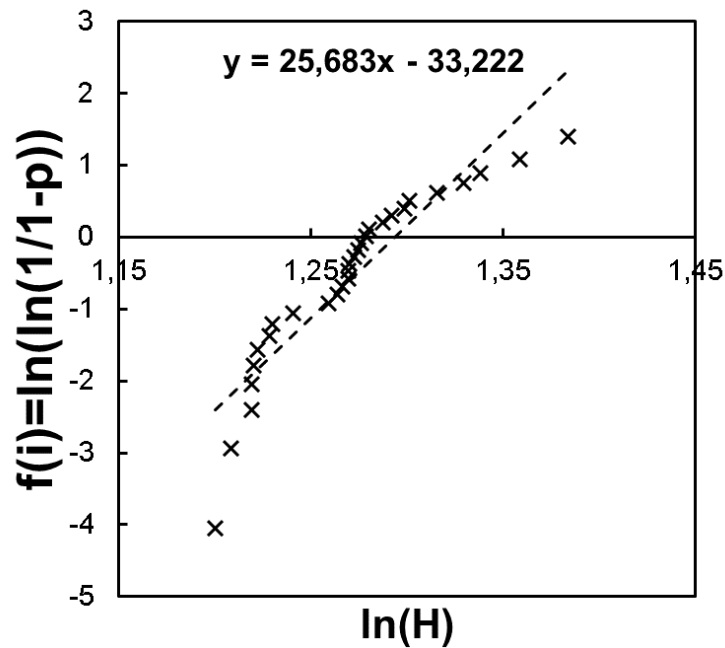


Figure 2.1.6 A typical plot based on Weibull distribution which is used in the analysis of scattered hardness data points to determine the characteristic value (x_0) of parameter H and the measure of scatter, M , which are 1.29 GPa and 25.683 for this plot, respectively.

2.2. Characterization

2.2.1. Powder Form of Top Coat and Bond Coat

EDS, XRD crystallography and EDS mapping techniques used to characterize the BC and TC powders in the manner of elemental content, crystal structure and elemental distribution.

The elemental content of BC and TC powders are demonstrated in Table 2.2.1 and Table 2.2.2, respectively, with weight and atomic distributions of the dominant elements. EDS results showed that BC powder is an alloy of nickel (Ni), chromium (Cr), aluminum (Al) and yttrium (Y). EDS results of TC powder showed that particles contain substantial amount of yttrium (Y), zirconium (Zr) and oxygen (O). The atomic proportion of oxygen and zirconium (2:1) and small amount of yttrium showed that the powder material is yttrium stabilized zirconium oxide (ZrO_2 -zirconia) ceramic.

Table 2.2.1 Weight and atomic percentage distribution of the elemental content of the bond coat powder particles.

	Ni	Cr	Al	Y
% Wt	62.17	22.31	14.02	1.48
% At	82.96	33.61	40.74	1.30

Table 2.2.2 Weight and atomic percentage distribution of the elemental content of the top coat powder particles.

	Y	Zr	O
% Wt	7.88	68.50	23.61
% At	3.82	32.40	63.74

In Fig. 2.2.1 and Fig. 2.2.2, the elemental scattering of BC and TC powders are presented respectively with respect to the dominant elemental contents by using EDS (or EDX) mapping. It is important to notice that EDS mapping is a time-consuming and non-precision technique, for instance the trace amount of yttrium in the bond coat powder cannot be observed in the mapping of bond coat particle in Fig.2.2.1. The EDS mapping analysis was done on powder particles stuck on a carbon tape. Therefore carbon distribution in Fig. 2.2.1(f) and Fig.2.2.2 (e) are showing the spacing between powder particles. Moreover, as a result of the angled position of the sensor with respect to the mapped surface, first there appear shadows on particles and carbon tape as in Fig.2.2.1(f), second the up-left surfaces of particles are over-mapped, as in Fig. 2.2.2(c). In Fig. 2.2.1, the fundamental elements of BC powder: nickel, chromium and aluminum are equally distributed through particles. Furthermore, there appear some spots of oxygen on the particles which may refer to small oxide spots. In Fig. 2.2.2, the fundamental elements of TC powder: yttrium, zirconium and oxygen are equally distributed through particles. In contrast to elemental map of BC powder, yttrium can be observed since it is dominant in the TC powder. To sum up, EDS mapping of BC and TC powder particles showed that both powders are elementally homogeneous which TC and BC layers can be considered chemically homogeneous after production.

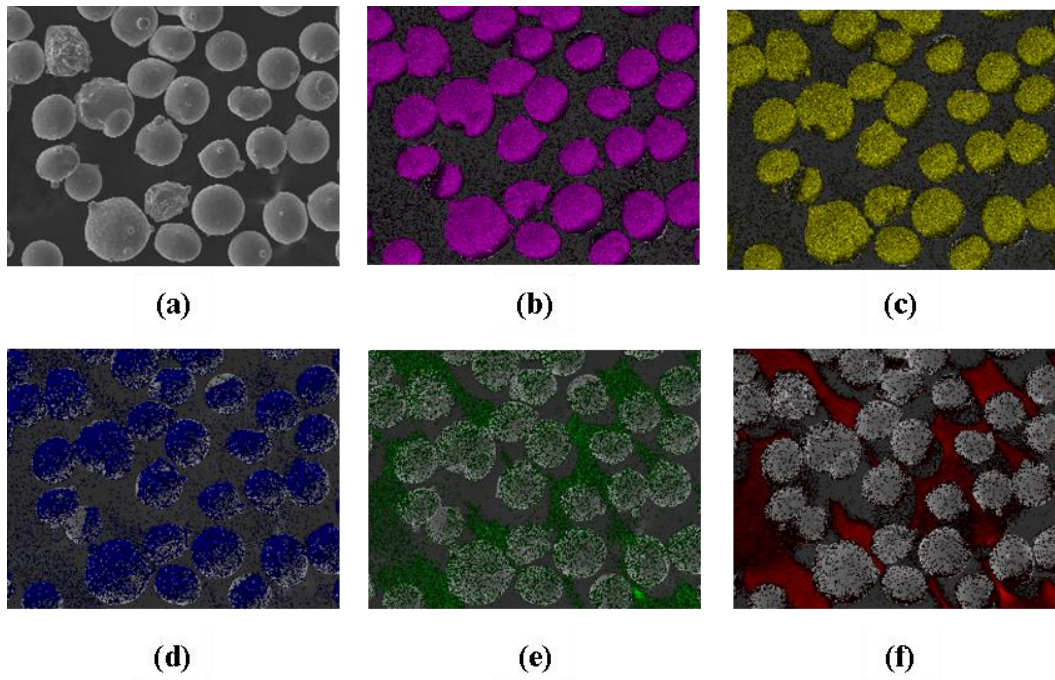


Figure 2.2.1 EDS mapping results at 600X magnification showing the elemental scattering of (a) bond coat powder particles in terms of (b) nickel, (c) chromium, (d) aluminum, (e) oxygen and (f) carbon.

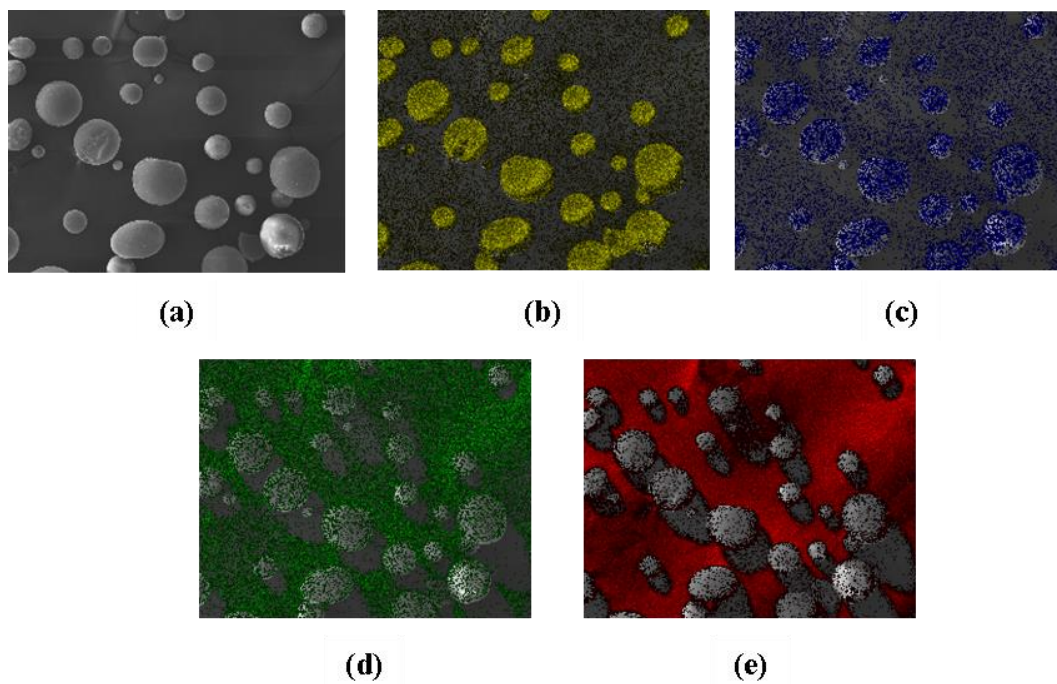


Figure 2.2.2 EDX mapping results at 600X magnification showing the elemental scattering of (a) top coat powder particles in terms of (b) zirconium, (c) yttrium, (d) oxygen and (e) carbon.

BC and TC powder high-pressure 300X SEM images are presented in Fig. 2.2.3. In the manner of shape and size, there are fundamental differences between BC and TC powders. First, BC powder particles have perfect spherical shape where most of the TC powder particles are egg-shaped. Second, BC powder particles are sized between 50 μm and 100 μm where TC powder contain miscellaneous sizes between 12 μm and 87 μm . Third, there are surface defects, shape irregularities and cracks on TC powder particles. These imperfections cause irregular microstructure and increase the air-gap in the as-sprayed specimens.

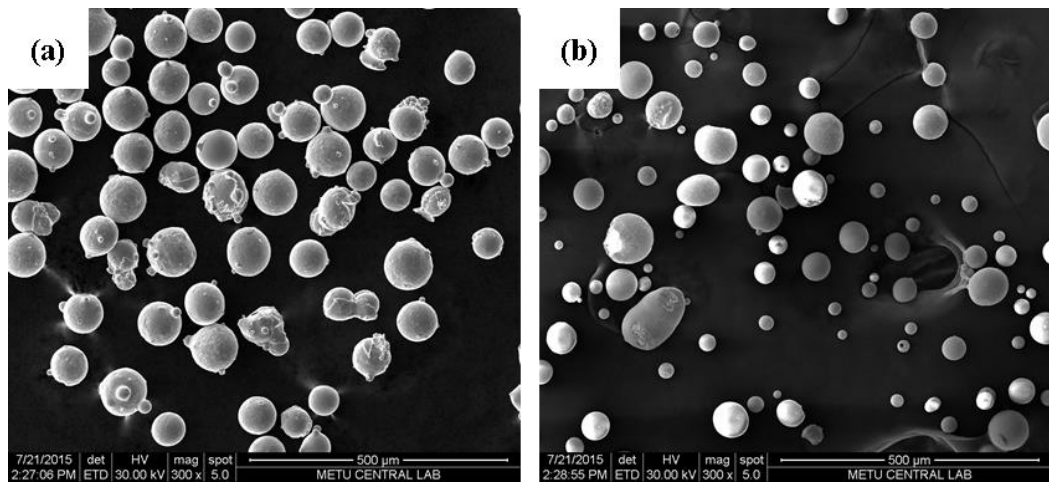


Figure 2.2.3 High pressure 300X SEM images of powder particles used to manufacture (a) bond coat (BC) and (b) top coat (TC).

2.2.2. Microstructural Analysis of As-sprayed TBC System

TBC coated rectangular (4 X 2 cm) coupons were manufactured for three different sets of specimen for three different coating thicknesses. Specimen sets having different coating thicknesses in Fig. 2.2.4 a, b and c will be mentioned as *thin*, *standard* and *thick*, respectively. Specimens in each set manufactured under same conditions at the same time. One specimen from each thickness was reserved for microstructural analysis.

The overall layer thicknesses of specimen sets are presented in Table 2.2.3 with the thickness deviation of each layer. Between different coating thicknesses, layer thickness distribution stays constant. Cross-sectional microscopies of thin, standard and thick specimens are shown in Fig. 2.2.4(a), Fig. 2.2.4(b) and Fig. 2.2.4(c), respectively. Surface roughness of TC and BC were calculated from 1 mm portion of each microscopy. The roughness are very close to the thickness deviation of layers. This meant that the roughness of plasma sprayed layers are equally distributed.

Table 2.2.3 Overall thickness and deviation between thin, standard and thick sets of TBC specimen

	Thin	Standard	Thick
TC	260 ± 24 µm	374.4 ± 50.3µm	569.4 ± 44 µm
BC	54 ± 35.5 µm	106.5 ± 30.5 µm	169.1 ± 32.3 µm
S	1597.5 ± 10.9 µm	1602.55 ± 6.9 µm	1580.8 ± 12.2 µm

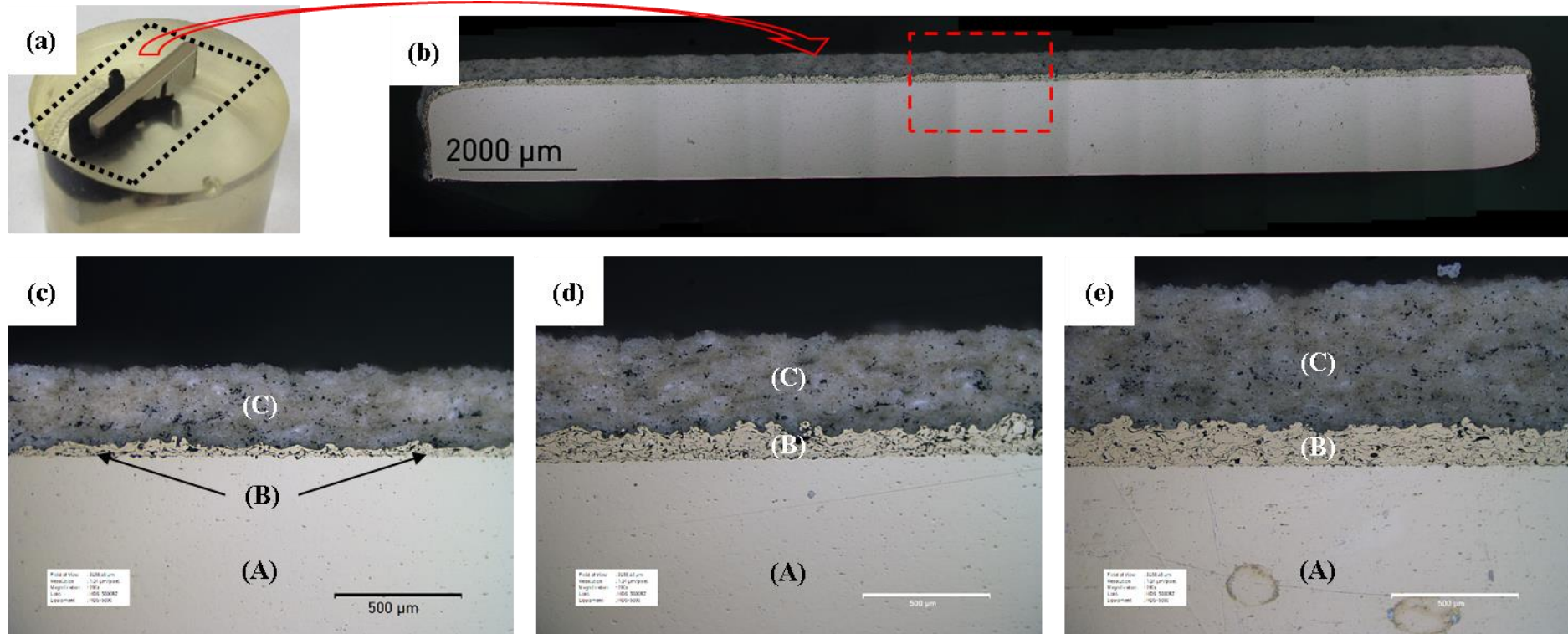


Figure 2.2.4 (a) A typical mounted and polished TBC specimen ready for the cross-sectional microscopy and (b) a tiled cross-sectional microscopy of a TBC specimen showing the region of microscopies in b, c and d. Cross-sectional microstructure of TBC containing superalloy substrate (A) for three different bond coat (B) and top coat (C) thicknesses of b) 55, 250 μm, c) 100, 375 μm and d) 165, 575 μm, which are labeled as thin, standard and thick, respectively.

BC and TC were applied on a nickel-based sheet superalloy substrate: Inconel 718 (Inco718). Before the BC was applied, the surface energy of the substrate is increased by shot peening to improve the adhesion between BC and substrate. As it is shown in Fig. 2.2.5, this application create undulations at the BC/S interface. As a result, interface flaws appear at all along the BC/S interface. Air gaps inheriting between the wrinkles of BC/S interface were observed for all specimen sets.

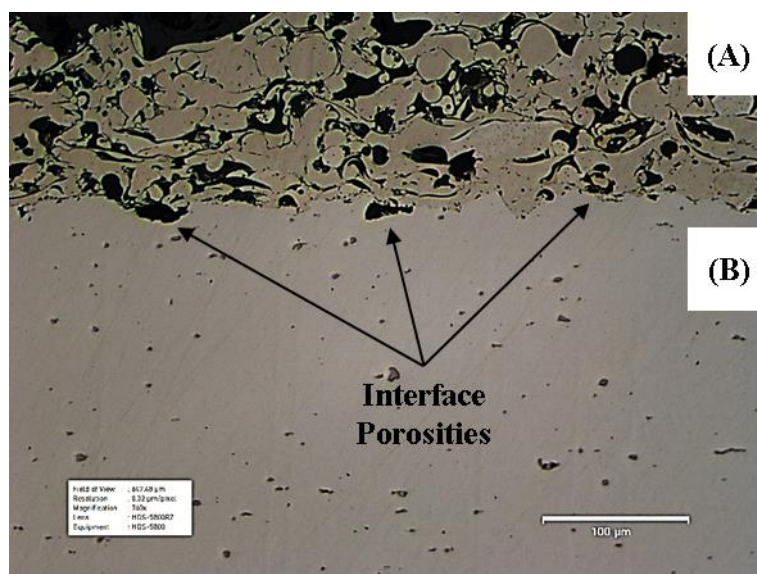


Figure 2.2.5 Microstructure of typical interface flaws appear in the vicinity of undulations at the interface between bond coat (A) and substrate (B).

Microscopy of TC/BC interface of an as-sprayed specimen is shown in Fig. 2.2.6. Typically, interface porosities, interface cracks and thermally grown oxide layer appear at the TC/BC interface and contribute to the mechanics of TBC system. Moreover, TC/BC interface roughness and flaws are the key feature of the failure mechanisms of TBC from the interface. Interface air gaps/porosities inherit TC/BC interface as well they inherit at the BC/S interface. The size of air gaps increase as the roughness increases. Some of these porosities are shallow enough to the interface that these features directly act like pre-cracks under-loading.

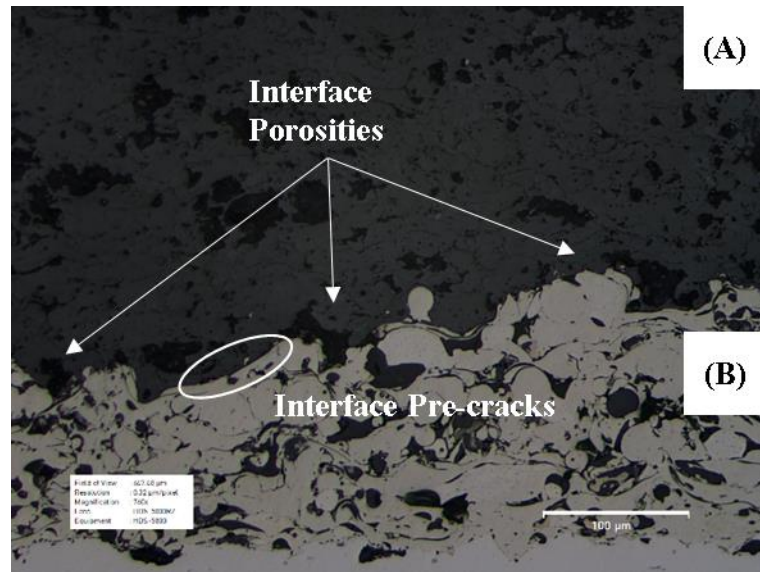


Figure 2.2.6 Microstructure of typical flaws appear in the vicinity of the interface between top coat (A) and bond coat (B).

To grow the TGO layer, a couple of TBC specimen was exposed to 1300 °C for 24 hours in a temperature controlled furnace, then cooled down to the room temperature. One of them is mounted and prepared for microstructural analysis. In Fig. 2.2.7 dark field microscopy images of the as-sprayed TBC specimen (a) and the thermally exposed TBC specimen (b) are shown and as advantage of DF microscopy only the ceramic ingredients are visible. In Fig. 2.2.7(a), a healthy TC is apparent with its white color, but in the left image, there are apparently two layers: TC (A) (white) and TGO layer (B) (dark blue). The specimen, in Fig. 2.2.7(a) demonstrates a typical microstructure of an as-sprayed TC. However, in Fig. 2.2.8(b) the microstructure of thermally exposed TBC specimen obviously degraded when it is compared to the as-sprayed condition. Thermal exposure degraded TC and the ceramic TC crumbled and also got thinner. However, TGO layer appeared at the TC/BC and measured as 10 μm. There is also a massive interface crack goes all along the TGO/TC interface. In Fig. 2.2.8, before (a) and after (b) conditions of the unmounted couple of thermally exposed specimen can be seen. The interface crack propagated and TC of the unmounted couple of thermally exposed specimen popped out under no-loading and in room temperature.

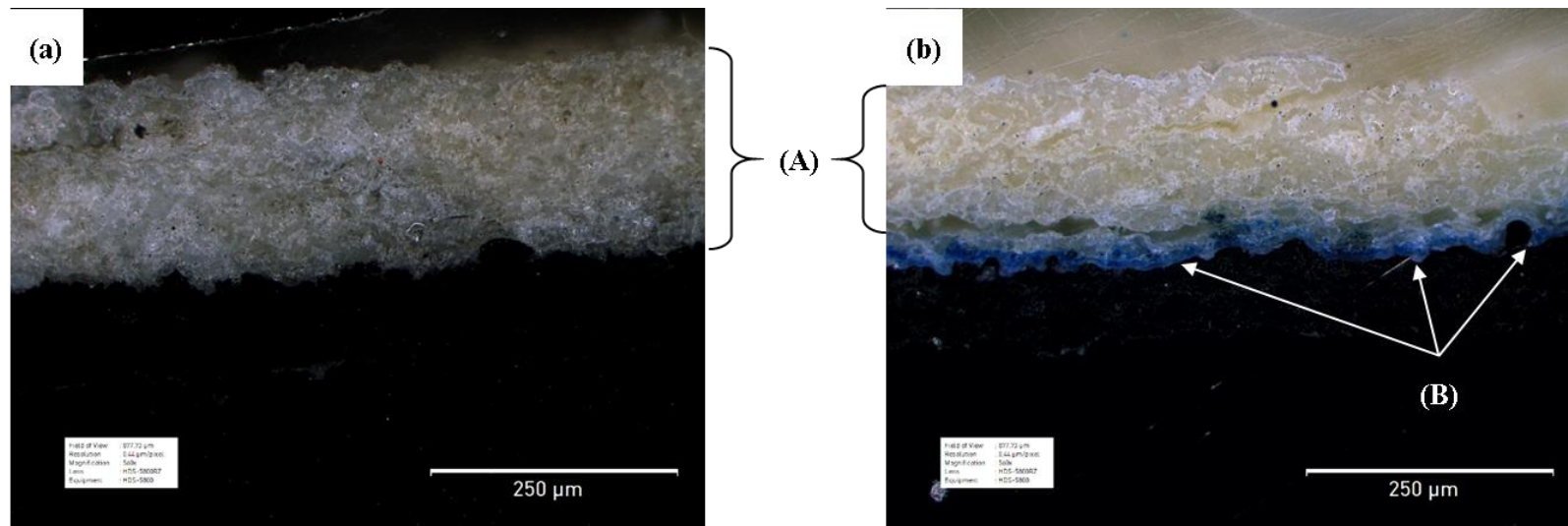


Figure 2.2.7 Cross-sectional dark-field microscopy of (a) TC (A) layer of an as-sprayed TBC specimen and (b) TC (A) and TGO (B) layers of 24 hours of thermally exposed TBC specimen at 1300 °C.

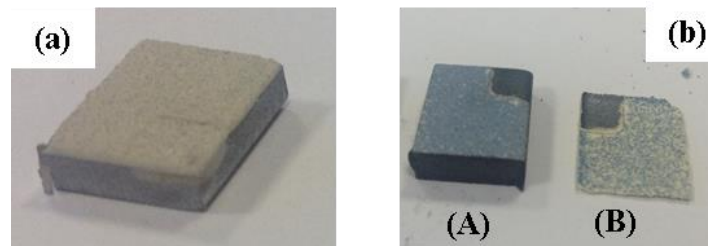


Figure 2.2.8 (a) As-sprayed TBC specimen before thermal exposure and (b) popped out TC (A) and BC-S couple (B) after 24 hours of 1300 °C exposed TBC specimen.

Microstructures of TC and BC are shown in Fig. 2.2.9(a) and Fig. 2.2.9(b, c), respectively. Both TC and BC contain similar flaws, such as porosities and pre-cracks and have similar microstructural features. Both layers have a porous and splatted structure as a result of APS technique. The splatted structure of BC is shown in Fig. 2.2.9(c) to make it more visible by an etching process with Kalling No.2 solution. Air gaps between splats can be seen in Fig. 2.2.9(a) and Fig. 2.2.9(b). Both layer contain intersplat micro cracks. These cracks are fundamentally different from the intersplat porosities. The inter splat pre-cracks appear because of the thermal mismatches between splats during production.

The surface quality of BC is an indicator of fracture resistance of the TBC. There are imperfections in the BC, these are porosities between splats, intersplat pre-cracks and interface porosities. There are existing interface porosities but these are very rare microstructural imperfections. In TC, it is well known that there is a splat like structure. But the porosity of this layer covers the splat like structure.

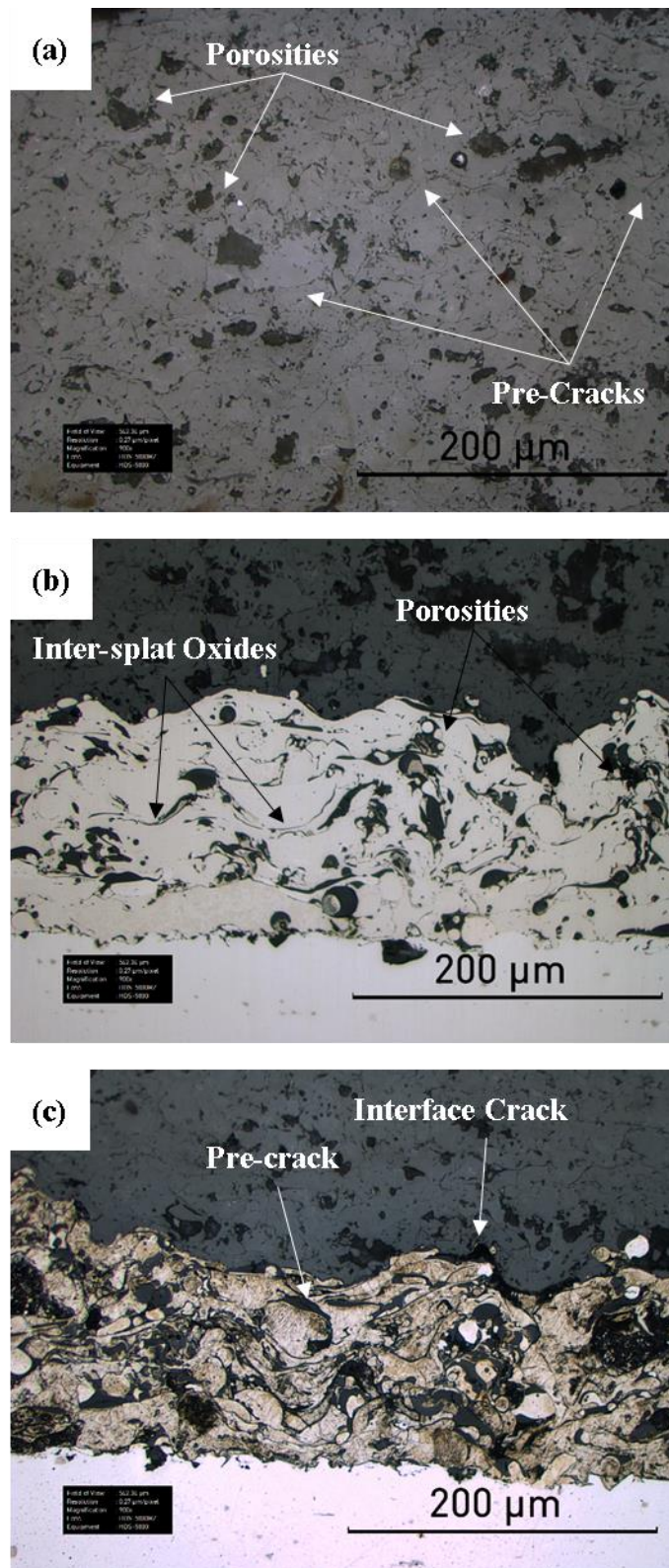


Figure 2.2.9 Microstructure of top coat (a), bond coat (b) and etched bond coat (c) showing typical layer flaws: porosities, pre-cracks and inter-splat oxides.

2.2.3. Determination of Elastic Moduli and Hardness of TBC Layers

Elastic modulus and hardness of TBC layers were determined by using Oliver and Pharr method and data obtained from nanoindentation tests. Different statistical approaches introduced in the previous section for each layer, which were used to come to conclusive values of elastic modulus and hardness.

2.2.3.1. Substrate: Inconel 718

Inconel 718 sheet (Inco718) nickel-based superalloy was used as the substrate material of the TBC system. Inco718 indentations were conducted at the cross-section of mounted specimens following the standards for instrumented indentation on metals [39]. Nanoindentations were conducted at 150 mN maximum load applied and 20 s of pause while keeping the loading and unloading time at 30 seconds. A typical penetration depth vs. load curve obtained from one of the Inco718 tests is presented in Fig. 2.2.10(a) showing the corresponding optical images of the resultant indent (Fig. 2.2.10(b)). Every indentation impression on Inco718 are symmetric and equally sized same as in Fig. 2.2.10(b), which means that the indenter tip perpendicularly penetrated the specimen surface.

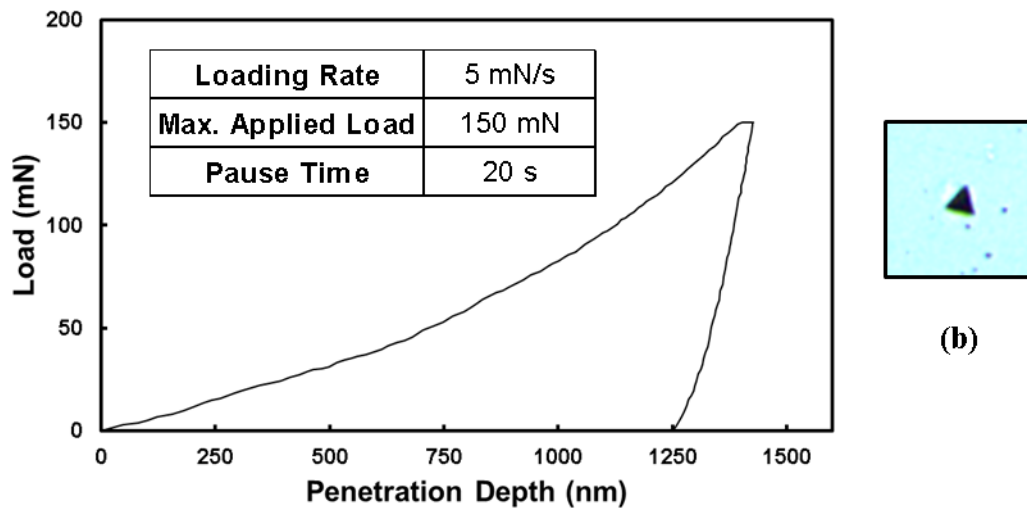


Figure 2.2.10 (a) Penetration depth (Pd) vs. load curve of a nanoindentation test on Inconel 718 substrate and the resultant indent (b) after indentation applied at 150 mN by 5 mN/s loading/unloading rate and 20 second of pause at maximum load.

Nine sets of indentations, containing total of 29 indentations were conducted by CSM Nano-hardness Tester (NHT) [17] at METU Central Laboratories on different days. One data set was disregarded due to uncalibrated measurements and 2 data sets were disregarded due to high deviation. The averaged data of measurements presented in Fig. 2.2.12(a) and Fig.2.2.12 (b) and raw data are tabulated in Fig. 2.2.12(c). Average of 9 measurements obtained from Hysitron TI950 Triboindenter [67] (Mustafa Kemal University, Hatay) is also shown in the table where the measurement is conducted at 50 mN.

CSM and Hysitron results are consistent for elastic modulus measurements. However, since the hardness of materials is dependent to depth, the hardness data are not alike. Hysitron data were obtained from Resultants are converging around 200 GPa for elastic modulus and 3.8 for hardness. Indentation modulus and hardness are plotted in Fig. 2.2.11 using the 26 data points. The average of indentation hardness and indentation moduli which are designated as dashed lines in Fig. 2.2.11, are found to be 3.57 GPa and 205 GPa, respectively. For homogeneous metals, nanoindentation results at same conditions should be focused around their average EIT and HIT. EIT vs HIT plot is used to recognizing the mistaken data and tests and to eliminate these data from analysis.

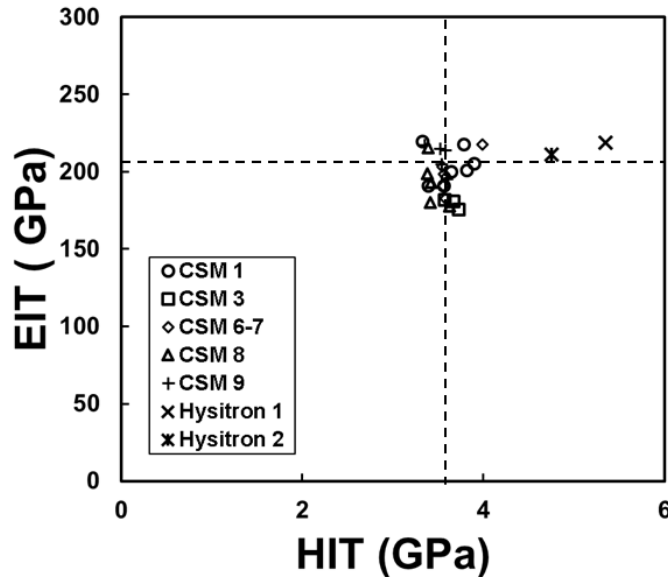


Figure 2.2.11 Indentation hardness (HIT) vs. indentation modulus (EIT) plot of Fig. 2.12c showing data scattering around the average values of EIT (205 GPa) and HIT (3.57 GPa) which were indicated as dashed lines.

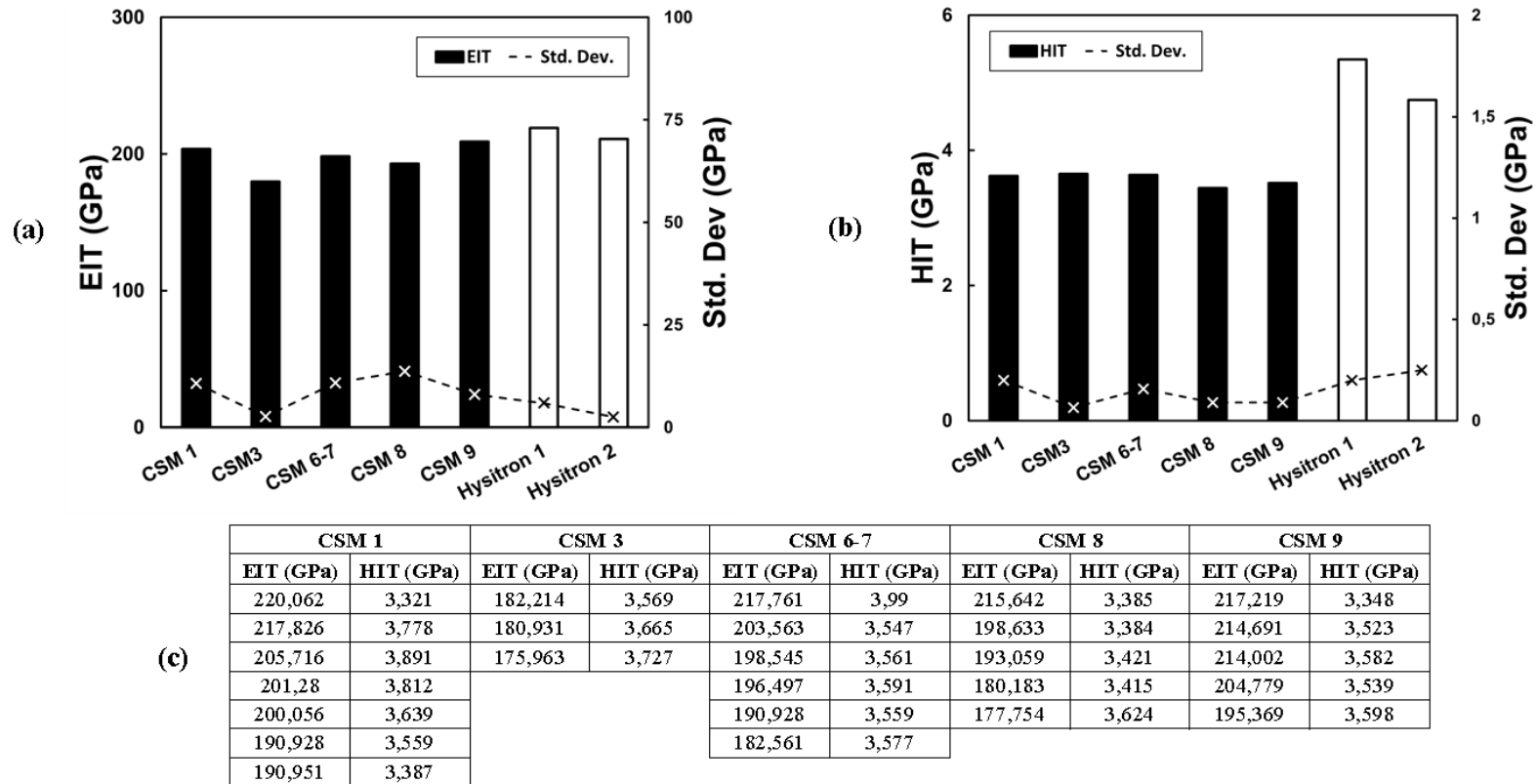


Figure 2.2.12 (a) Average indentation moduli of Inconel 718 measured from a group of tests conducted on CSM and Hysitron nanoindenters in conjunction with the standard deviation of EIT at each test, (b) Average indentation hardnesses of Inconel 718 measured from a group of tests conducted on CSM and Hysitron nanoindenters in conjunction with the standard deviation of HIT at each test, (c) EIT and HIT data sets used in (a) and (b) measured by CSM nanoindenter.

Detailed material data of Inco718 that contains the elastic properties is available on material data sheet [36]. Young's modulus of Inco718 is 29000 ksi at 70 °F and 29800 ksi at 100 °F corresponding to 199.94 GPa at 21 °C and 205.46 at 31 °C. Nanoindentations were conducted under 25 °C to 34 °C at most. Elastic modulus obtained from nanoindentation has %2.5 deviation from the stated elastic modulus value for design.

2.2.3.2. APS Layers: NiCrAlY Bond Coat and YSZ Top Coat

There are two different layers manufactured by air plasma spraying technique. Both layers have porous structures and require different indentation technique than the tests conducted for the metallic substrate Inconel 718. Both BC and TC indentation tests were conducted according to instrumented roadmap described in [46]. First trend charts of EIT and HIT were plotted which contain small sets of indentations conducted at different maximum loads then a stabilization load was decided and large sets of tests were conducted to determine characteristic values of EIT and HIT of layers from Weibull distribution method. Every single indentation test was conducted by the same test cycle: loading and unloading of the material was applied in 30 seconds and paused for 10 seconds at maximum load.

A typical penetration depth vs. load curves obtained from one of BC and TC indentations presented in Fig. 2.2.13(a) and in Fig. 2.2.14(a), respectively. Moreover, microscopy of the resultant indents of BC and TC indentations were also presented in in Fig. 2.2.13(b) and in Fig. 2.2.14(b), respectively. Only the curves without any dents were used in statistical analysis. Every indentation impression was checked by the integrated microscope in case of any symmetry problem and size variation between indents (clue for dent).

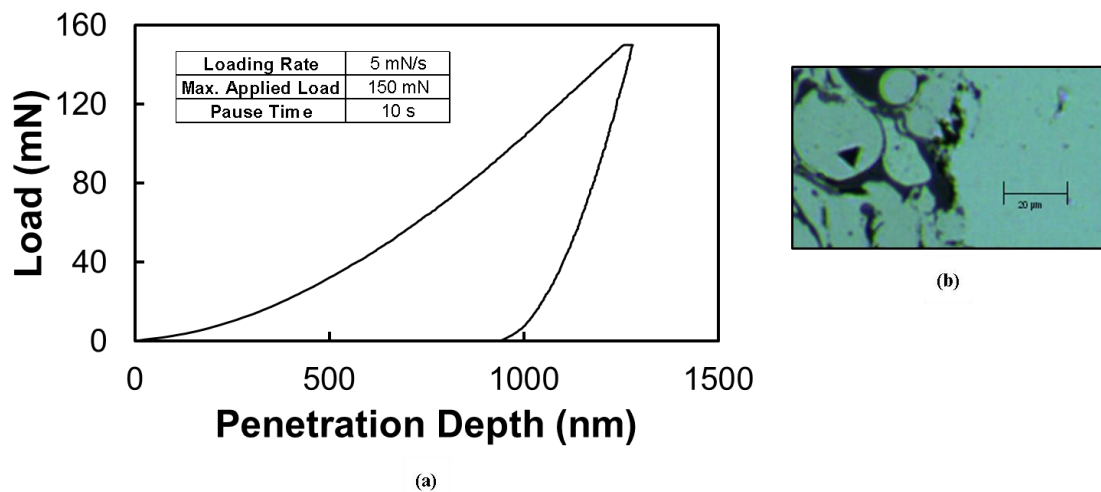


Figure 2.2.13 (a) Penetration depth (Pd) vs. load curve of a nanoindentation test on NiCrAlY bond coat and the resultant indent (b) after indentation applied at 150 mN by 5 mN/s loading/unloading rate and 10 second of pause at maximum load.

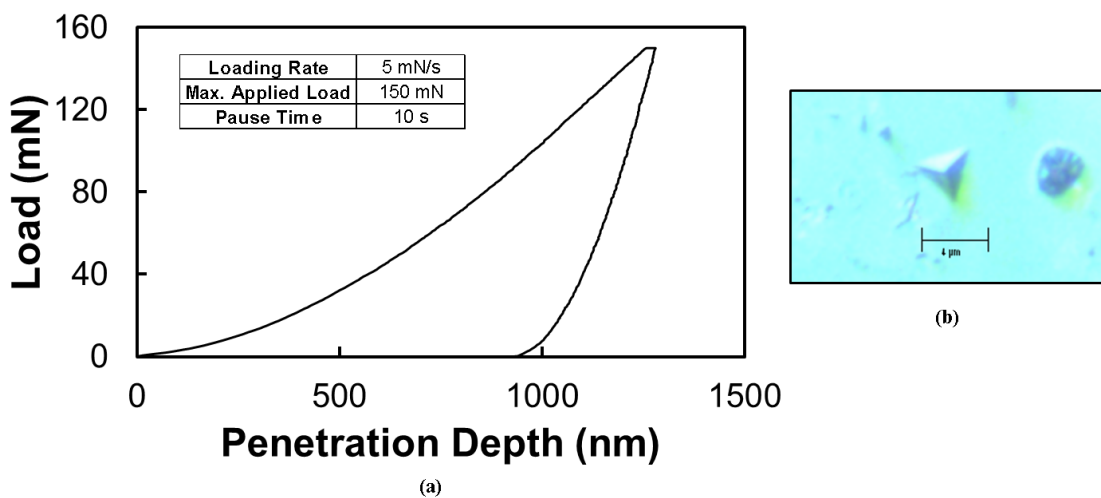


Figure 2.2.14 (a) Penetration depth (Pd) vs. load curve of a nanoindentation test on YSZ top coat and the resultant indent (b) after indentation applied at 150 mN by 5 mN/s loading/unloading rate and 10 second of pause at maximum load.

Eight indentations were done at five different maximum loads for two APS layers to determine the critical load where porosity, heterogeneity and size effects diminish. Variation of EIT and HIT with the changing the applied load was shown in Fig. 2.2.15(a) and Fig. 2.2.15(b), respectively. In both plots, there are five data points which represent the overall EIT and HIT measurements from 8 indentations.

In Fig. 2.2.15(a), there is a dramatic drop in the measured EIT data as the applied load increases. This behaviour was also presented in Inconel 718 measurements where Hysitron measurements were little bit higher than the CSM measurements as the applied maximum loads are 50 mN and 150 mN, respectively. The overall EIT data drops until 150 mN and between 150 mN and 300 mN it converges to an overall value. This behaviour of porous materials under indentation is very similar to the size effect mentioned in [46].

In Fig. 2.2.15(b), there is a continuous drop in the measured HIT data as the applied load increases. For BC, overall value drop is not as drastic as TC because BC is less sensitive to size effect by its metallic structure. This phenomena is also well explained in [46]. The HIT results came as how they expected.

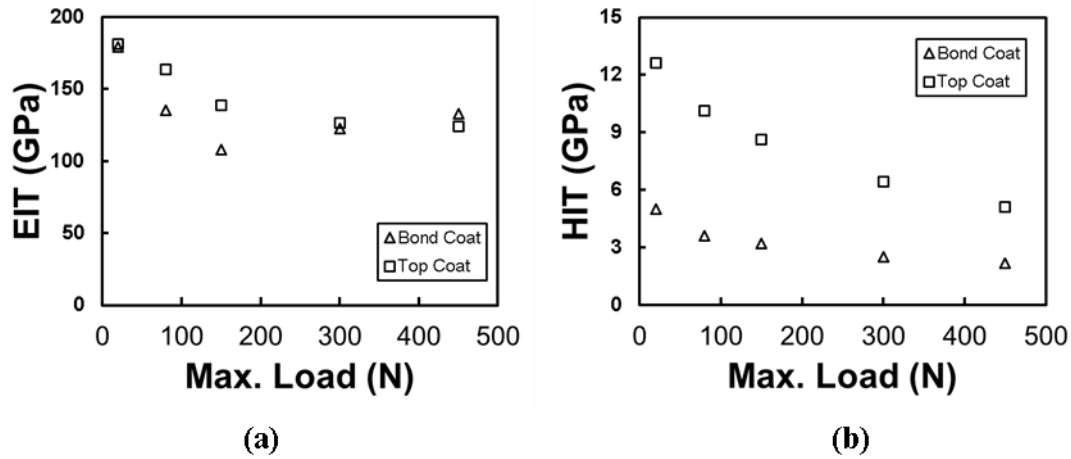


Figure 2.2.15 Average (a) indentation moduli (EIT) and (b) indentation hardness (HIT) results of BC and TC from 40 indentations total at five different maximum loads.

2.2.3.2.1. Bond Coat: NiCrAlY

From the preliminary measurements conducted at different loads, it was shown that consistent EIT and HIT measurements can be achieved between 150 mN and 300 mN applied load. Therefore, as a second phase of the BC measurements, thirty-five indentations were conducted at 300 mN maximum load. In Fig. 2.2.17, Weibull distribution plots are presented which are also used in the determination process of the characteristic EIT and HIT values for BC. In the following plots, each indentation was added into the Weibull distribution plots as explained in the methodology part of this chapter and each EIT and HIT data designated as a cross. In both figures, the data points are linearly fit and the equation of this line was also attached at the top of each plot.

According to the data set containing 35 measurements, the characteristic indentation modulus of NiCrAlY based bond coat is 115.6 GPa where the mean of the data set is 108 GPa with 17.9 GPa standard deviation. And, the characteristic indentation hardness of BC is 3.27 GPa where the mean of the data set is 3.05 GPa with 0.57 GPa standard deviation.

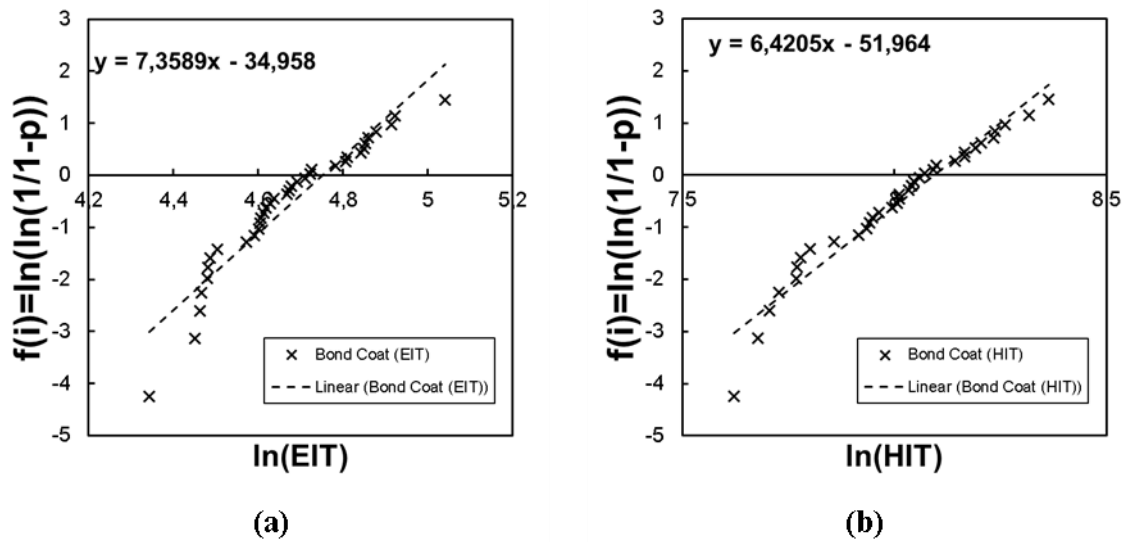


Figure 2.2.16 Weibull distribution plots of a set of indentations at BC layer containing 35 indentations at 300 mN maximum load applied at the rate of 600 mN/s and 10 seconds of pause time for (a) indentation moduli (EIT) and (b) indentation hardness (HIT).

2.2.3.2.2. Top Coat: YSZ

From the preliminary measurements, it was shown that consistent EIT and HIT measurements can be achieved between 150 mN and 300 mN applied load same as bond coat. Therefore, at the second phase measurements fifty indentations were conducted at 300 mN maximum load. Weibull distribution plots are presented in Fig. 2.2.18(a) and (b) for EIT and HIT measurements, respectively. In the following plots, each EIT and HIT data designated as a cross. In both figures, the data points are linearly fit and the equation of this line was also attached at the top of each plot.

According to the data set containing 50 measurements, the characteristic indentation modulus of YSZ based top coat is 118 GPa where the mean of the data set is 110 GPa with 19.86 GPa standard deviation. And, the characteristic indentation hardness of TC is 7.07 GPa where the mean of the data set is 6.39 GPa with 2.06 GPa standard deviation.

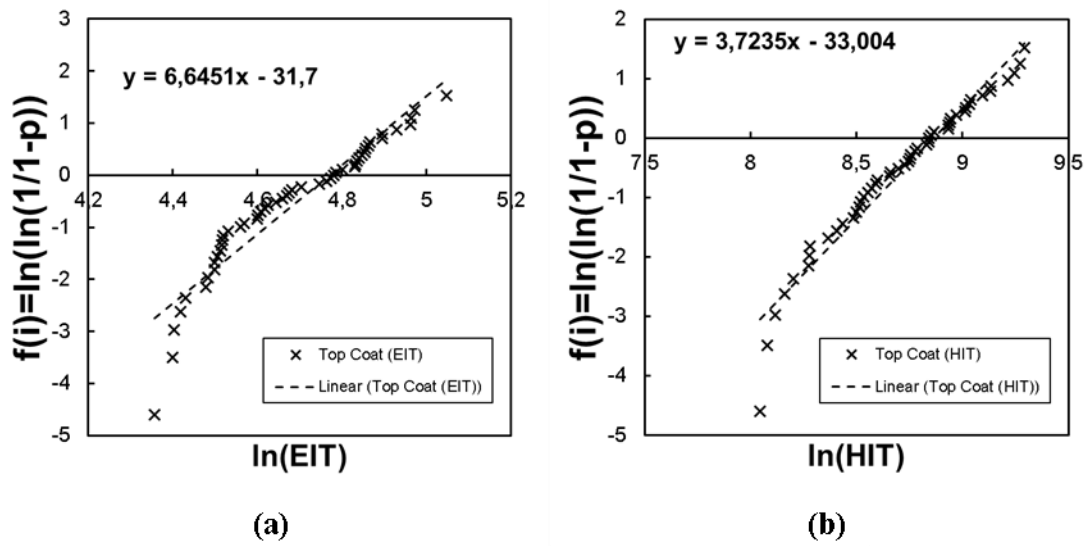


Figure 2.2.17 Weibull distribution plots of a set of indentations at TC layer containing 50 indentations at 300 mN maximum load applied at the rate of 600 mN/s and 10 seconds of pause time for (a) indentation moduli (EIT) and (b) indentation hardness (HIT).

CHAPTER 3

DEVELOPMENT OF 4-PT BENDING TEST METHODOLOGY FOR INTERFACE FAILURE

In this chapter, two different approaches to find an interface toughness and energy release rate of a bi-material composite beam interface under 4-pt bending are validated by using an Al-PMMA bi-material composite beam. First, the geometry of a typical specimen and the loading case is introduced. Second, an Euler-Bernoulli Beam Theory based energy release rate formula for a composite beam is derived for four point bending case. Third, fracture mechanics based mixed-mode interface toughness and energy release rate formula is derived as briefly introduced in [11]. Then, the benchmark test setup and the literature review of the instrumentation used in the validation test is introduced. Finally, tests conducted for an Al-PMMA fracture toughness and energy release rate of the Al-PMMA interface was calculated from tests conducted by using 2D DIC and an in-house-built acoustic emission crack detector by using the correlation. Then, the availability of these correlations is discussed for a relatively small-scale 4-pt bending tests of TBCs.

3.1. Geometry

In Fig. 3.1.1, a bi-material composite flexural beam is demonstrated. The bi-material beam consist of a notch cutting the material 1 to two pieces at the opening side of the loading and a symmetric pre-crack lying along both sides of the interface which is positioned inside the inner loading points. The pre-crack is subjected to a constant opening moment between the inner loading points.

The failure of this system should exhibit steady state failure characteristics and a plane strain conditions.

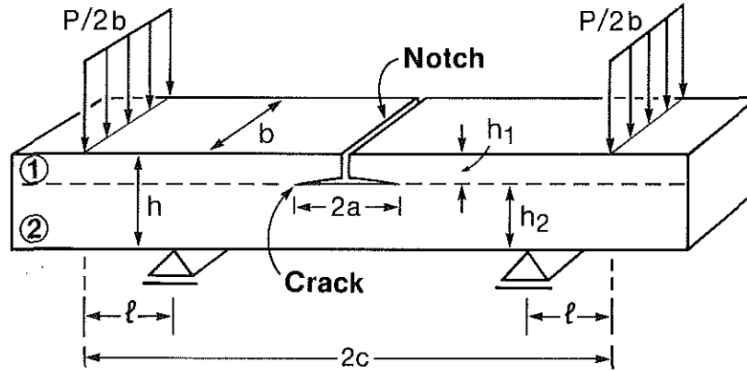


Figure 3.1.1 Illustration of a typical bi-material beam with a notch and an interface pre-crack under 4-pt bending [11].

3.2. Analytic Solution Based on Euler-Bernoulli Beam Theory for Energy Release Rate of a Composite Beam under Four-Point Bending

The geometry introduced in Fig. 3.1.1 is symmetric with respect to the notch. Therefore, a free-body diagram of half of the beam is sufficient for further analysis. In Fig. 3.2.1., the free-body diagram and the portions of the beam is demonstrated to proceed with Euler-Bernoulli Beam Theory based analysis. The beam depicted in Fig.3.2.1 (a) can be portioned into three pieces from where the interface crack ends. The first portion is a beam composed of two materials without any interface crack. The second portion of the beam is the notched and cracked beam made of only material 1. Each side of this beam is traction free except the left hand side. As a result, this beam carries no load in Fig. 3.2.1(b). The third portion is the cracked part of the material 2 which can carry load at its both ends.

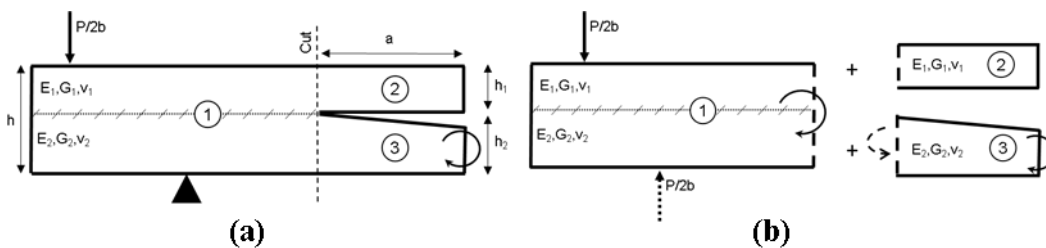


Figure 3.2.1 (a) Free-body diagram of half of the composite bi-material beam illustrated in Fig. 3.1.1 (b) Superposed parts of the composite beam used in the Euler-Bernoulli Beam Theory based energy release rate formulation.

Each of these beams contains a strain energy with respect to load they carry. From Euler-Bernoulli Beam Theory the strain energy density can be expressed as,

$$U=(1-\nu^2)M^2/2EI. \quad (3.2.1)$$

When the above equation is applied on the system defined in Fig. 3.2.1, the total strain energy of the system can be found as a function of crack length, “a”. It is well known that the energy release rate can also be defined as the total energy needed to create new surfaces [46]. Therefore, the energy release rate of a composite beam with an initial interface crack with a length of a can be defined as,

$$G_{ss}=\frac{\partial U}{\partial a}=\frac{M^2(1-\nu_2^2)}{2E_2}\left(\frac{1}{I_2}-\frac{\lambda}{I_c}\right), \quad (3.2.2)$$

where,

$$\lambda=\frac{E_2(1-\nu_1^2)}{E_1(1-\nu_2^2)}. \quad (3.2.3)$$

As the moment is constant at the magnitude of $Pl/2b$, the analytic solution of the energy release rate can be expressed as a function of material properties (E_1 , E_2 , ν_1 , ν_2), geometry (I_1 , I_2 , I_c , h_1 , h_2 , l) and the applied load (P)

$$G_{ss}=\frac{P^2l^2(1-\nu_2^2)}{8b^2E_2}\left(\frac{1}{I_2}-\frac{\lambda}{I_c}\right), \quad (3.2.4)$$

where

$$I_c=\frac{h_1^3}{12}+\frac{\lambda h_2^3}{12}+\frac{\lambda h_1 h_2 (h_1+h_2)^2}{4(h_1+\lambda h_2)}, \quad (3.2.5)$$

$$I_2=\frac{h_2^3}{12}. \quad (3.2.6)$$

Since this is a mixed-mode cracking, both complex stress intensity factor and crack tip opening displacements can be written in exponential form as follows,

$$K_I + iK_{II} = |K|e^{i\psi}, \quad (3.3.3)$$

where “ ψ ” is the phase angle of stress intensity factor, and

$$\Delta u_y + i\Delta u_x = \left[(\Delta u_x)^2 + (\Delta u_y)^2 \right]^{\frac{1}{2}} e^{i\phi}, \quad (3.3.4)$$

where “ ϕ ” is the phase angle of the crack-tip displacement function.

Then Eqs. 3.3.1, 3.3.2, 3.3.3 and 3.3.4 can be combined to find a crack tip displacements equation can be written as follows,

$$\left[(\Delta u_x)^2 + (\Delta u_y)^2 \right]^{\frac{1}{2}} e^{i\phi} = \frac{2 \left[\frac{1-\nu_1}{G_1} + \frac{1-\nu_2}{G_2} \right] K \sqrt{r/2\pi} r^{i\epsilon}}{(1+2i\epsilon) \cosh(\pi\epsilon)}, \quad (3.3.5)$$

$$\left[(\Delta u_x)^2 + (\Delta u_y)^2 \right]^{\frac{1}{2}} e^{i\phi} = \frac{\sqrt{2/\pi} \left[\frac{1-\nu_1}{G_1} + \frac{1-\nu_2}{G_2} \right] |K| e^{i\psi} r^{i\epsilon + \frac{1}{2}}}{(1+2i\epsilon) \cosh(\pi\epsilon)}. \quad (3.3.6)$$

To simply this equation, the highlighted complex terms have to be written in an exponential form. To do this following fundamental calculus and complex number manipulations should be followed. A complex number can be defined in many ways as,

$$z = x + iy = (x^2 + y^2)^{\frac{1}{2}} \cdot (\cos(\theta) + i \sin(\theta)) = R \cdot e^{i\theta}, \quad (3.3.7)$$

where

$$\theta = \arctan\left(\frac{y}{x}\right), \quad (3.3.8)$$

$$R = (x^2 + y^2)^{\frac{1}{2}}. \quad (3.3.9)$$

The highlighted complex term at the numerator in Eq. 3.3.6 is manipulated as follows,

$$r^{i\epsilon + \frac{1}{2}} = R \cdot e^{i\theta}, \quad (3.3.60)$$

$$\left(i\epsilon + \frac{1}{2}\right) \ln(r) = \ln(R) + i\theta, \quad (3.3.71)$$

$$\ln(R) = \ln(r^{\frac{1}{2}}) \quad \text{and} \quad \theta = \ln(r) \epsilon, \quad (3.3.82)$$

where

$$r^{i\epsilon + \frac{1}{2}} = r^{\frac{1}{2}} \cdot e^{i\epsilon \ln(r)}. \quad (3.3.93)$$

Aside, the highlighted complex term in the denominator in Eq. 3.3.6 is also manipulated as follows,

$$1 + 2i\epsilon = x + iy = (x^2 + y^2)^{\frac{1}{2}} \cdot e^{i \arctan\left(\frac{y}{x}\right)}, \quad (3.3.104)$$

$$x = 1 \quad \text{and} \quad y = 2\epsilon, \quad (3.3.115)$$

where

$$1 + 2i\epsilon = (1 + 4\epsilon^2)^{\frac{1}{2}} \cdot e^{i \arctan(2\epsilon)}. \quad (3.3.16)$$

After the complex number manipulations where done, the Eq. 3.3.6 become as,

$$\left[(\Delta u_x)^2 + (\Delta u_y)^2 \right]^{\frac{1}{2}} e^{i\phi} = \frac{\sqrt{2/\pi} \left[\frac{1-v_1}{G_1} + \frac{1-v_2}{G_2} \right] |K| e^{i\psi} r^{\frac{1}{2}} e^{i\epsilon \ln(r)}}{(1 + 4\epsilon^2)^{\frac{1}{2}} \cdot e^{i \arctan(2\epsilon)} \cosh(\pi\epsilon)}. \quad (3.3.17)$$

Let the highlighted terms will be “ q ”;

$$q = \sqrt{2/\pi} \left[\frac{1-v_1}{G_1} + \frac{1-v_2}{G_2} \right] / \cosh(\pi\epsilon). \quad (3.3.128)$$

Then,

$$\left[(\Delta u_x)^2 + (\Delta u_y)^2 \right]^{\frac{1}{2}} e^{i\phi} e^{i \arctan(2\epsilon)} = q \left[\frac{r}{(1+4\epsilon^2)} \right]^{\frac{1}{2}} |K| e^{i\psi} e^{i\epsilon \ln(r)}, \quad (3.3.19)$$

$$\left[(\Delta u_x)^2 + (\Delta u_y)^2 \right]^{\frac{1}{2}} e^{i\omega} = q \left[\frac{r}{(1+4\epsilon^2)} \right]^{\frac{1}{2}} |K| e^{i(\psi-\beta)}, \quad (3.3.20)$$

where

$$\omega = \phi + \arctan(2\epsilon) \text{ and } \beta = \epsilon \ln(r). \quad (3.3.13)$$

Reorganize the equation to obtain $|K|$;

$$|K| = \frac{1}{q} \left[\frac{(1+4\epsilon^2) ((\Delta u_x)^2 + (\Delta u_y)^2)}{r} \right]^{\frac{1}{2}} e^{i(\omega-\psi+\beta)}. \quad (3.3.142)$$

Since $|K|$ is a scalar value and corresponds to the magnitude of the complex stress intensity factor, complex term should be unit multiplier, as,

$$e^{i(\omega-\psi+\beta)} = 1, \quad (3.3.23)$$

when,

$$\psi = \omega + \beta. \quad (3.3.24)$$

As a result $|K|$ is in the following form,

$$|K| = \frac{1}{q} \left[\frac{(1+4\epsilon^2) ((\Delta u_x)^2 + (\Delta u_y)^2)}{r} \right]^{\frac{1}{2}}. \quad (3.3.25)$$

Then from Kanninen and Popelar [41], stress intensity factor and energy release rate of a mixed mode crack can be correlated as follows,

$$G = \frac{\frac{1-\nu_1}{G_1} + \frac{1-\nu_2}{G_2}}{4 \cosh^2(\pi\epsilon)} K \bar{K} \quad (3.3.26)$$

where,

$$K \bar{K} = |K|^2 \quad (3.3.27)$$

Therefore, the energy release rate can also be calculated and compared with the energy release rate calculated from Eq. 3.2.4, introduced in the previous section.

$$G = \frac{\frac{1-\nu_1}{G_1} + \frac{1-\nu_2}{G_2}}{4 \cosh^2(\pi\epsilon)} \frac{1}{q^2} \frac{(1+4\epsilon^2) ((\Delta u_x)^2 + (\Delta u_y)^2)}{r} \quad (3.3.28)$$

$$G = \frac{\pi}{8 \left(\frac{1-\nu_1}{G_1} + \frac{1-\nu_2}{G_2} \right)} \frac{(1+4\epsilon^2) ((\Delta u_x)^2 + (\Delta u_y)^2)}{r} \quad (3.3.29)$$

3.4. Benchmark Four Point Bending Test

A benchmark four point bending test is designed to verify both analytic methods introduced in previous sections. The following figure illustrates the test setup, a typical bi-material specimen, the coordinate system and the simultaneous instrumentation.

There are three different instruments used to measure displacement, applied load, crack opening displacements and exact time of crack occurrence as depicted in Fig.3.4.1. First, the applied load and displacement of the upper supports are measured from Shimadzu universal testing machine. Second, crack opening displacements in x and y axis is measured by GOM Digital Image Correlation system and Aramis analysis software. The exact position of the crack tip can also be obtained after the DIC based stress analysis. Third, an in-house-built acoustic emission sensor placed at the interface to detect small cracks occurred during loading. To correlate all these data in time, three lab personnel have to initialize each instrument simultaneously.

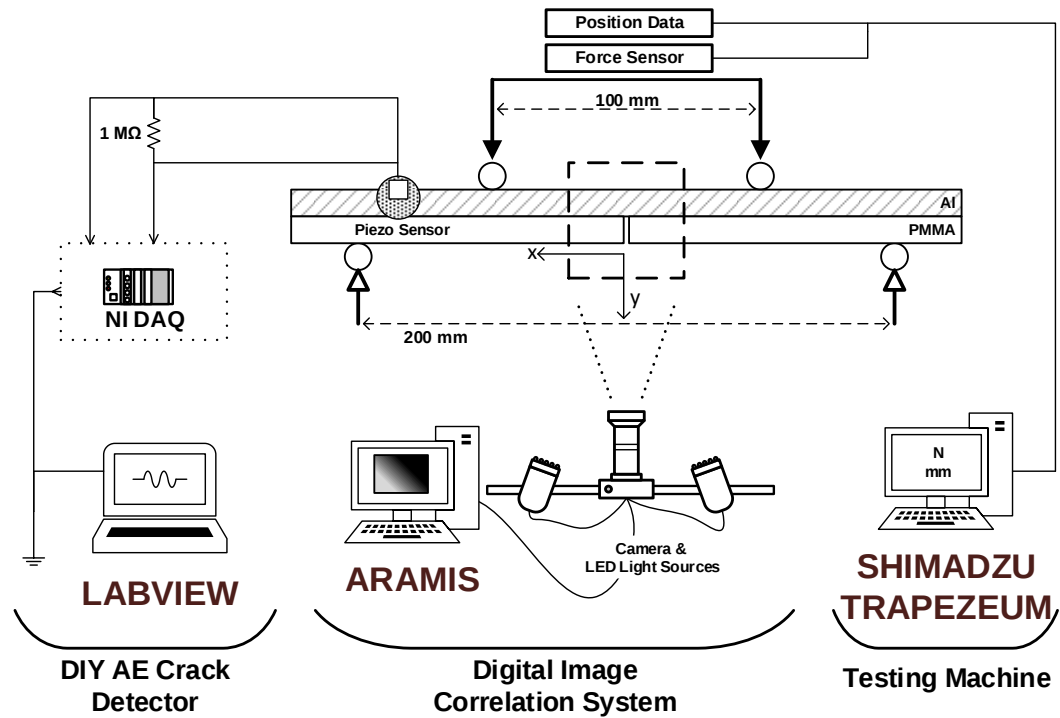


Figure 3.4.1 Benchmark 4-pt bending test showing the instrumentation for crack detection strain and load-displacement measurements operated by LabVIEW, Aramis and Shimadzu Trapezeum respectively.

3.4.1. Digital Image Correlation

Strain gages and extensometers are traditional strain measurement devices that are essential for experimental studies in mechanics. These classical methods are practical in operation but limited to point-wise measurements. Device dimensions, sensor capabilities and environmental conditions may become a significant obstacle in front of the experimental studies. Traditional methods are also insufficient to satisfy the full-field measurement needs of solid mechanics. Development of optical measurement methods, such as photoelasticity and digital image correlation (DIC) has had an impact on the field of experimental mechanics. Optical methods can provide full-field measurement data from surfaces without touching it. Independent from scale and environmental conditions, such as high-temperature and corrosive environment, [8] engineers can experiment on their studies. These operational advantages and growing academic access make full-field optical measurement methods more attractive every day.

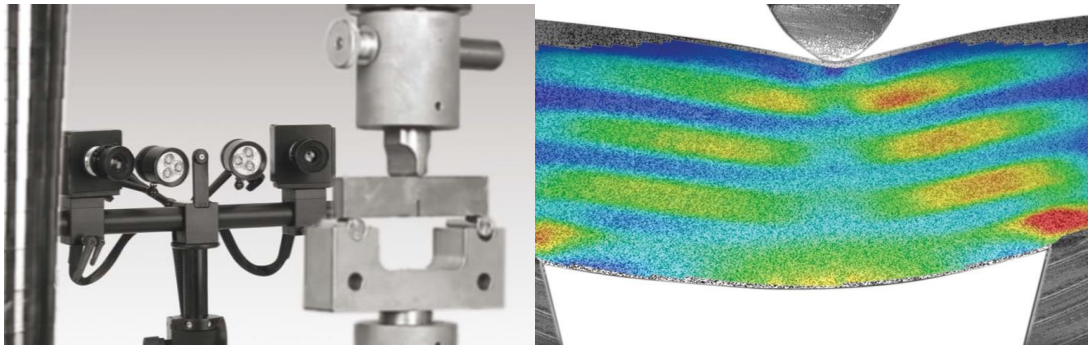


Figure 3.4.2 (a) A typical stereo (3D) type DIC imaging system and (b) a typical DIC analysis result showing stress by coloring the facets.

Digital image correlation method is a full-field optical displacement measurement method that was first applied to experimental mechanics by Sutton and co-workers in 1983 at the University of South Carolina [64]. In Fig. 3.4.2., a typical stereo (3D) type DIC system is illustrated with a typical result image of a composite beam bending showing different stresses in different colors. The method measures displacement fields of stressed surfaces from the image series obtained during the experiment [62, 64]. The ease of use and vast application areas have made the DIC technique one of the most fundamental measurement techniques, especially in the field of nano/micro-mechanical testing. For instance, Hemker et al. [22, 23] conducted state of the art micro mechanical tests on thermal barrier coatings to investigate the failure and to calculate interfacial fracture toughness by using DIC as a solid measurement tool.

DIC method is based on the analysis of images recorded by camera(s) to calculate deformations of small portions of the surface. Common to all digital imaging systems, the image is acquired by an optical sensor that is made of many sensing nodes (pixels) sensitive to light. Pixels generate an output signal corresponding to the color of the sensing light. Color values of pixels indicate black-white intensity for grayscale images or red-green-blue (RGB) intensity for color images of the acquired light. Data (image) received by the optical sensor are stored in a data set containing color values and coordinates of pixels. Image recognition is based on analysis of pixelated series of images applying different numerical methods. In the analysis, color values and coordinates of pixels are used to recognize the geometric patterns. However, there are fundamental methodological problems in image analysis, and DIC is not an exception.

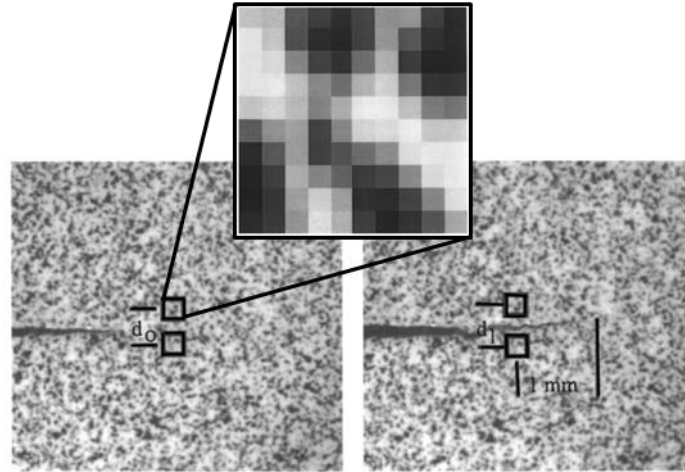


Figure 3.4.3 A typical stochastic pattern of a cracked surface to calculate crack opening displacements on facets at each side of the crack tip.

In DIC method, pixels should be traced through the image set to get a displacement field. Indeed, determination of pixel correspondence between images becomes a critical issue. The grayscale value of a pixel is used to identify it between images. However, a pixel may not have a unique grayscale value. Multiple pixels, have the same grayscale value (uniqueness problem of a pixel), raise “correspondence problem” of a pixel in the image set [63]. Pixels should be traced with further data provided by neighboring pixels. Furthermore, orientation of motion vector of a traced pixel still may not be unique, which raises “aperture problem” [63]. The aperture problem is simply the uncertainty of the motion vector of a repeating pixel pattern where endpoints are out of the aperture. This problem is illustrated in a crane operator’s dilemma in Appendix A.

Measuring strain from pixels of a surface requires some special treatment on it in order not to face the above problems at any stage of analysis. The surface should be uniquely and irregularly textured [63, 70]. Furthermore, the texture should be able to deform with the surface. Random (stochastic) speckle pattern is well suited to these needs. A random speckle pattern can both provide the neighboring pixel data needed to eliminate the uniqueness and correspondence problem, and give irregular texture to surfaces to overcome aperture problem. Two application techniques (laser and spray paint patterning) of the speckle pattern may vary with the size of the investigated area.

Depth of field cannot be perceived from an image set obtained by a single camera. Therefore, mono-camera DIC (2D DIC system) can only detect in-plane deformations. However, the depth of field can be perceived by using simultaneously acquired image sets from dual camera systems. Thus, stereo camera systems (3D DIC system) can also measure the out-of-plane deformation fields. Analysis of 3D DIC experiments requires large processing and storage capacity. Latest advances in computer science have increased the data processing capacity of computers and allowed enhanced analyzes of large data sets in a few minutes. In addition, digital camera technology has dramatically improved since DIC first developed. Today, digital cameras used in daily life have more than enough features (resolution, frame rate) for a DIC analysis. Consequently, DIC has become a standard measurement method in the field of experimental solid mechanics.

Image sets analyzed in DIC can be obtained by different imaging systems at different scales of space and time. High-speed camera systems can be used to obtain measurements of highly dynamic phenomena occurring in nano/microseconds. Scanning electron microscope (SEM) or atomic force microscope (AFM) can be used to detect displacements at nano/micrometer scale. Even satellite images can be used in the field of geophysics to observe deformations in land forms.

In this study, DIC was used to measure crack opening displacements and to determine the position of crack tip. Deformed surface is traced through the image series starting from the basis image (which is preliminarily defined). The basis image is taken as undeformed state of the experiment, and the deformation field of the stressed surface can be achieved from an analysis software.

3.5. Results

3.5.1. Bi-material Specimen

AL-PMMA bi-material couple was used as a specimen for the 4-pt bending test. The specimen geometry and interface are shown in Fig.3.5.1. First, both PMMA and aluminum bar is machined to 8 X 10 X 250 mm rectangular prism as depth, thickness and length respectively and glued surfaces are roughened by 80 grid metal sandpaper. Aluminum is machined from 5083 series rolled aluminum block which has 72 GPa of Young's Modulus and 0.33 of Poisson's ratio. The critical load applied is set to 800 N

not to cause any permanent deformation during tests. PMMA is a standard transparent PMMA sheet which has Young's Modulus between 1.8 GPa and 3.1 GPa. PMMA bar was cut to two pieces equally and glued 1mm apart from each other on one piece aluminum bar. To create a pre-crack, the tip of PMMA strips are masked by a paper tape and unmasked after the glue is applied. Therefore, there appear an un-glued portion at middle of the material couple act as a pre-crack under loading.

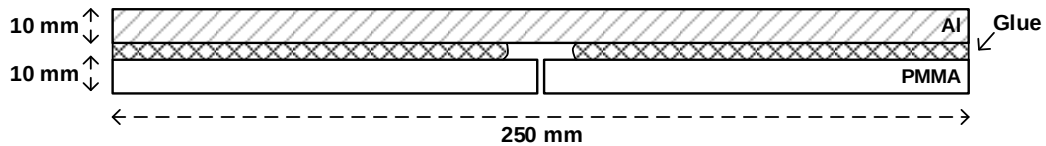


Figure 3.5.1 Typical specimen geometry and interface.

Bonding two dissimilar material is a difficult process. Tested adhesives are listed in Table 3.5.10. To prepare the specimen, first sections of aluminum and PMMA bars are masked by a paper tape except the surface that adhesive applied. Then, a portion of that surface is also masked to create a pre-crack. Adhesives were applied on surfaces according to the instructions of the adhesives. Then, the material couple clamped and let the adhesive cure. After each adhesive tested, the adhesive was cleaned by acetone and the surfaces are grinded to remove residuals of the adhesive.

3.5.2. Adhesive Materials

Five different adhesives were tested in the process of verification. It is important to notify that at each adhesive has different adhesion performance and cracking mechanism. First, bisphenol-A based Pattex universal mix epoxy is used to bond Al-PMMA couple. The Pattex universal mix epoxy used specimen was broke in hand by a small loading. After the crack propagated, it was inspected that the applied epoxy only stacked to PMMA surface. Second, 1 Ton Devcon Epoxy is applied as an interface but similar to Pattex universal mix epoxy, the crack propagated very easily. Third, Bison Metal epoxy which is developed special for repair of metal parts was applied on both surfaces according to the instructions. Metal epoxy adhered both surfaces and test was done successfully for 10 mm initial crack length. However, it is seen that the crack gradually propagated. Fourth, Bison universal contact adhesive is applied to both surfaces. However, the adhesive was still very elastic even after the ultimate cure time. As a result, the adhesive layer between Al and PMMA stretched

during the test and the crack did not propagated. Finally, Loctite 469 instant metal adhesive is applied on both surfaces. The adhesion was successful but the crack dynamically propagated at an instant of time.

Table 3.5.1 Tested adhesives and observations.

Adhesive	Observation
Pattex Universal Mix Epoxy	Unsuccessful Adhesion
1 Ton Devcon Epoxy	Unsuccessful Adhesion
Bison Universal Contact Adhesive	Successful Adhesion and No Cracking
Bison Metal Epoxy	Successful Adhesion and Gradual Cracking
Loctite 469 Instant Metal Adhesive	Successful Adhesion and Sudden Cracking

3.5.3. Testing Procedure

There are three different instruments used in the test setup. These are universal testing machine, digital image correlation system and the acoustic emission testing system. Aside, there are three successful tests for three different adhesive interfaces. For each test, 5 mm pre-crack is used except the test of Metal epoxy.

All three tests were conducted on the same test setup under displacement controlled loading with a 0.5 mm/min loading rate when a Photron 1 MP camera is recording a 25 mm X 25 mm frame. The acoustic emission system is also recording the sound by 30000 Hz. Each instrument was initiated at the same time by three different operators.

3.5.4. Load-Stroke Results

The applied load and the stroke of the testing head is measured from Shimadzu universal testing machine. The load vs stroke curves of three different adhesive interfaces are shown in Fig.3.5.2 (a). The slope of the load-stroke curve in Fig. 3.5.2 (a) represents the overall elastic modulus of the bi-material couple with the intended adhesive interface. The change of load at each time step are also shown in Fig. 3.5.2 (b), (c) and (d) for metal epoxy, contact adhesive and Loctite 496 adhesive, respectively. The change in the slope of the curve is calculated by taking derivatives at each time step. Therefore, plots in Fig. 3.5.2 (b), (c) and (d) are both representing the dynamic change in the elastic modulus of the system during testing and the exact time of crack propagations as where the slope of the load-stroke curve is negative.

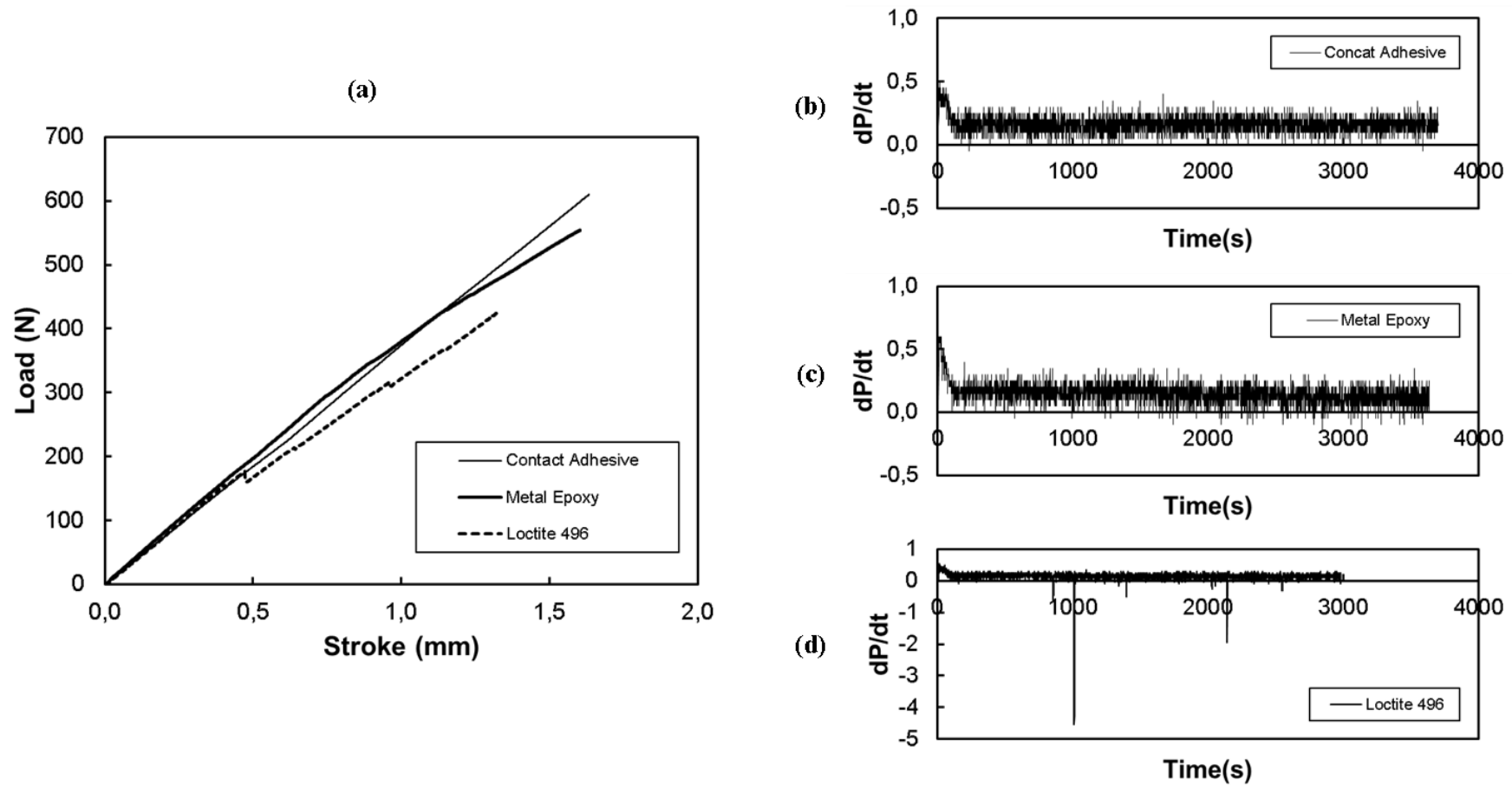


Figure 3.5.2 Load vs stroke plot of three different interfaces under 4-pt bending showing the derivative of load curve of (b) metal epoxy (c) contact adhesive and (d) Loctite 496 interfaces where negative derivatives are corresponding to the load drops.

In Fig. 3.5.2 (a), it is clear that each system with different adhesives had similar overall elastic modulus at the first steps of the test where the pre-crack stays stable. However, each system has different characteristic behavior through the test.

In Fig.3.5.2(c), the first load drop in metal epoxy interface is detected in 28.3 second at 114.85 N loading and 0.284 mm stroke corresponding to 0.2852 Nm moment around the crack tip. After the first load drop, it is observed that the crack length stays stable until the second load drop occurred in 49.5 second at 185.2 N loading and 0,46 mm stroke corresponding to 0.4626 Nm moment. The slope of the load-stroke curve does not change drastically until the load reaches around 300 N. The crack length increases steadily that the change in overall elastic modulus of the system changes linearly.

Second adhesive interface is the contact adhesive. The load-stroke results of the contact adhesive showed that there is not any crack propagation through the test. The derivative of the load-stroke curve stays positive which means the contact adhesive used bi-material system keeps the load carrying capacity through the entire test.

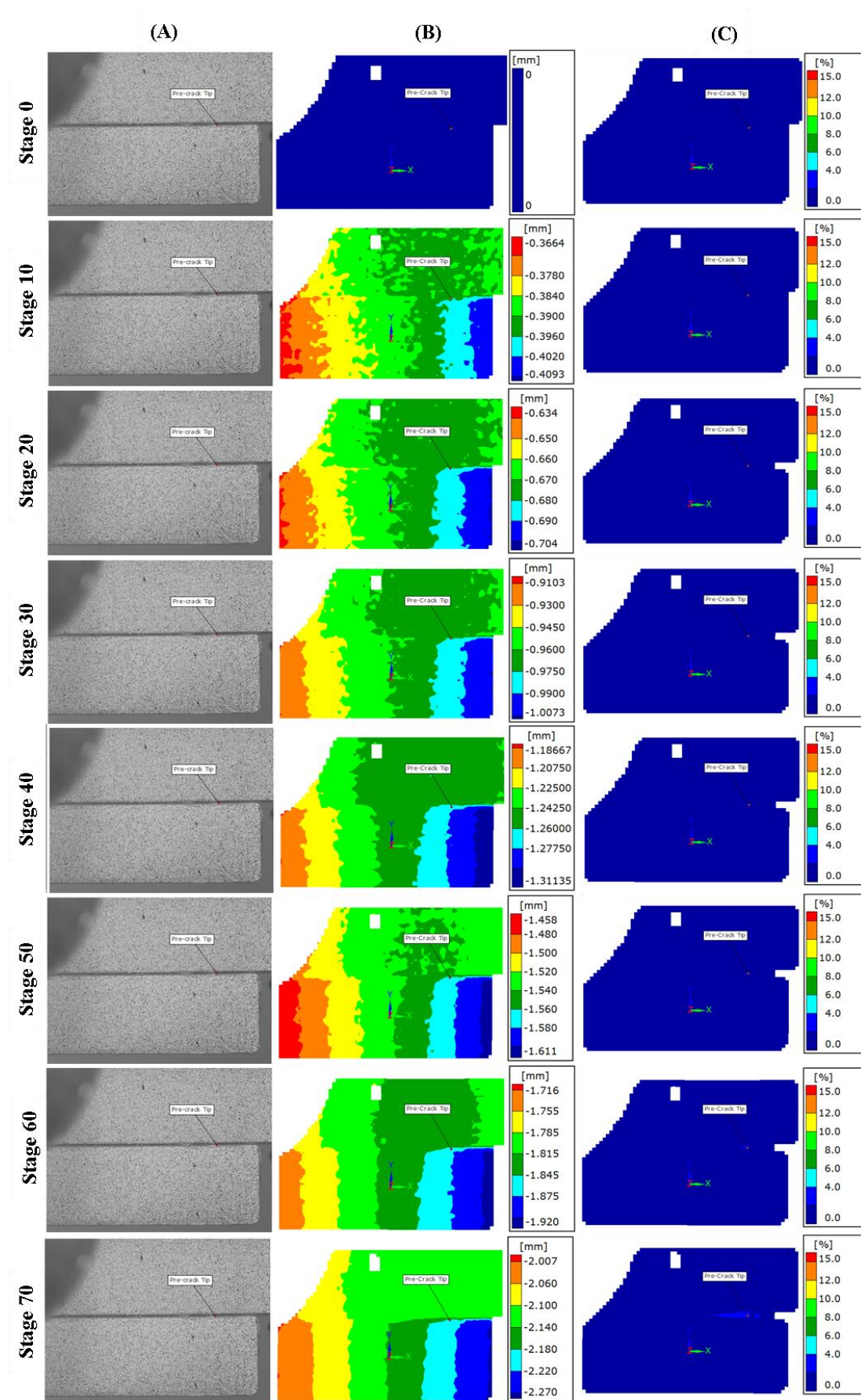
Third tested adhesive interface is Loctite 496 instant glue. The super glue adhesive interface cracked dynamically with dramatic load drops occurred in the load-stroke curves. The failure of Loctite 496 interface is very similar to the interface failures of composite laminates [71]. The first load drop in 7.65 second at 37.15 N loading and 0.92 mm stroke corresponding to 0.0929 Nm moment around the crack tip. The first load drop is very limited since the slope of the load-stroke curve did not change. Then the ultimate failure of the interface occurred in three dynamic crack propagations. First failure is in 42.6 second at 154 N loading and 0.406 mm stroke corresponding to 0.3858 Nm moment around the crack tip. Second failure occurred in 50.3 second at 176.6 N loading and 0.4726 mm stroke corresponding to 0.4415 Nm moment around the crack tip. Third failure occurred in 69.65 second at 213 N loading and 0.63 mm stroke corresponding to 0.53 Nm moment around the crack tip. The cracks propagated through the interface and stopped at the nearest loading point.

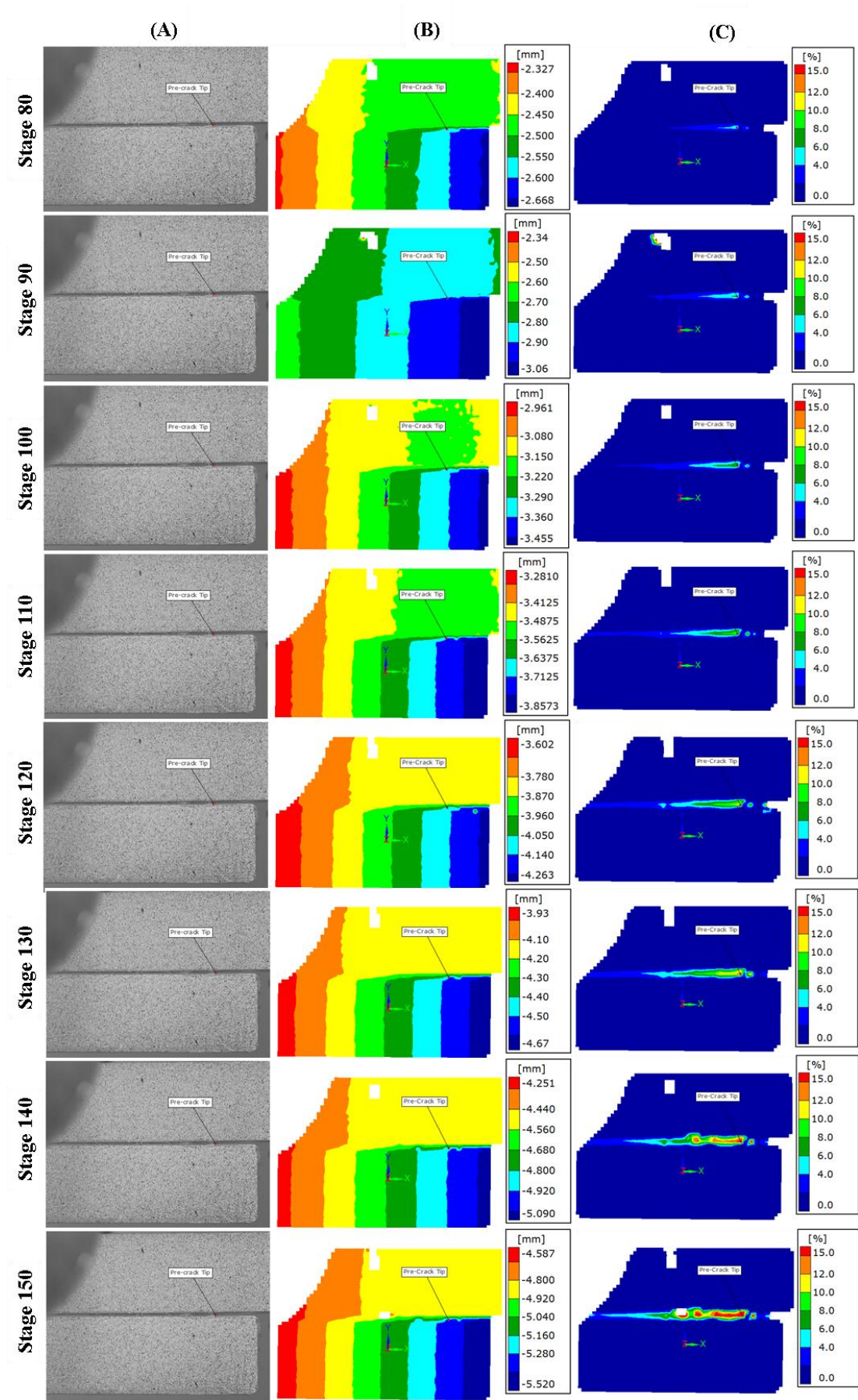
3.5.5. 2D DIC Results

The location of the crack tip and mixed-mode crack tip opening displacements are measured from 2D DIC results.

3.5.5.1. Bison Universal Contact Adhesive

Bison universal contact adhesive is a one component adhesive. The contact adhesive was chosen to increase the interface strength and increase the initial crack load. According to the technical data sheet of contact adhesive, this material can stick both on plastics and metals. The contact adhesive was applied on both surfaces according to the instructions by giving 10 mm pre-crack symmetrically at both sides of the Al-PMMA interface. Loading rate set to the minimum of the tensile testing machine and camera acquire image at each second. The test was followed up to the critical load not to deform aluminum bar permanently. Totally, 185 images were acquired from the test. The load-stroke data was analyzed in the previous section and it was observed that there is no load drop at any part of the loading. From this evidence, it was assumed that the crack stood stable. In Fig. 3.5.3, Von Mises strain and y-axis displacement contours achieved from DIC analysis of the test images were presented at columns C and B, respectively. The designated stage point is the estimated location of the tip of the 10 mm pre-crack. At the column B of Fig. 3.5.3, the y-axis displacements were presented. As the load increases first the bar started to deform under loading and in the 20 seconds of the testing, crack location can be detected by the y-axis displacements. The designated estimated crack tip location is very close to the estimated crack tip from y-axis displacement results. After the first 20 seconds of the test, the contact adhesive interface starts to stretches and opens, but no crack propagation. This phenomena is also supported from the Von Mises strain results in Fig. 3.5.3 column C. Strain concentration increases at all along the interface as the loading increases. Another important remark is that the contact between two bars and the interface never lost in all DIC results. Occasionally, if there is a crack and opening, some of the facets would deform over its maximum allowable deformation range. Contact adhesive test does not properly suit the preliminary estimations of the energy release rate calculations, since the interface plastically deforms and overrules the fully elastic estimation. Therefore, neither Euler-Bernoulli beam bending theory based analytic solution nor the bi-material solution can be applied on the case of contact adhesive interface.





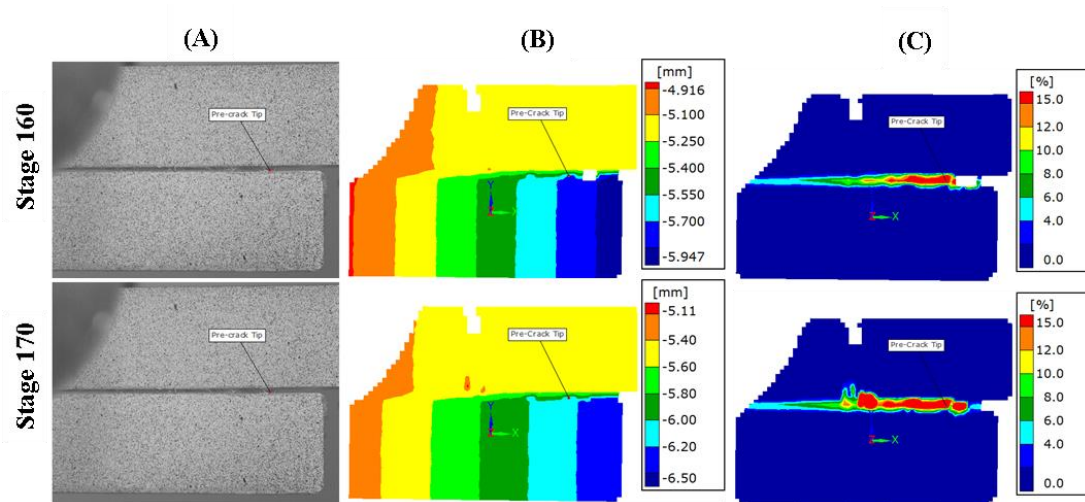


Figure 3.5.3 Contact adhesive test results showing (A) image as a stage data at every 10 seconds , (B) measured displacement in y-axis and (C) calculated Von Mises strain contours generated by ARAMIS DIC analysis software where the stage point designates the estimated location of the pre-crack tip at 10 mm inside the middle of the Al-PMMA bi-material couple.

3.5.5.2. Bison Metal Epoxy

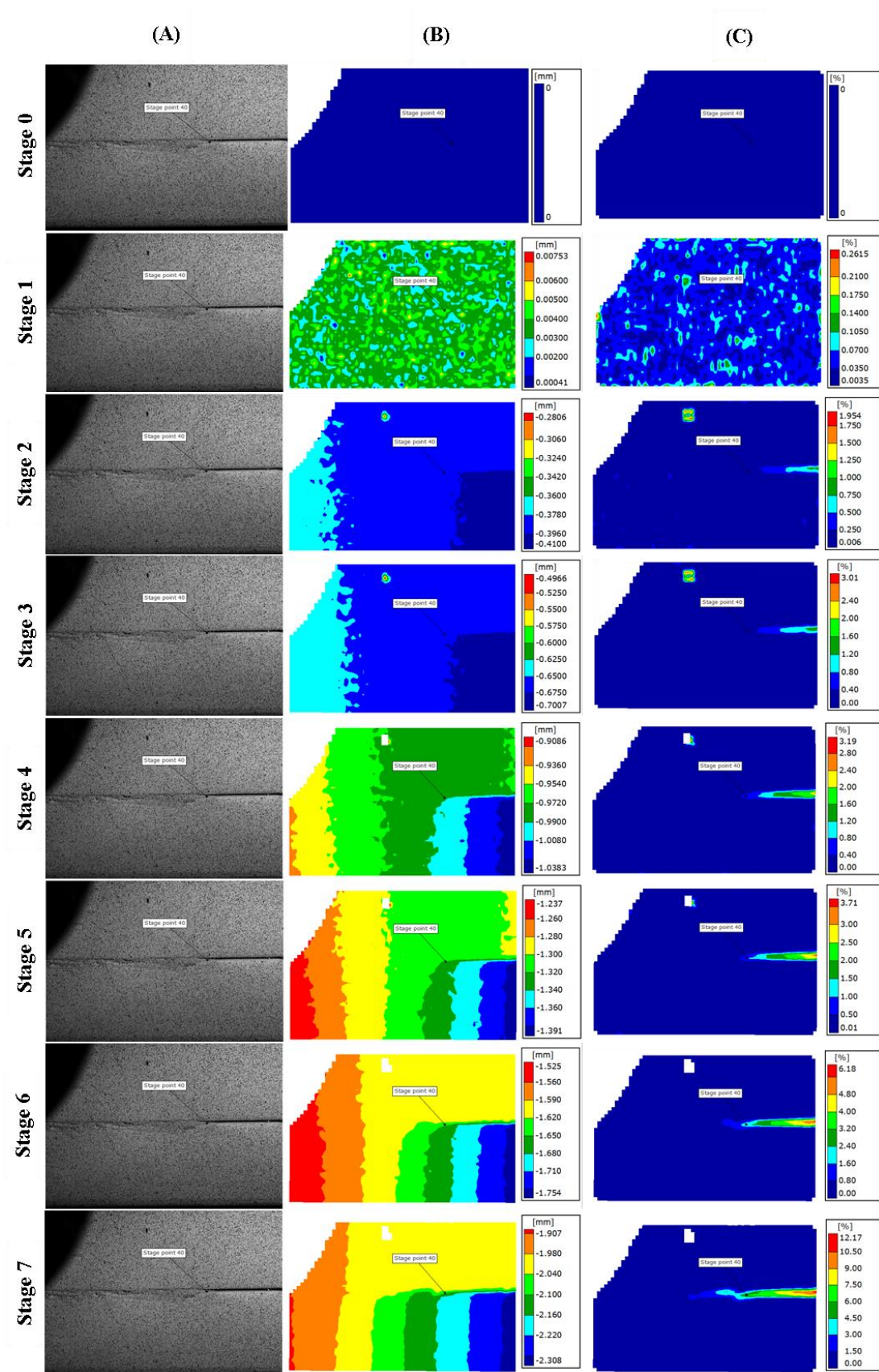
Bison metal epoxy is developed for repairing purposes of metal parts. The Bison metal epoxy chemically contains adhesive and filler properties at the cured state. This material was chosen intentionally in the first place to achieve a good adhesion between aluminum and PMMA bars. According to the technical data sheet, the Bison metal epoxy is capable of bonding metals to synthetics and resistant to extreme conditions like loading, vibration, so on. Simple subjective observations of the adhesive capabilities of the tested adhesives which are presented in Table 3.5.1 also confirms these properties.

The metal epoxy was applied according to the instructions introduced in the technical sheet of the material by giving 10 mm pre-crack at both sides of the Al-PMMA interface, symmetrically. The loading rate set to the minimum of its range and at each 0.1 mm stroke one image of the specimen was shot by the camera setup for DIC analysis. 17 images including the initial stage of the experiment were shot which starts with “Stage 0” denoting the initial stage. 17 images are corresponding to 1.6 mm total stroke applied on the specimen. The test was stopped after the crack propagation reached one of the loading points where the crack cannot grow anymore.

In the previous section, the load-stroke data is analyzed and some hypothesis is made to decide when the crack propagated. According to the load drop history, between which stages the crack grew is known. However, to confirm this information and ensure the location of the crack tip for further fracture mechanics analysis, displacement in the y-axis (vertical axis) and the Von Mises strains are needed. In Fig. 3.5.4, Bison metal epoxy test results showing the image as a stage data, measured displacement in the y-axis and the calculated Von Mises strain contours generated by ARAMIS DIC analysis software are presented for 13 stages including the initial stage. The stage images are taken at every 0.1 mm stroke applied, as discussed before. Moreover, a stage point is used to point the estimated location of the pre-crack tip which is at 10 mm inside the middle of the Al-PMMA bi-material couple. The stage point can be shown as long as the facet that they attached is shown. Therefore, for further stages, the stage point is lost.

The y-axis displacement was used to locate the crack tip and crack propagation through the images. A 10 mm part of the bar moves in the three dimension as a bar supporting from one end under bending, since the cracked surfaces are traction free. If the interface bonding is good enough, the stress can transmit through the interface and the bonded parts of the composite beam can act like a whole body. Therefore, displacement in the y-axis can be used to locate the crack tip. Moreover, it is well-known that if a stress concentration occurred around the crack tip, measured strains would concentrate around the crack tip. For this purpose, Von Mises strains can also be used to locate the crack tip like displacement in the y-axis.

According to the load-displacement curve, there are 2 load drops which corresponds to the time interval between stages 2-3 and 4-5 in Fig. 3.5.4. However, according to y-axis displacement and Von Mises strain, crack tip location changes as it grows. Therefore to measure a better energy release rate, both DIC results will be analyzed.



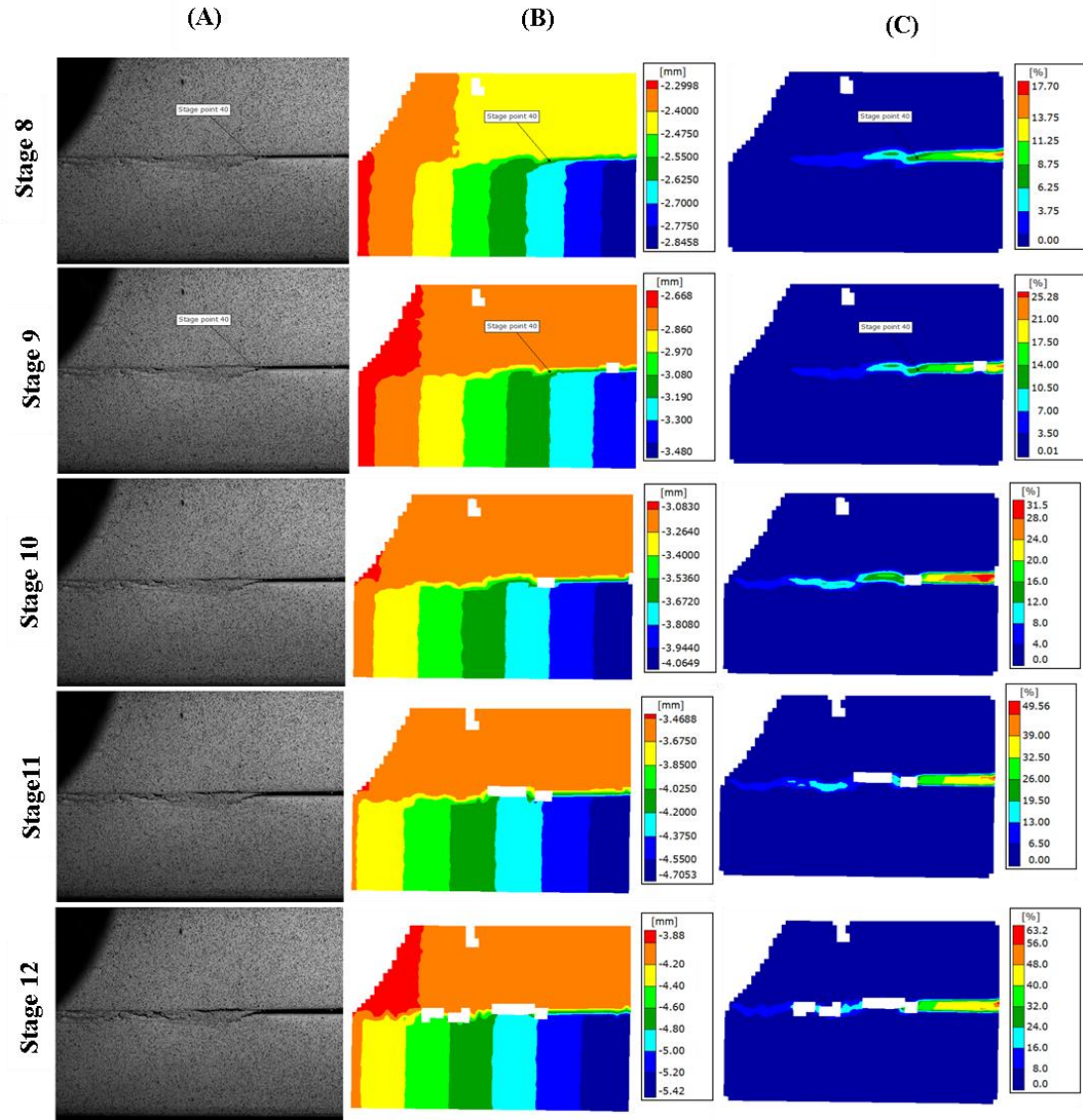


Figure 3.5.4 Bison metal epoxy test results showing (A) image as a stage data at every 0.1 mm stroke, (B) measured displacement in y-axis and (C) calculated Von Mises strain contours generated by ARAMIS DIC analysis software where stage point 40 designates the estimated location of the pre-crack tip at 10 mm inside the middle of the Al-PMMA bi-material couple.

In Fig. 3.5.5, the measured crack lengths are presented with respect to the load applied. The crack lengths were measured by using Von Mises strain contours from DIC. As load increases the crack length increases gradually. These load and crack length data will be used for analytical solutions. As it is seen from stage 2 crack length, the initial crack length is 8 mm.

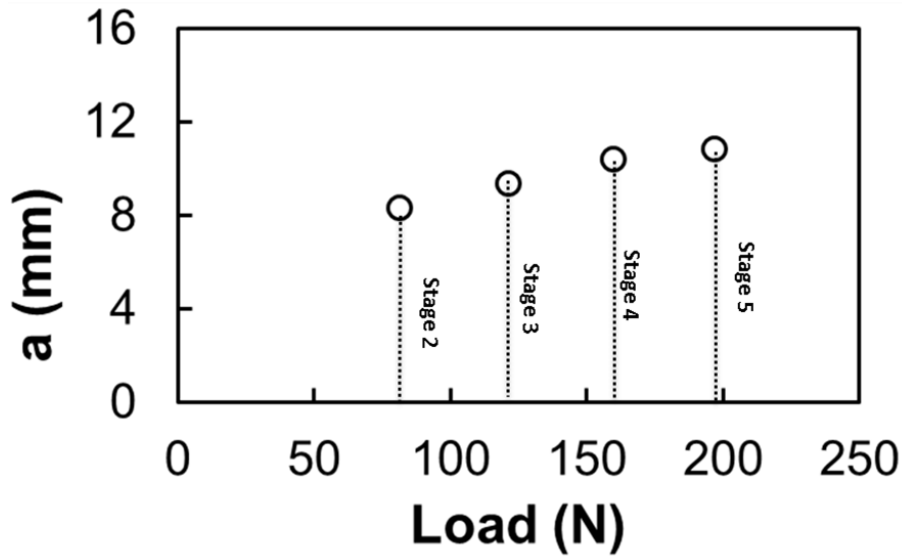


Figure 3.5.5 Crack length of metal epoxy interface through the test measured by using Von Mises strain contours.

In Fig. 3.5.6, energy release rate of the metal epoxy interface was calculated by two different analytic solutions which are Euler-Bernoulli beam bending theory and the bi-material solution of the specified geometry. In Euler-Bernoulli method, energy release rate can be calculated by using the load-displacement curve and the geometric parameters of the specimen. In bi-material solution of metal epoxy interface, energy release rate can be calculated at different locations behind the crack tip by using load, crack opening displacements and some specimen and material constants. Therefore, exactly locating the crack tip by DIC becomes critical for the bi-material based solution. Two different DIC results can be used to locate the crack tip: displacement-y data and Von Mises strain data. Averaged energy release rates presented in Fig. 3.5.6, are the data measured by using Von Mises strain to locate the crack tip. First, Von Mises strains used to locate the crack tip and bi-material based energy release rate calculations were done afterwards. Each data point contains 4 different energy release rates at different distances from the crack tip. Second, the same procedure was

followed expect y-axis displacements were used to locate the crack tip. As a result Von Mises strain data give more consistent results. There are slight difference between Von Mises strain based bi-material solution and Euler-Bernoulli based analytic solution. Both energy release rate results exhibit an increasing trend as the load increases.

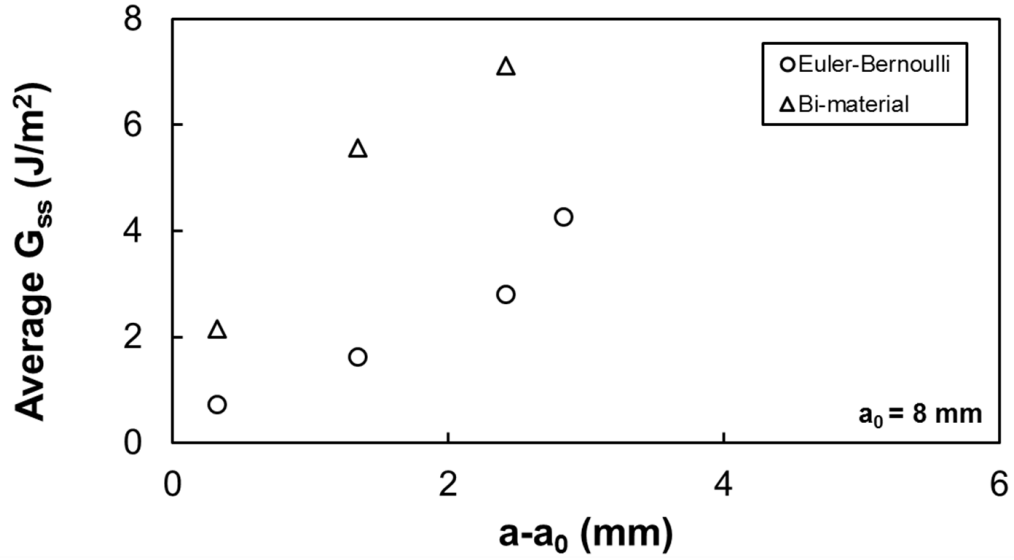


Figure 3.5.6 Energy release rate of the metal epoxy interface averaged by calculating the G_{ss} at different “r” and different crack lengths corresponding to different loads.

As introduced in Fig. 3.5.4, energy release rates were calculated at four different loads which also corresponds to 4 different crack lengths. In Fig. 3.5.6, the energy release rates calculated by bi-material method using Von Mises strain to locate the crack tip were presented. The energy release rate of the interface increasing as the crack length increases. At different locations energy release rate calculations varies but they vary in a specific band. As commented on load displacement curve, metal epoxy interface cracks very gradually. The same cracking mechanism can be observed in Fig. 3.5.6. The energy release rate increases as crack length increases.

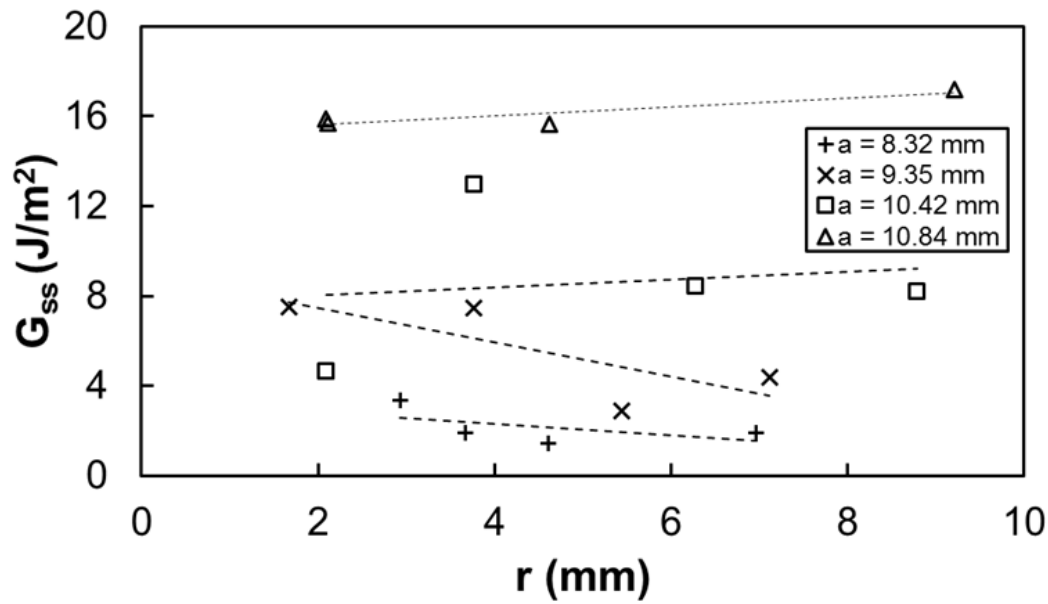
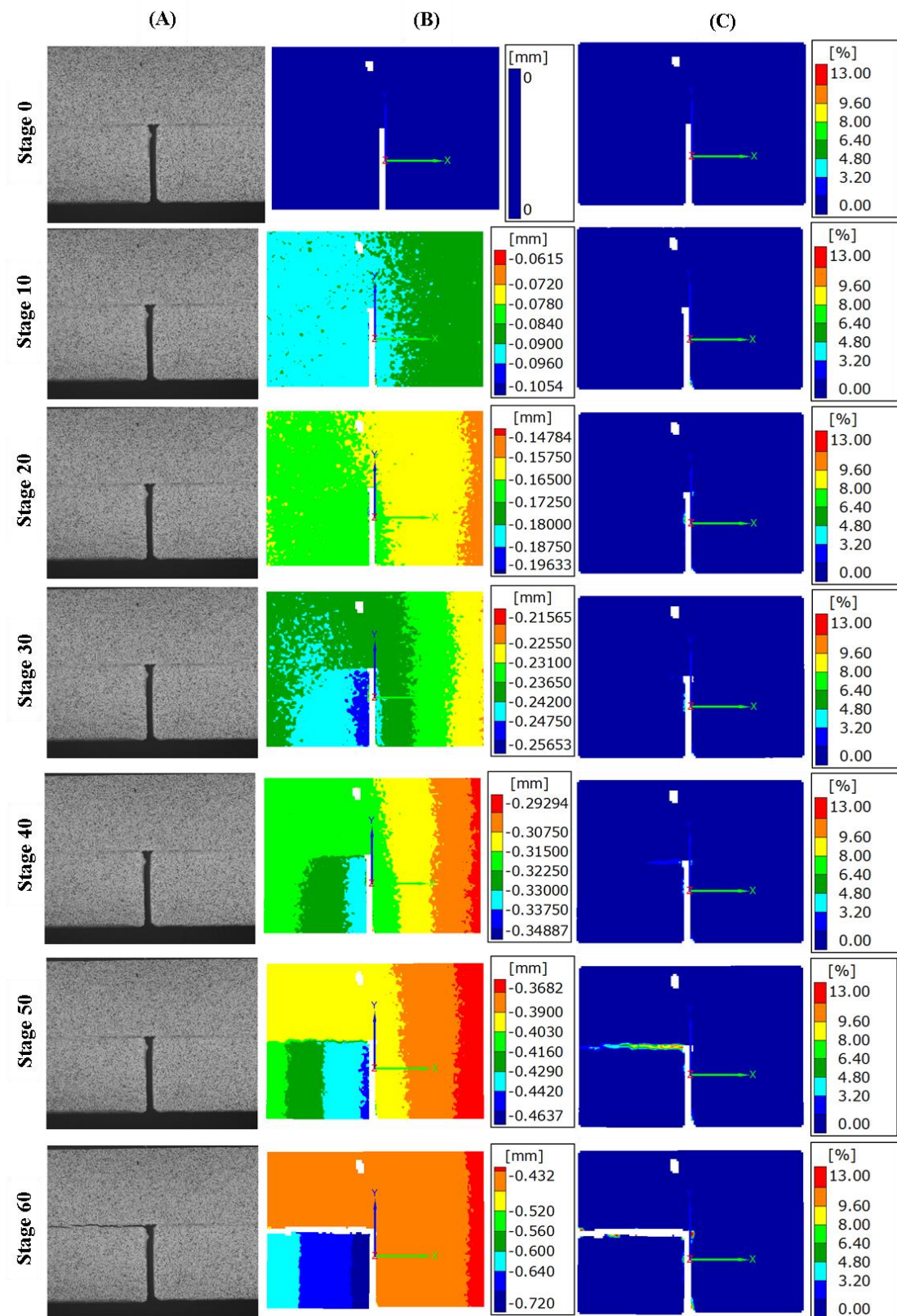


Figure 3.5.7 Bi-material method based energy release rates (crack located by Von Mises strain) at different distances from the crack tip and at different crack length which corresponds to different loads.

3.5.5.3. Loctite 496

Loctite 496 is an instant glue which is more brittle than the previous two adhesives. Loctite 496 was applied on both surfaces of the bars by giving a 5 mm initial crack at one side of the symmetric specimen. Loading rate set at the minimum of the machine while camera acquiring one image per second. Aside, there were 3 dynamic crack growths recorded from the load-stroke data. In Fig. 3.5.8, the images showing that the crack growth is presented in column A, the y-axis displacement results are presented in column B and the Von Mises strain contours presented in column C.

According to the y-axis displacement and Von Mises strain results obtained from DIC, the cracking mechanism of Loctite 496 interface was occurred faster than the previous tests. The crack propagation started at the stage 7 which corresponds to 7th second of testing and grow a little bit. Then, after the stage 35, the crack started propagated rapidly and followed by the third and fourth cracking at 42th and 50th seconds. Even the cracking seem to be very dynamic, between images it can be seen that the crack increases steadily at a point and then propagated dramatically.



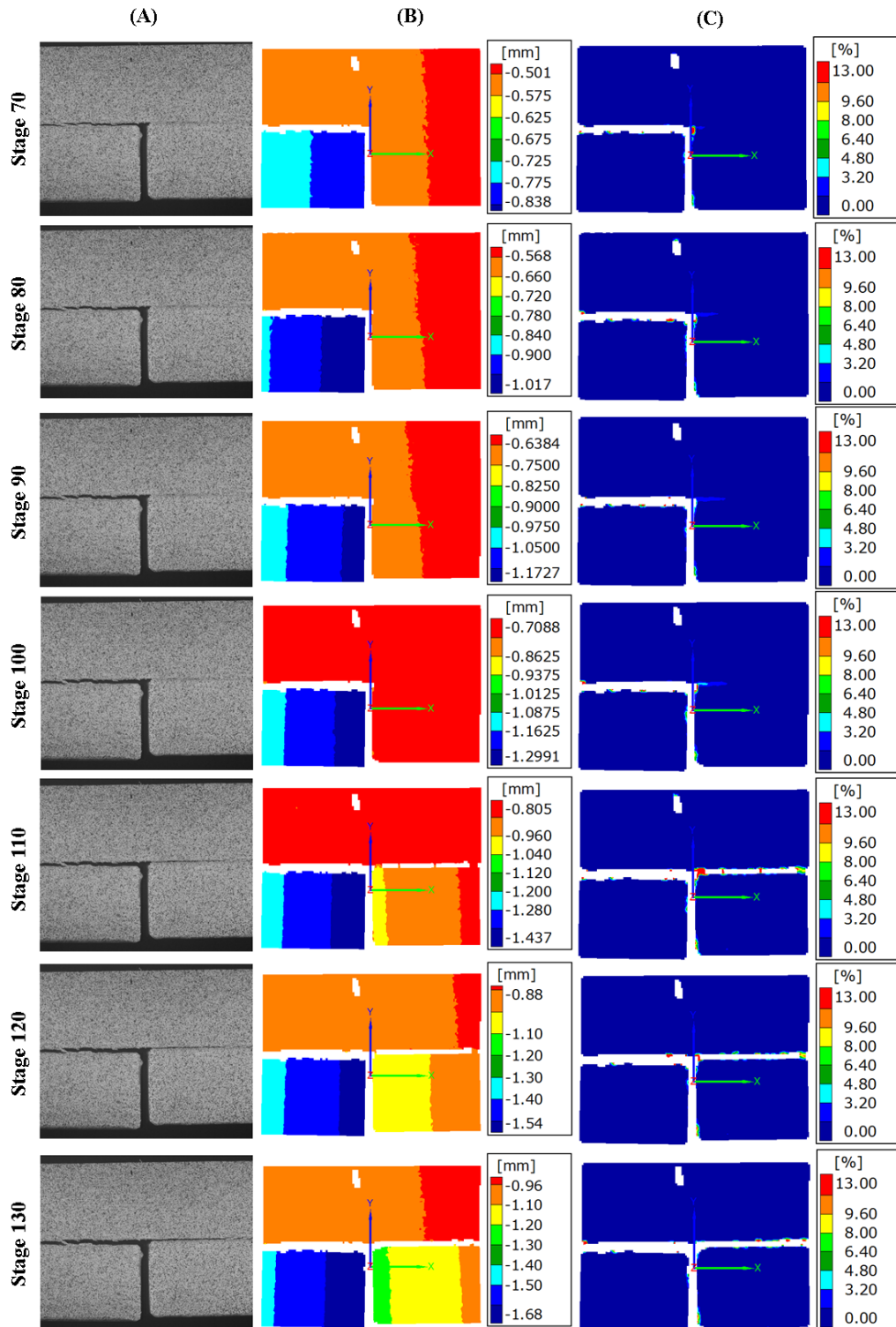


Figure 3.5.8 Loctite 496 test results showing (A) image as a stage data at every 10 seconds , (B) measured displacement in y-axis and (C) calculated Von Mises strain contours generated by ARAMIS DIC analysis software where the stage point designates the estimated location of the pre-crack tip at 10 mm inside the middle of the Al-PMMA bi-material couple.

In Fig. 3.5.9, the measured crack lengths are presented with respect to the load applied. The crack lengths were measured by using Von Mises strain contours from DIC. As load increases the crack propagates instantaneously. These load and crack length data will be used for analytical solutions.

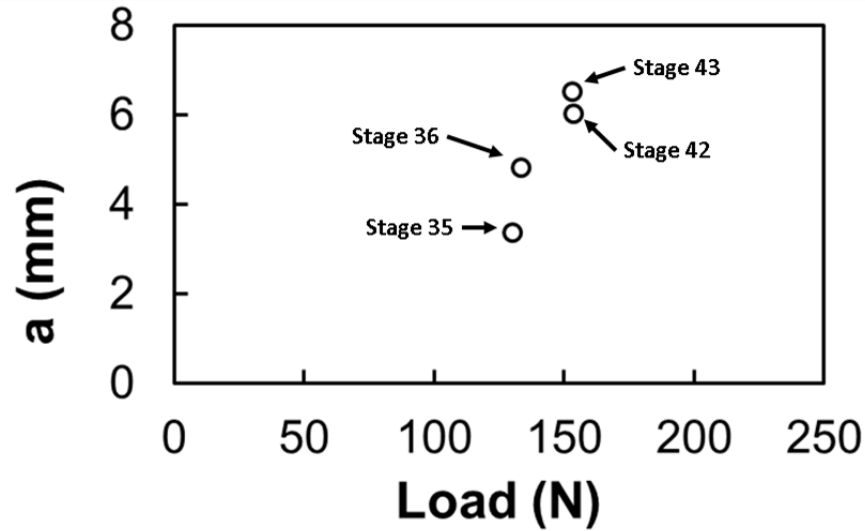


Figure 3.5.9 Crack length of Loctite 496 interface through the test measured by using Von Mises strain contours.

In Fig. 3.5.10, the energy release rate of the Loctite 496 interface which was calculated by two different methods are presented. Each data point contains 5 to 7 different energy release rates calculated at different distances from the crack tip. For Loctite 496 tests, the interface is so thin that only some meaningful measurements can be taken after 130 N loading and corresponding displacement from DIC. Therefore, for Loctite 496 test, energy release rate can only be calculated for three loads which are also the critical loads where crack grew. In the calculation procedure, the crack tip was located at the tip of the Von Mises strain contours at the interface. Each data set was fit linearly to show how two methods fit each other. For Euler-Bernoulli beam bending theory based analytic solution, the energy release rate slightly increases as the load increases. For the bi-material solution, at each load the averaged energy release rates came very different but they also stay in a band restricted between 1.7 J/m^2 and 3.8 J/m^2 . As a result, both the crack tip locating technique and the methods used to calculate energy release rate were verified for a brittle interface as Loctite 496.

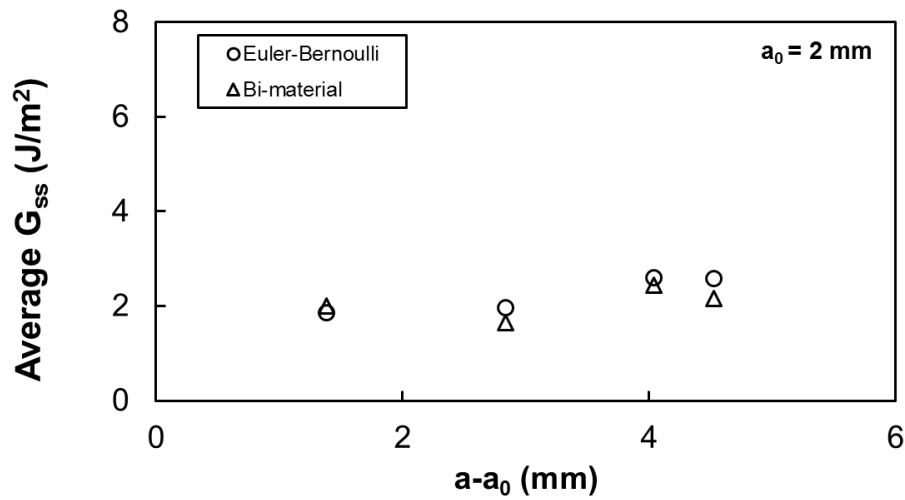


Figure 3.5.10 Energy release rate calculated by two different methods of the Loctite 496 interface averaged by calculating the G_{ss} at different “r” and different crack lengths corresponding to different loads.

In Fig. 3.5.11, the energy release rate of the Loctite 496 calculated by the bi-material method at different locations are presented. Each shape in the figure designates different crack lengths. The crack length increases and the energy release rate stays in the band between 3.2 J/m^2 and 1.5 J/m^2 except the ones very close to the crack tip. If the distance between the crack tip and the crack opening displacements were measured is too close, the method stays in the plastic zone.

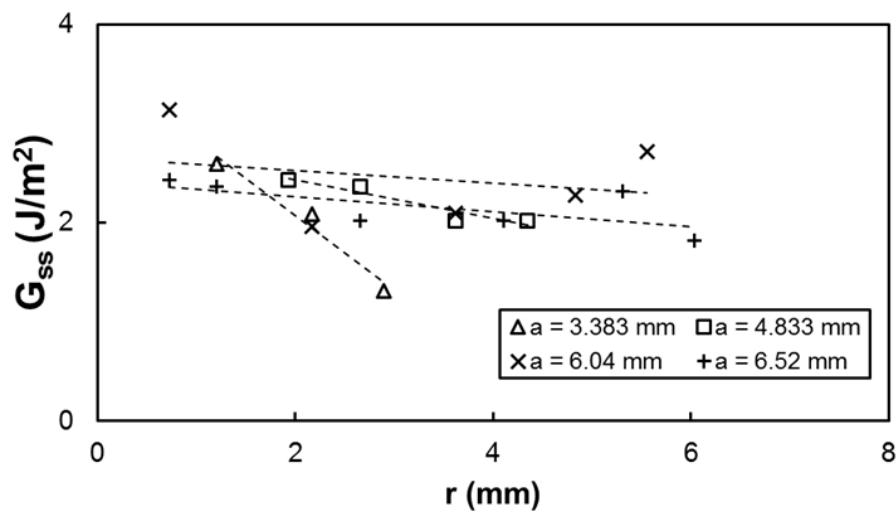


Figure 3.5.11 Bi-material method based energy release rates of Loctite 496 interface (crack located by Von Mises strain) at different distances from the crack tip and at different crack length which corresponds to different loads.

CHAPTER 4

DETERMINATION OF FRACTURE PROPERTY OF SPECIMENS WITH THERMAL BARRIER COATING

In this chapter, TBC specimens with three different thicknesses were tested by a micro-bending test which was developed for this purpose specifically. The main purpose of the micro-bending test is to measure at least one of the fracture properties of the TC/BC interface and investigate the complete mechanism of the TBC specimens under pure bending. First, the micro-bending test methodology was introduced with the equipment used in it. There are two different results was expected. First one is the fracture mechanism and order of occurrences during the cracking. Second one is the energy release rate of a TC/BC interface which were calculated by the validated method in Chapter 3.

4.1. Methodology

4.1.1. Specimens

There are three TBC specimens with different coating thicknesses tested by micro-bending test. Specimens were cut by an abrasive cutting disk and grinded to diminish the cutting flaws at the cut edges. Then, each specimen cross-section was painted by a special powder paint adequate to DIC system. In Table 4.1.1, specimen geometries and the overall coating thicknesses are presented. Specimens were named after their coating thicknesses which are *thick*, *standard* and *thin* corresponding to maximum, standard and minimum coating thicknesses between specimens.

Table 4.1.1 Geometry and coating thicknesses of the specimen set used in micro-bending test.

Specimen	Width (mm)	Top Coat (mm)	Bond Coat (mm)	Substrate (mm)
Thick	6.41 – 6.66	0,60	0,16	1,58
Standard	6.4 – 7.2	0,38	0,10	1,6
Thin	9.1	0,26	0,05	1,59

4.1.2. Experimental Setup

A micro-bending test was used to measure one of their fracture properties of a TBC specimen. Micro-bending test was designed as the scaled down version of the benchmark four point bending test introduced in Chapter 3.

In Fig. 4.1.1(a), an image of the whole experimental setup was presented. There are two major components of the micro-bending test. First, the microscope imaging system which was used as the image acquisition system. The imaging system is Huvitz HDS- 2520Z which is a travelling microscope that also has an integrated light source. The microscope was positioned accurately with respect to the testing frame and the specimen. Then, the imaging system taped on the table. A close up image of the microscope and the specimen is also presented in Fig. 4.1.1(b). The microscope acquires in 25X magnification where the resolution is 0.01 mm per pixel. The 0.01 mm/pixel resolution corresponds to 0.13 mm facet size for a 13X13 pixels of facets. Second, a universal testing machine was used to operate the micro-bending test. The experimental fixture was presented in Fig. 4.1.1(b). The upper supports are galvanized and low friction standard lower supports of the three point bending fixture. They were set to 28 mm precisely and mounted on the upper side of the machine. The lower support is a wire erosion cut steel which was also precision grinded to make the surfaces perpendicular to each other. The surfaces of the lower support which touch the specimen was also coated by one-side-adhesive Teflon tape to minimize the friction interference on testing. Peaks of the lower support position 14 mm away from each other. In Fig. 4.1.1(c) the free body diagram of the loading conditions of a typical TBC specimen was shown.

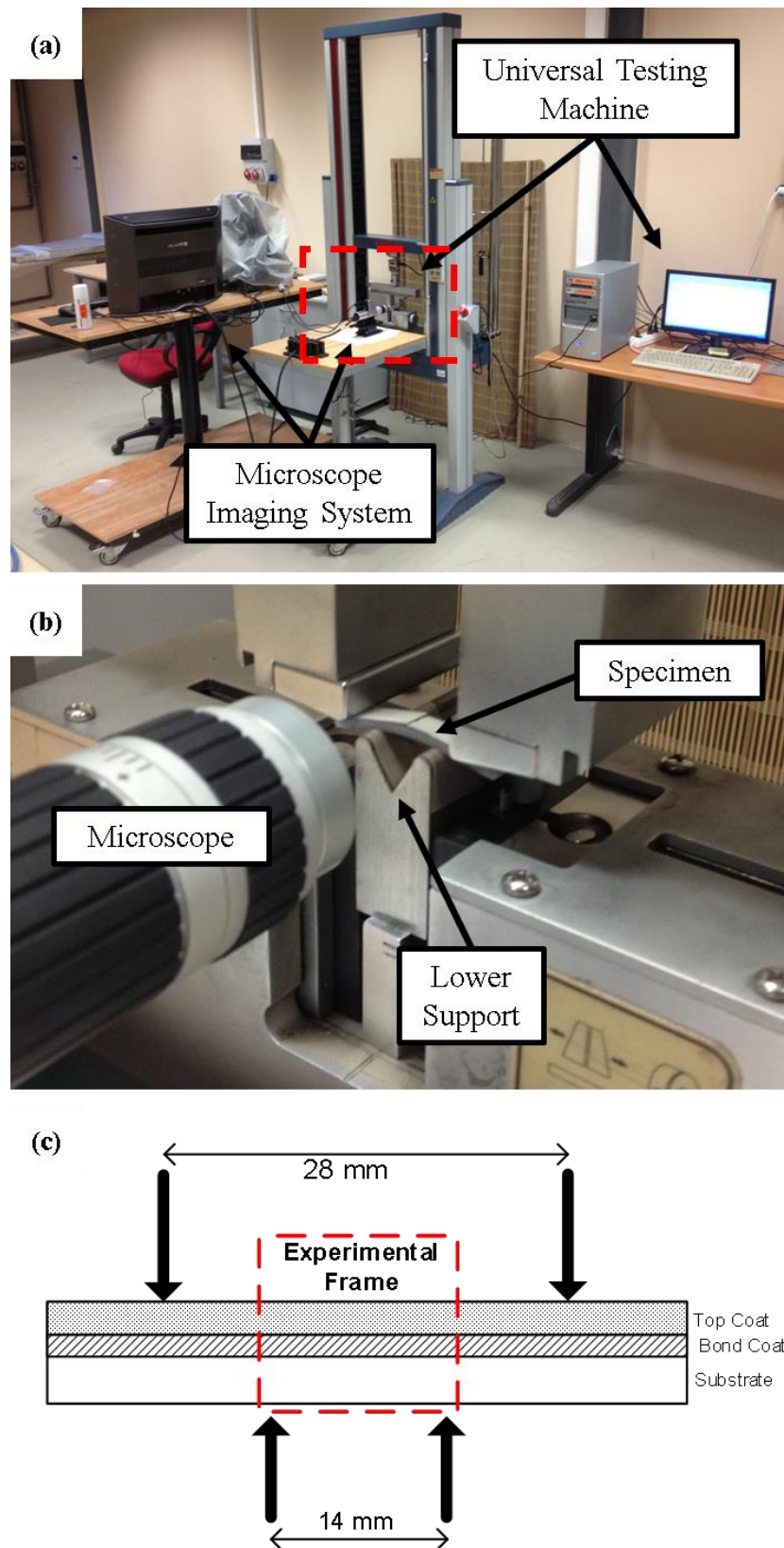


Figure 4.1.1 (a) Experimental setup of the micro-bending test, (b) the close up view of the experimental space and (c) free-body diagram of the loading condition.

4.1.3. Testing Procedure

The upper support was lowered to bend the specimen. First, the upper support touched on the surface of the TBC specimens and loaded up to 10 N to press the specimen between supports. The loading rate was set to 0.25 mm/min and one image was acquired for every two seconds. The testing procedure was followed up to an obvious interface cracking was acquired.

4.2. Results

There are three major results of a micro-bending test on three different TBC specimens. First, the load-stroke data can be achieved after the testing. Second, the image series of each test and DIC analysis of these images can reveal the cracking mechanisms and the order of occurrence of the failure. Third, as the load, stroke and exact time of the interface failure are known, the critical energy release rate of the interface can be calculated by either Euler-Bernoulli beam bending theory based energy release rate formula or the bi-material solution implemented in Chapter 3.

4.2.1. Load-Stroke Data

In Fig. 4.2.1, the load-stroke data of each specimen is presented while specimens having different coating thicknesses marked according to coating thicknesses. The maximum applied stroke for each specimen was different, since the stopping criteria of the test is the ultimate failure of the interface. There are two types of arrows in Fig. 4.2.1 which are indicating different stages of failure. Red arrows indicate the initial moment that there appear a vertical cracking at the TC. Black arrows indicate the initial moment that there appear an interfacial cracking or a kinking of the vertical crack to TC/BC interface. In Fig. 4.2.2, the load-stroke plot in Fig.4.2.1 is normalized with respect to the width of specimens since width of the specimens are effecting the maximum load applied and slope of the elastic range of the load-stroke data.

Three different TBC specimens having different coating thicknesses were subjected to same loading conditions and the load-stroke data is plotted in Fig. 4.2.1. For similar materials tested, slope of elastic range of the load-stroke data should be similar. However, in Fig. 4.2.1, the slope of thin specimens seems to be very different from thick and standard specimen. In Fig. 4.2.2, the load-stroke data in Fig. 4.2.1 is normalized with respect to the width of the specimen. As a result, the slope of the elastic range of the load-stroke data comes closer to each other.

In Fig. 4.2.1, the ultimate failure load of thick, standard and thin specimens are 1.8 kN, 2.0 kN and 2.7 kN, respectively. Therefore, it can be said that the ultimate strength of coatings decreasing as the coating thickness increases. The specimen which has the maximum coating thickness was failed quicker than the other specimens. Therefore, thick specimen data set is shorter than the rest of the specimen data. Initial vertical cracking first appeared in thick specimen. Then it appeared in standard and thin specimens successively. Similar to the vertical cracking, the first interfacial cracking was also seen in thick specimen and followed by standard and thin specimen successively. As a result the TBC coating gets thicker, the interfacial strength of the TC/BC get lower.

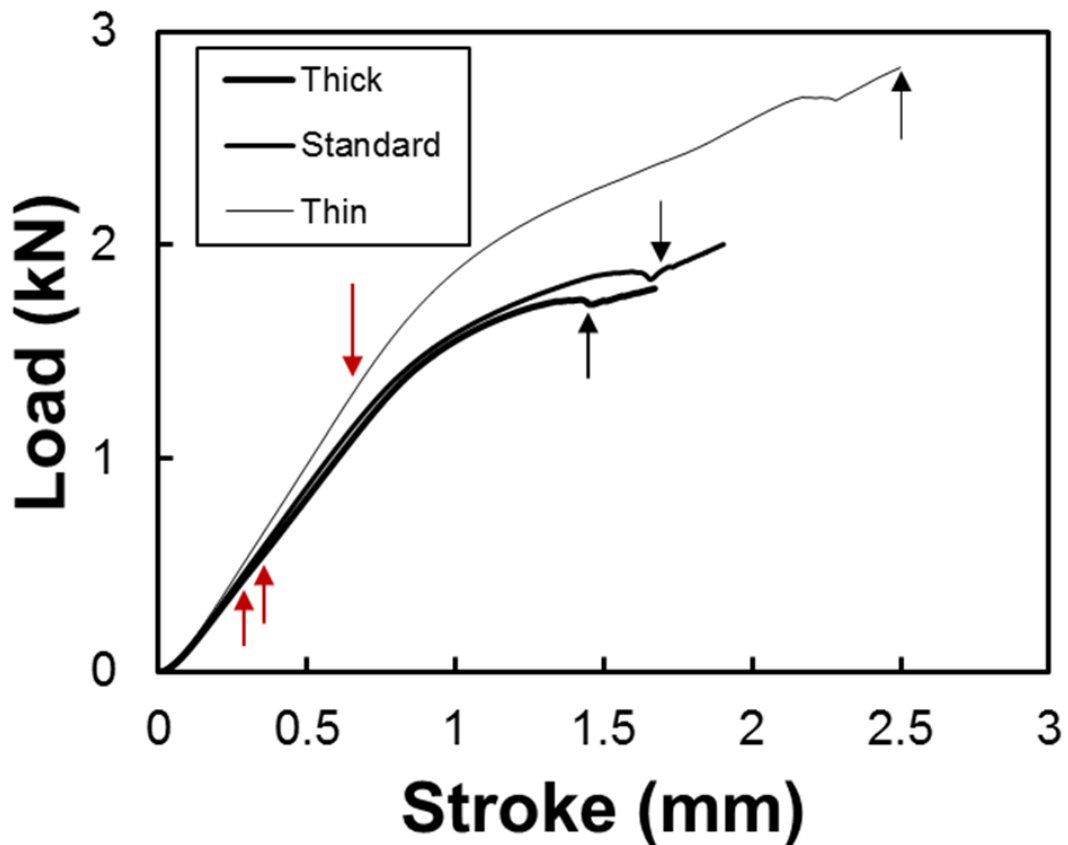


Figure 4.2.1 Load vs stroke data of the micro-bending test of TBC specimens, labeled according to their relative coating thicknesses.

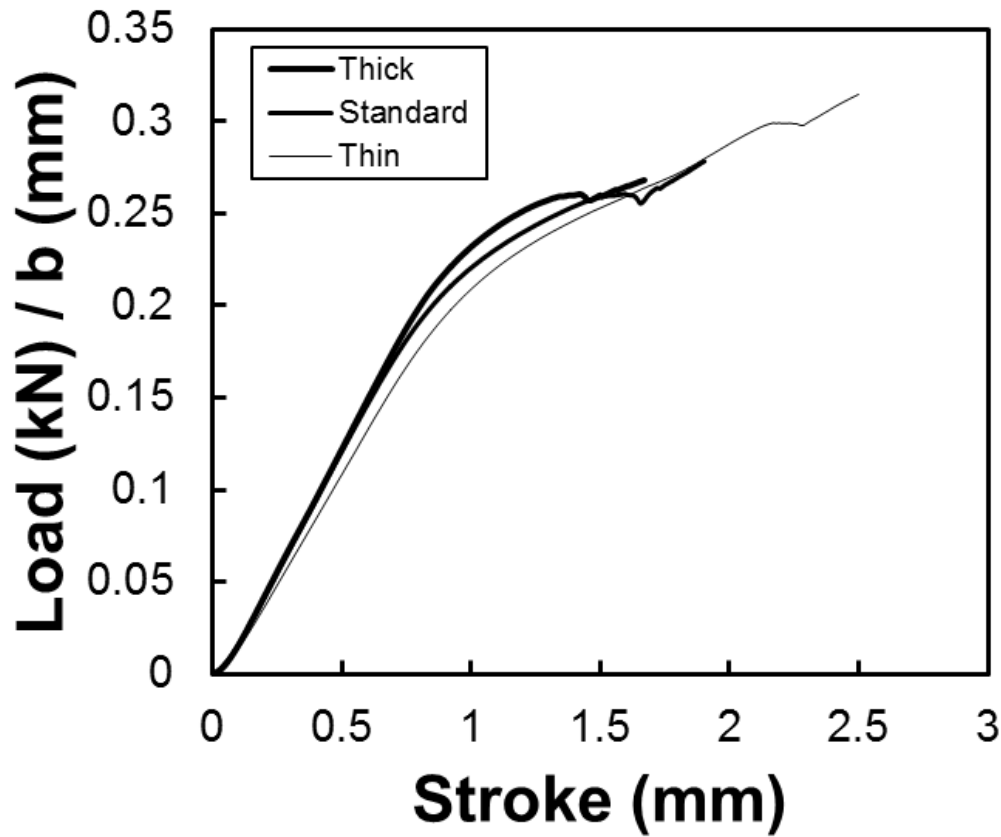


Figure 4.2.2 Load vs stroke curve in Fig. 4.2.1 normalized with respect to the width of each specimen.

4.2.2. Cracking Mechanisms of APS TBCs

Thermal barrier coatings are very brittle coatings coated on ductile metallic substrates. The cracking mechanism of such systems are very important for both design and performance point of view. Failure of a TBC is directly related to the TC/BC interface failure. Therefore, the driving mechanisms of the interface failure and the order of occurrence should be examined.

As a preliminary test, specimens were subjected to same loading conditions without any image acquisition. The resultant images are very similar to each other. In Fig. 4.2.2, cross-sectional microscopy of one of the failed TBC specimen is presented. There are two types of cracking occurred in TBC systems under four-point bending. First one is the vertical cracking of coatings and second one is the interface cracking. There are also cracks propagated horizontally in the TC but these cracks are only extensions of the vertical cracks in TC. It is evident that the interfacial cracking can chop off TC from BC.

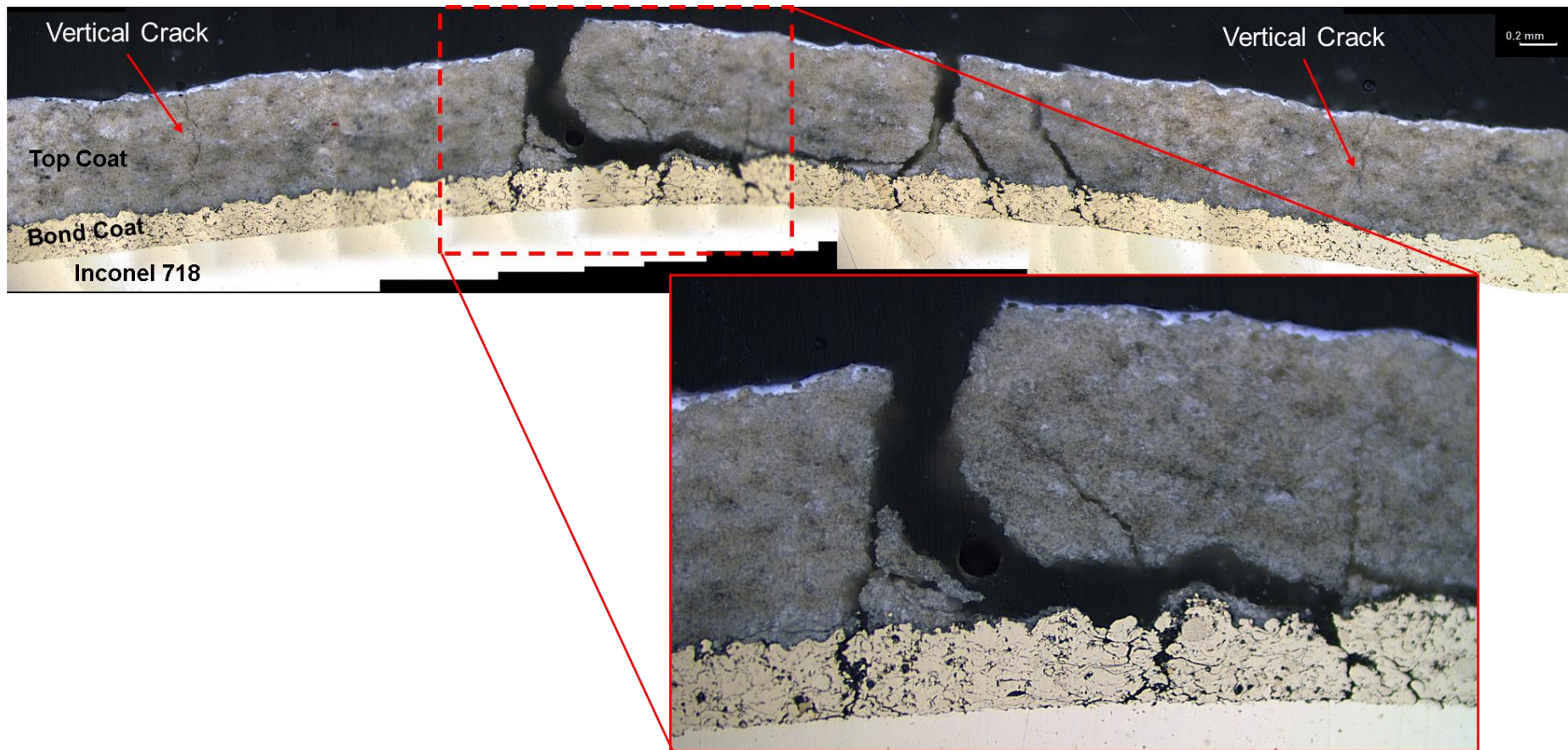


Figure 4.2.3 Cross-sectional microscopy of a typical TBC cracked after a micro-bending test showing the delamination between TC and BC and the vertical cracking on layers lead to the ultimate failure of the TBC.

In Fig. 4.2.4, Fig. 4.2.5 and Fig. 4.2.6 DIC results of thick, standard and thin specimens showing Von Mises strain contours are presented. The scale of each image set was adjusted to most appropriate value to visualize the vertical and interfacial cracking. The following images are also the history of cracking. For each different specimen, similar cracking mechanisms were followed but each one of them occur differently.

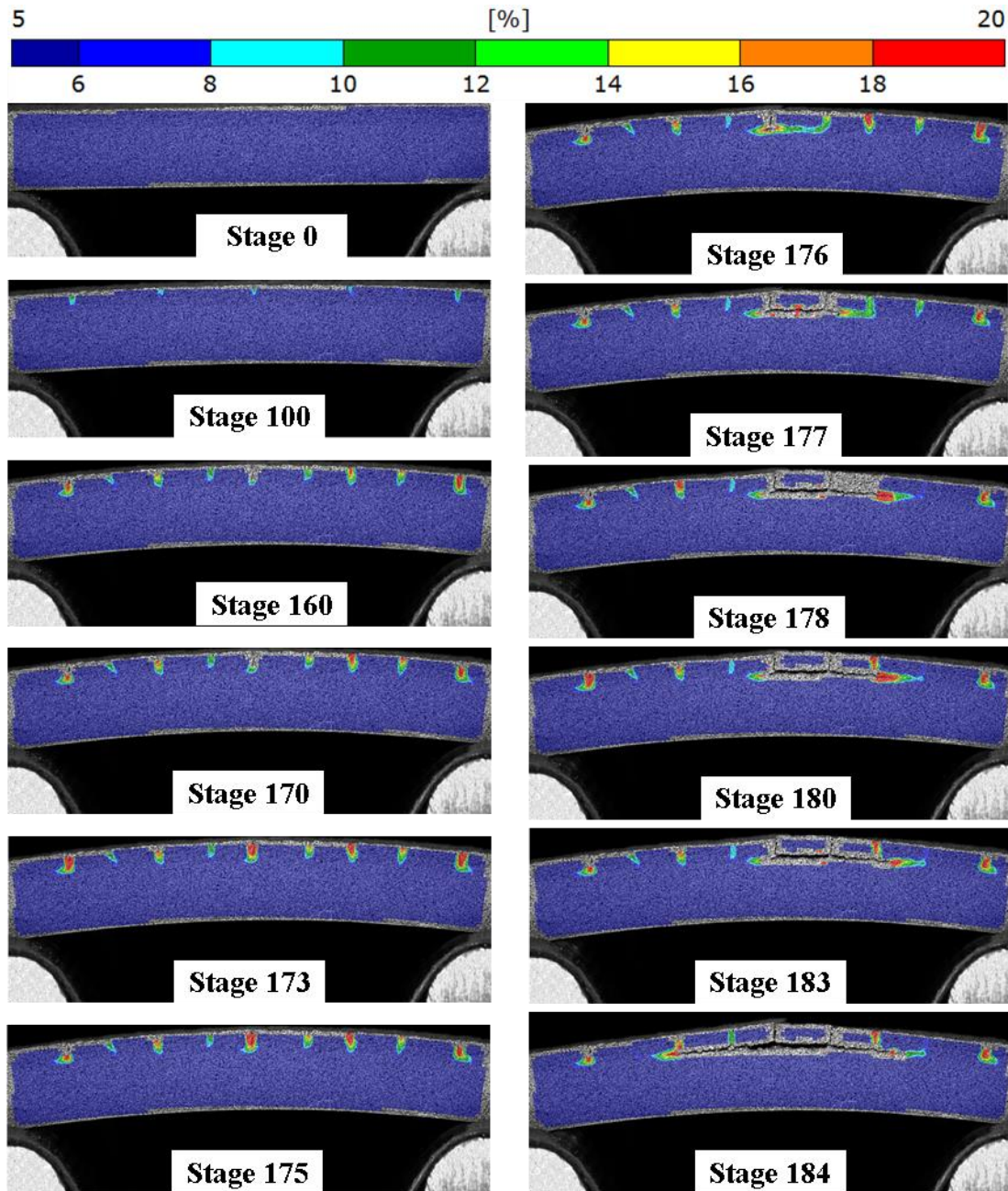


Figure 4.2.4 Thick specimen test results showing Von Mises strain contours and microscope images generated by ARAMIS DIC analysis.

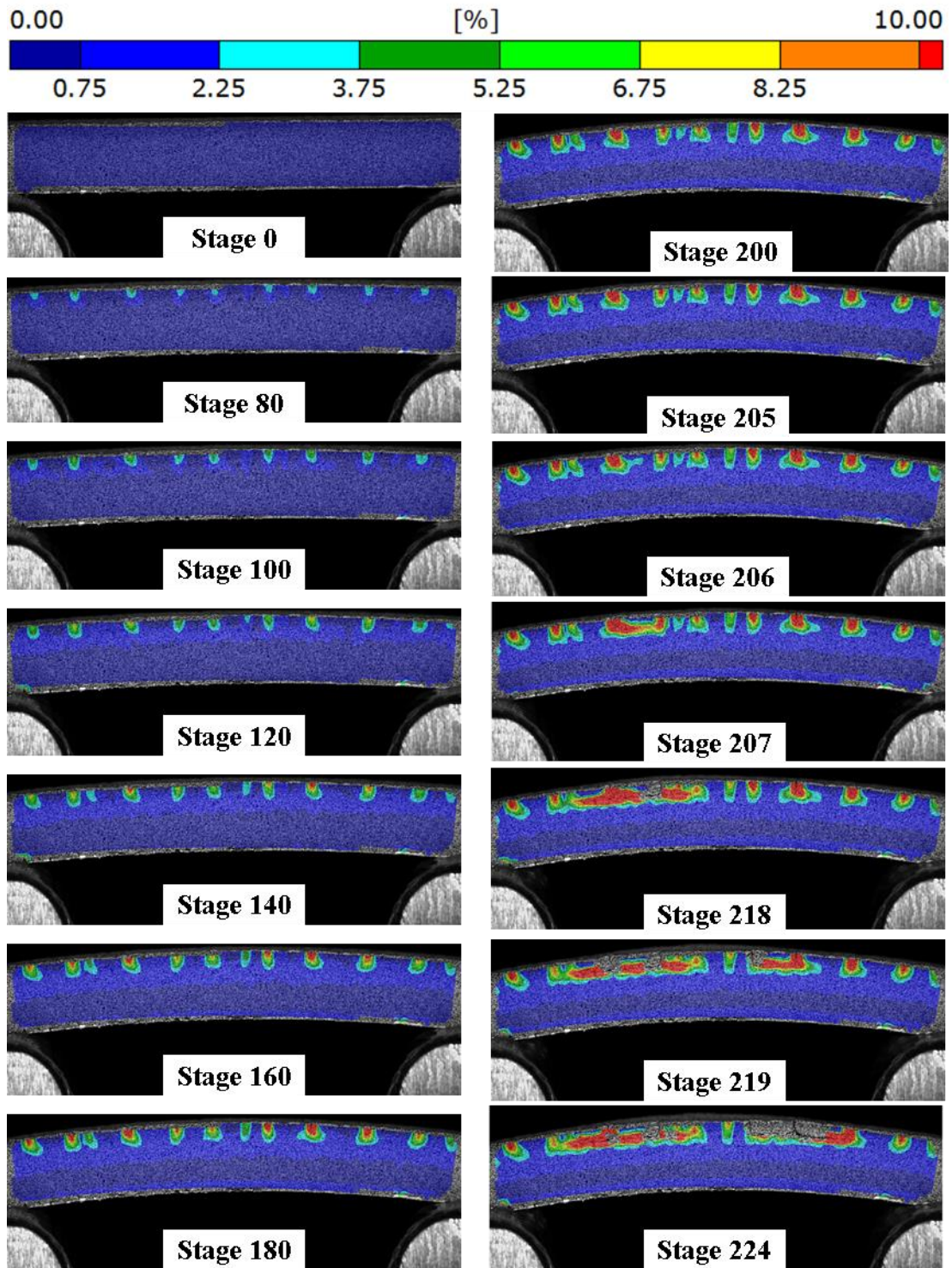


Figure 4.2.5 Standard specimen test results showing Von Mises strain contours and microscope images generated by ARAMIS DIC analysis.

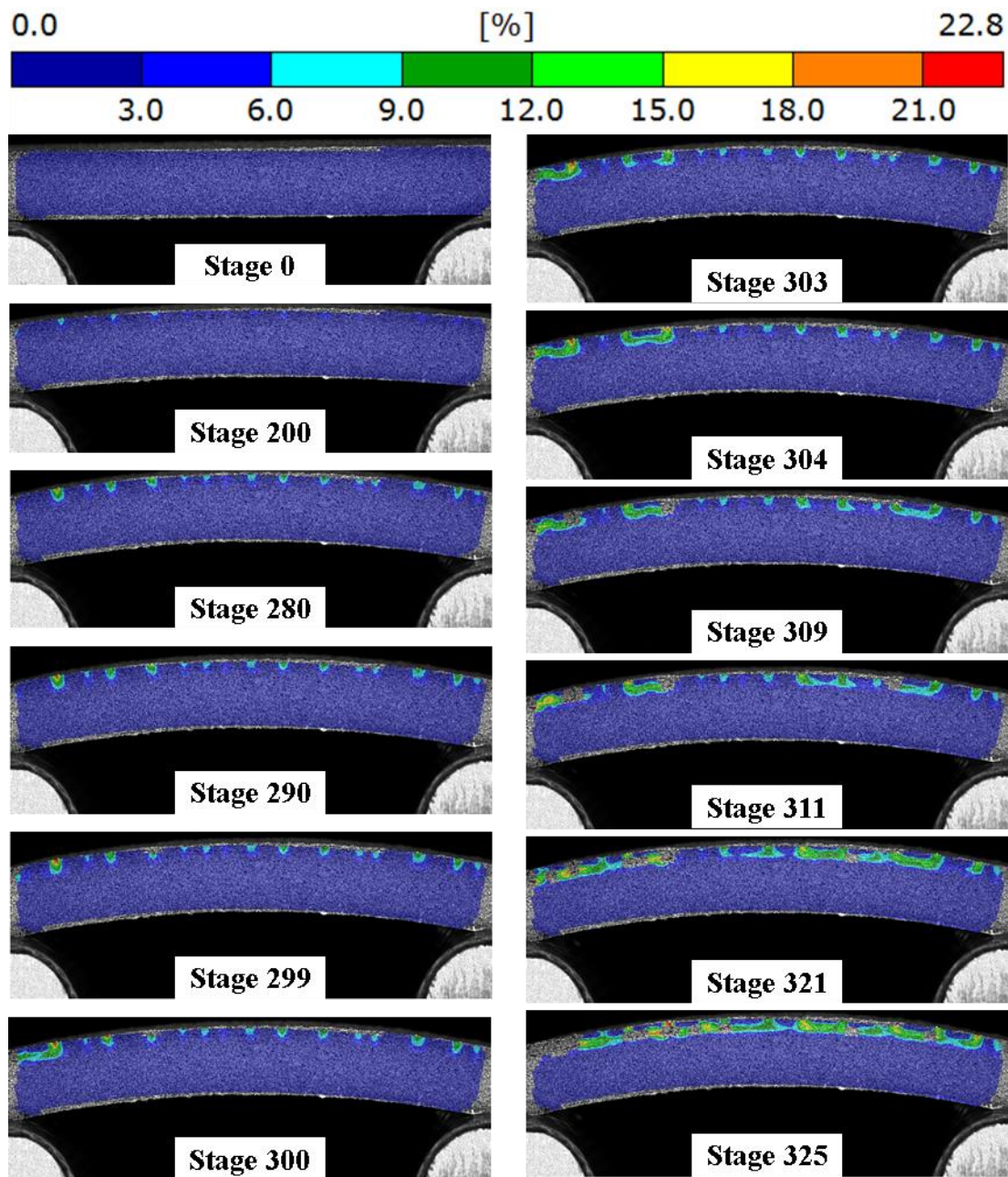


Figure 4.2.6 Thin specimen test results showing Von Mises strain contours and microscope images generated by ARAMIS DIC analysis.

Cracking mechanism of different TBC specimens are similar. First, there appear multiple vertical cracks. These cracks propagate up to the TC/BC interface. As crack tips of the vertical cracks touch the interface, the propagation stood still for a while. There appear a certain spacing of vertical cracks correlated with the thickness of the coating. Second, the vertical crack tips at the TC/BC interface start to kink to the interface and propagate through the interface.

Total duration of the interface failure is also related with the spacing of the vertical cracks. For thick and standard specimens the interfacial failure took more time than the thin specimen. Thin specimen failed at its interface in a very short time. As spacing between two vertical cracks decreasing, the interface cracks can conjoin together in a shorter time. In Fig.4.2.7, the measured spacing for three different specimens having different coating thicknesses is presented. The definition of the spacing of vertical cracking is illustrated in Fig. 4.2.7(a). As the thickness of the coating increases, the average distance between vertical cracks increases.

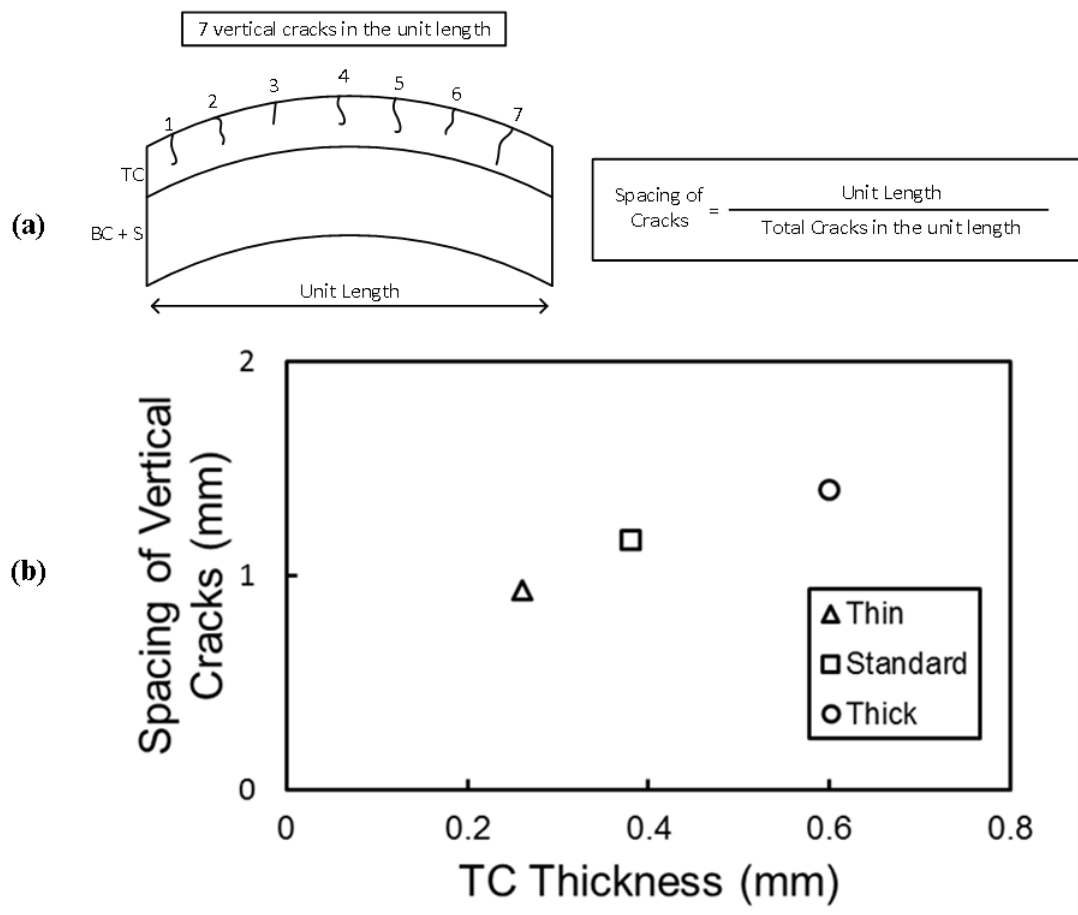


Figure 4.2.7 (a) Illustration of the definition of crack spacing, (b) Spacing of vertical cracks at the end of the tests for varying coating thicknesses.

4.2.3. Critical Energy Release Rate of TC/BC Interface

The energy release rate of an interface can be calculated by two different analytic methods introduced in the previous chapter. Both of these methods based on experimental measurements. First, Euler-Bernoulli beam bending theory based solution can be used. There are two requirements to use this method: the detection of crack propagation and the exact load that the crack propagates. Second, bi-material solution of an interface of a composite beam can be used. There are four requirements for this method: the detection of crack propagation, exact location of the crack tip, crack opening displacements (COD) and the distance between the crack tip and the location of COD measurements. For both methods, acquired image series and DIC analysis are used to satisfy these requirements especially to detect the crack propagation, measure CODs and locate the crack tip.

In Fig. 4.2.8, the last acquired images of thick, standard and thin specimens are presented. As the coating thickness increases the interface cracks becomes more visible. TC of the thick specimen is completely delaminated at the TC/BC interface. However, the images presented in Fig. 4.2.8 was acquired at the end of the micro-bending test. Therefore, the measurement capabilities of DIC should be re-examined for such a small area.

As mentioned at the previous section, for both critical energy release rate calculation method DIC is essential. For Euler-Bernoulli beam bending theory based solution, DIC is needed to detect the exact time and load that interface cracking initiated. For a bi-material solution COD should be measured by DIC and crack tip also need to be located. However, for such exact measurements, DIC needs more pixels as a result more deflection. From DIC results of TBC tests, COD are so small that DIC could not acquire any meaningful data. Therefore, DIC could only be used for visual aid which is needed to detect the crack location. As a result, critical energy release rates of TC/BC interfaces for three different specimens were calculated by Euler-Bernoulli beam bending theory based method.

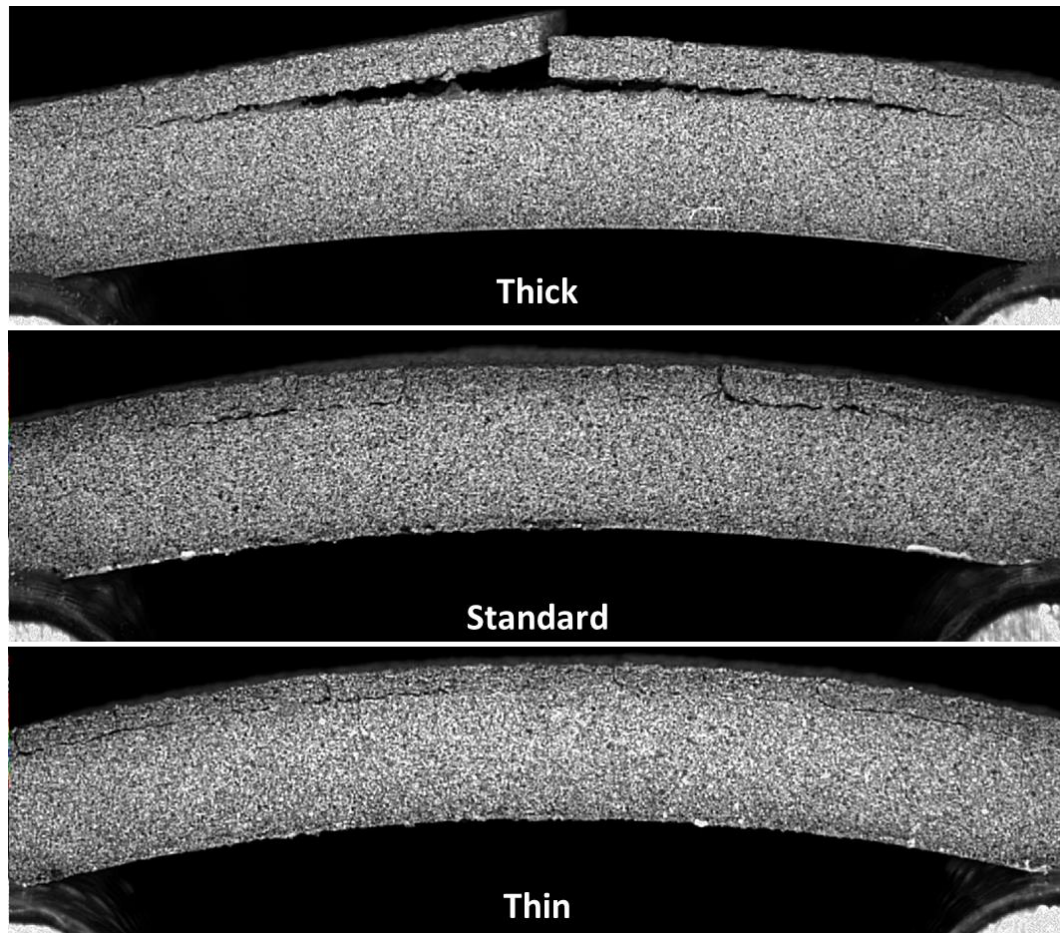


Figure 4.2.8 The final images acquired from the micro-bending test of specimens (a) thick, (b) standard and (c) thin.

Critical energy release rate of three different TBC specimens having three different coating thicknesses calculated by Euler-Bernoulli beam bending theory based method. The calculated critical energy release rates of three different coatings, corresponding loads, moment and their normalized values with respect to the width of specimens are presented in Table 4.2.1. As mentioned in the Load-Stoke results, ultimate failure load increases as the thickness of the coating decreases. However, critical energy release rate of TC/BC interface is increasing as the coating thickness increases. This is also the fact that the coating thickness affects critical energy release rate. The critical energy release rate formula is not dependent to the width of the specimen.

Table 4.2.1 Critical energy release rate of three different TBC specimens having different coating thicknesses calculated by Euler-Bernoulli beam bending theory based method.

	Thick	Standard	Thin
G_{critical} (kJ/m²)	8.27	7.36	6.80
Load (kN)	1742	1892	2833
Load (kN) / b (mm)	272	291	311
Moment (N/m)	610	662	992
Moment (N/m) / b (mm)	95	102	109

In Fig. 4.2.9, the critical energy release rate of three different specimens are plotted with respect to the coating thickness of the specimen. Three critical energy release rates are linearly changing with respect to the coating thickness. Therefore a fourth specimen having another coating thickness can be estimated from the plot in Fig. 4.2.9.

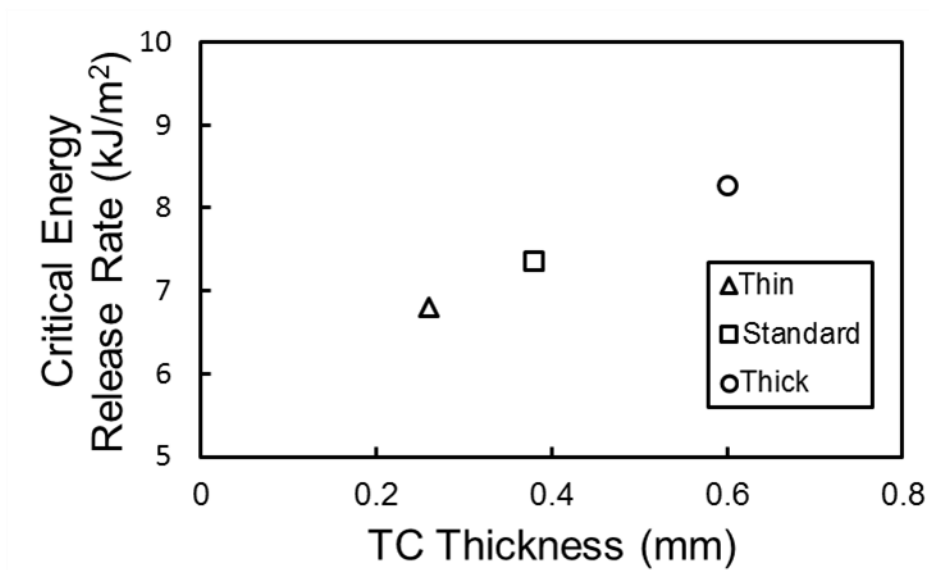


Figure 4.2.9 Critical energy release rate of three different TBC specimens having three different thicknesses.

CHAPTER 5

SUMMARY AND CONCLUSIONS

5.1. Summary

The objective of this thesis is to investigate the failure of an air plasma sprayed thermal barrier coating system using quantifiable experimental methods such as the four-point bending test. The developed method is then used for a specific TBC system and the interfacial toughness is measured for three different thicknesses.

First, the historical background of the TBCs is given. The engineering challenges faced by the industry and the way lead aviation to use thermal barrier coatings in the propulsion systems. Then, the ultimate goal in propulsion, “Green Aircraft” motto is illustrated and the importance of TBC and TBC based researches in the way to a greener aircraft. Then, the fundamental cooling methods where TBCs are mentioned in the literature is presented with other cooling methods, such as internal cooling holes and film cooling. Then, the failure of thermal barriers are discussed and micro mechanics of the failure of a TBCs are illustrated by giving examples from the literature. The studies on experimental evaluation of thermal barrier coatings are also introduced in the first chapter. Drory & Hutchinson [21] evaluating the entire possible testing techniques are presented as the fundamental article and some similar articles were summarized.

In the second chapter, characterization studies of the specimens were presented. First, the metallurgical and mechanical testing methods are given within a comprehensive literature review of these testing methods. Then, microstructure of a typical APS TBC specimen is investigated. Second, the nanoindentation tests conducted to measure mechanical properties of the layers in the TBC specimens were presented. By doing

this, the specimen set is defined with their microstructure and mechanical properties which are also used in the last chapter.

In the third chapter, two different analytic solutions for the same geometry and loading conditions to calculate the energy release rate and interface toughness is introduced by giving the derivations. These two analytic solutions are Euler-Bernoulli beam bending theory based composite beam solution and a bi-material based solution introduced by Charalambides et al. [11]. The verification of two analytic methods by using DIC and load-displacement measurements for three different interfaces. Different adhesives constructed different interface properties and gave different results. For Loctite 496 and Bison Metal epoxy interfaces the methods were verified. However, it is seen that the measuring a critical energy release rate of such thin interfaces is nearly impossible by measuring crack-opening displacements from DIC.

In the last chapter, micro bending tests were performed on three different TBC specimens having different thicknesses. From these tests, the cracking mechanisms of TBC specimens were introduced and a critical energy release rate was calculated by using the analytical solution based on Euler-Bernoulli beam bending theory.

5.2. Conclusions

In this thesis, failure of an air plasma sprayed thermal barrier coating is investigated by using quantifiable experimental methods for a set of typical APS TBC specimens for three different coating thicknesses.

In chapter 2, *“metallurgical and mechanical characterization of thermal barrier coating specimens”*, components that construct TBCs were metallurgically and mechanically studied from their powder form to their as-sprayed coated form as a structural layer. In this chapter the following conclusions were reached:

- BC and TC layers of TBC systems are elementally homogeneous since the raw materials used to produce TC and BC are elementally homogeneous. Therefore, EIT measurements does not depend on the location of indents.
- The splat like structure of BC can be observed by using Kalling No.2 solution. Oxides and pre-cracks in BC is more obvious on etched specimens.

- Microstructure of thermally effected TBC specimens were changed drastically. Pre-cracks in TC propagated because of the thermal expansion mismatches between materials. As a result, it can be said that the microstructure of TBCs was severely affected from elevated temperatures.
- Inconel 718 data sheet available in the Internet and the elastic modulus of Inconel 718 is indicated as 205 GPa for room temperature. Nanoindentation tests on Inconel 718 substrate was conducted to verify the data sheet and to increase experience on nanoindentation.
- HIT and EIT measurements on TC and BC layers vary with the applied load up to a threshold applied load where the size effect of the indent diminish. A series of measurements were conducted at different loads to determine the threshold load. Characteristic EIT and HIT of TC and BC layers were measured at a certain maximum load applied defined according to the threshold load determined from the previous measurements.
- Microstructural flaws and porous structure of BC and TC layers scatters the nanoindentation measurements. Therefore, nanoindentation measurements on TC and BC layers were analyzed by the Weibull distribution method to determine characteristic EIT and HIT values. This data is used in the analysis of the developed experimental method to measure interface toughness of TBC systems in Chapter 4.
- Different batches of TBCs have similar microstructure characteristics and same mechanical properties as long as the material and production procedure is the same.

In chapter 3, “*development of 4-pt bending test methodology for interface failure*”, the needed methodology to measure critical energy release rate of TC/BC interface of TBC specimens is developed by using another model geometry. Charalambides et al. [11] derived the analytical solution for a bi-material interface to solve an interface failure problem using finite element method. In this study, the same method was applied to DIC based experimental testing procedure. The model geometry is a pre-cracked Al-PMMA composite beam glued together by using different adhesives subjected to a 4-pt bending test. In this chapter the following conclusions were reached:

- Three different adhesives were used in 4-pt bending tests. Different adhesives exhibit different interfacial characteristics. Contact adhesive exhibited a stretching interface without a crack propagation. Metal epoxy interface exhibited a steady state cracking. Loctite 496 interface exhibit a dynamic crack growth.
- Critical energy release rate of metal epoxy and Loctite 496 interfaces were measured by using two different methods. However, these two different methods gave similar critical energy release results for the two different interfaces.

In chapter 4, “*determination of fracture property of specimens with thermal barrier coating*”, micro-bending test developed in the previous chapter was applied on TBC specimens having different coating thicknesses to study the failure mechanism and to measure critical energy release rate of TC/BC interfaces. In this chapter the following conclusions were reached:

- Three different TBC specimens were found to fail in a similar manner in terms of failure mechanism. However, final interface varied between intact the interface to separated interface as the thickness increases.
- Critical energy release rate of an interface increases with increasing TC thickness. Therefore, the critical energy release rate of TC/BC interface of a specimen indicates the failure energy instead of the interface strength.

5.3. Future Work

In this study an experimental methodology was developed and applied on TBCs to measure fracture properties of TC/BC interface. In the future the following topics can be considered to achieve more conclusive results and increase our understanding on TBC materials:

- TBC specimens used in this thesis were manufactured by TEI which are for after-burner parts. Therefore, microstructure of TBC specimens are not state-of-the-art compared to the typical combustion chamber APS TBC microstructures. If better quality TBC specimens are used in tests, more consistent results can be achieved.
- More nanoindentation measurements on TC and BC layers would increase the reliability level of characteristic values of EIT and HIT. At least 100 measurements are needed to achieve %90 reliability for each layer.
- Effect of thermal exposure on TBCs should be examined. Extra specimens can be thermally exposed and tested by following the developed method.
- Substrate thickness of Inconel 718 is around %80 percent of the total thickness of TBC specimens. For thinner substrate thicknesses better DIC measurements can be conducted and imaging systems can be focused on a smaller area.
- The FEM model of 4-pt bending tests of both Al/PMMA composite beam and TBC specimens should be generated to validate the procedure that was followed.

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APPENDIX

A. Crane Operator's Dilemma

There was a crane operator who needed to put a cargo into a dark hole. Crane operator on the ground has to operate the crane on a helicopter in a cloudy weather. He could only see the black non-reflecting carbon wire with the cargo hanging below the clouds. Crane operator started leveling the cargo down at a constant speed. Under these circumstances, crane operator's view is blocked by cloudy weather from above and the edge of the hole from below. Suddenly the crane got broken, and cargo started falling in the hole. Since the crane operator saw only a small portion of the wire, he could not realize that the cargo was falling and could not push the emergency lock button. After a disastrous event at the hole, the crane operator got another job at the shipyard. He needed to put another cargo, this time, on the ground under same conditions. While he was leveling the cargo, suddenly the crane broke down similar to what happened at the hole. Since crane operator had full vision of the system, he immediately realized that the cargo was falling. As a result, crane operator could trigger the emergency brakes, and saved the cargo and the day.

B. Bison Metal Epoxy Load Drop History

Time(s)	Force(N)	Moment (Nm)	Stroke(mm)	dP/dt
<i>Stage 2</i>				
28,3	114,9	0,2871	0,2840	-0,05
<i>Stage 3</i>				
<i>Stage 4</i>				
49,5	185,1	0,4626	0,4663	-0,05
<i>Stage 5</i>				
61,7	224,8	0,5620	0,5713	-0,05
63,7	231,6	0,5790	0,5883	-0,05
<i>Stage 6</i>				
74,1	267,9	0,6698	0,6783	-0,05
<i>Stage 7</i>				
81,9	294,2	0,7354	0,7457	-0,05
83,1	297,3	0,7431	0,7560	-0,05
85,9	305,4	0,7634	0,7800	-0,05
86,9	308,2	0,7705	0,7883	-0,1
<i>Stage 8</i>				
88,3	312,7	0,7818	0,8007	-0,05
95,5	335,6	0,8390	0,8627	-0,05
<i>Stage 9</i>				
104,1	358,6	0,8965	0,9367	-0,05
104,7	360,0	0,9000	0,9417	-0,1
104,9	360,7	0,9016	0,9433	-0,05
105,1	361,1	0,9028	0,9450	-0,05
106,3	364,3	0,9108	0,9553	-0,05
107,3	367,3	0,9181	0,9640	-0,05
<i>Stage 10</i>				
112,1	381,6	0,9540	1,0053	-0,05
112,5	382,9	0,9571	1,0090	-0,05
114,3	387,6	0,9689	1,0243	-0,1
114,9	389,4	0,9735	1,0293	-0,05
115,5	391,0	0,9775	1,0347	-0,05
116,3	393,4	0,9834	1,0417	-0,1
117,9	398,1	0,9953	1,0553	-0,05
120,5	405,6	1,0139	1,0777	-0,05
<i>Stage 11</i>				
127,7	426,2	1,0654	1,1393	-0,1
128,9	428,8	1,0720	1,1493	-0,05
129,3	429,4	1,0734	1,1530	-0,05
129,55	429,7	1,0743	1,1550	-0,05

130,5	432,3	1,0806	1,1633	-0,05
133,1	438,8	1,0969	1,1853	-0,05
<i>Stage 12</i>				
135,9	445,7	1,1141	1,2093	-0,05
138,7	452,3	1,1308	1,2333	-0,05
138,9	452,7	1,1316	1,2350	-0,1
139,3	453,2	1,1329	1,2387	-0,05
141,9	459,6	1,1490	1,2610	-0,05
142,1	460,1	1,1501	1,2627	-0,15
142,5	461,1	1,1528	1,2660	-0,05
143,1	462,5	1,1561	1,2713	-0,05
144,1	464,9	1,1623	1,2797	-0,05
<i>Stage 13</i>				
151,9	482,7	1,2068	1,3470	-0,05
152,7	484,6	1,2115	1,3540	-0,1
153,5	486,6	1,2165	1,3607	-0,05
154,1	488,0	1,2199	1,3660	-0,05
<i>Stage 14</i>				
155,300	490,7	1,2266	1,3763	-0,1
156,7	494,3	1,2356	1,3883	-0,15
158,9	499,9	1,2496	1,4070	-0,05
161,9	507,2	1,2679	1,4330	-0,05
162,7	509,2	1,2730	1,4400	-0,05
163,9	512,1	1,2803	1,4500	-0,1
167,5	521,0	1,3024	1,4810	-0,05
<i>Stage 15</i>				
170,7	528,9	1,3221	1,5083	-0,1
171,9	531,7	1,3293	1,5187	-0,05
172,3	532,5	1,3311	1,5223	-0,1
175,1	539,2	1,3479	1,5463	-0,05
176,1	541,3	1,3533	1,5550	-0,05
176,3	541,9	1,3548	1,5567	-0,05
176,5	542,3	1,3558	1,5583	-0,05
177,3	544,5	1,3611	1,5653	-0,05
177,9	545,8	1,3645	1,5703	-0,05
179,1	548,4	1,3709	1,5807	-0,05
179,3	548,8	1,3720	1,5823	-0,1
<i>Stage 16</i>				